

## OPTIMIZATION OF POTASSIUM (K) FROM DOMESTIC SEWAGE WATER PHYTORID SEWAGE TREATMENT PLANT: A STUDY USING BOX-BEHENKEN DESIGN

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### ABSTRACT

Removal of the K from the domestic sewage was studied by using phytoid sewage treatment plant at Agriculture College Maharajbag, Nagpur during the year 2013-2014. The objective of this investigation was to study the efficacy of the phytoid sewage treatment plant in K removal from the domestic sewage and to determine the optimum condition using the response surface methodology. A Box- Behnken model has been employed as an experimental design. The effect of three independent variables namely hydraulic loading i.e. flow (50 - 150 m<sup>3</sup> d<sup>-1</sup>), dilution (10 - 80 %) and spatial length (16 - 100 %) has been studied on the K removal from the sewage in bench mode of the experiment. The optimal conditions of the K removal were found to be flow: 100 m<sup>3</sup> d<sup>-1</sup>, dilution: 27.29 per cent and spatial length: 16 per cent. Under these experimental conditions, the experimental K removal obtained was 40 per cent. The proposed model equation using the RSM has shown good agreement with the experimental data, with a correlation coefficient (R<sup>2</sup>) of 98.71 %. The result showed that optimised condition could be used for the efficient removal of the K from the domestic sewage. Therefore, Box- Behnken design and response surface methodology could economically and efficiently be applied for modelling of some pollutant concentration removal from the phytoid sewage treatment plant.

(Keywords: Pollutant, Box-Behnken design, sewage water, dilution, optimization, response surface modelling)

### INTRODUCTION

The increase in the human population together with rapid industrial and urban development has resulted in a sharp rise in the demand for water while the available fresh water supplies have remained nearly constant. Increasing need for water has resulted in the emergence of domestic wastewater application for agriculture and its relative use. The waste water is mixture of sewage water, agricultural drainage, industrial waste effluents and hospital facilities. It is well known that the well water from domestic origin contains pathogens, suspended solids, nutrients (Nitrogen and Phosphorous) and other organic and inorganic pollutants (Andrew *et al.*, 1997). This Complex nature of sewage made it difficult to obtain the significant K removal efficiency from the sewage.

In view to minimise the environmental health hazards, these pollutants needs to be brought under safe permissible limit for safe disposal of the waste water. Therefore, removal of organic contaminants and pathogens from the waste water is utmost important for its reuse in agriculture and different activities ( Scholes *et al.*, 1999).

The conventional waste water treatment technologies as adopted in industrialised nations are expensive to build, operate and maintain (Muzumdar and

Roy, 2000) especially for the decentralised community. Research efforts are underway (Wang and Hongewei., 2006) for the development of treatment technologies suited to this development communities. Artificial wetland system is a potential method for sewage treatment to recycle the treated water for irrigation.

Selection of technology for sewage treatment should be based on the criteria such as treatment plant which work without electricity, requires minimum maintenance and the most importantly the technology should be self-sustainable. Therefore, it is very important to develop the technology based upon natural method of treatment of sewage using constructed wet land. Use of plant species with their root system along with natural attenuation process can be combined together to get phytoid technology. It is one such solution which can be easily implemented in cities as well as in rural areas for treatment of waste water. The system is based on use of specific plants normally found in natural reed with filtration and treatment capacity (Rai, 2008). This system can be utilized for wide variety of application. It can be used for secondary and tertiary treatment of municipal waste water, sludge management; treatment of industrial and agricultural effluent as well as landfills leachates. Phytoid bed is a scientifically developed, sustainable constructed wet land

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treatment methodology for domestic waste water. It combines physical, chemical and biological processes. In the methods based absorption, it is desirable to have knowledge of the process variables and their influence on absorption capacity to maximise removal efficiency of the contaminants in sewage treatment plant and to bring them below phytotoxicity limit.

The K concentration plays an important role in the reuse of the waste effluents particularly in the agriculture for irrigation purpose. Phytorid sewage treatment plant based on wetland engineering technology showed good potential for K removal from the domestic sewage water. To date, research has mainly focussed on the use of phytorid sewage treatment plant for the treatment of domestic sewage water (Nicholas *et al.*, 1997). Phytorid sewage treatment plant is designed and operated in accordance with environmental protection agency guidelines and has provided good removal rates for the COD, BOD, Micronutrient, Nitrogen and Phosphorous. Sewage treatment plants based on CW's are now well-established methods for wastewater treatment in tropical climates (Burchell *et al.*, 2007). Recent, research has shown that sewage treatment plant based on constructed or restored wetlands can remove sediments and nutrients from nonpoint sources, including agricultural discharges (Jordan *et al.*, 1999).

Recently, much attention has been paid to phytorid sewage treatment for converting the domestic sewage into irrigation water resource because of the many advantages such as simple construction works on gravity, no electric power requirement, scalable technology, easy to maintenance, adds to the aesthetics, cost effective (Anon, 2005). The objective of the present study was to investigate the K removal from domestic sewage water through phytorid sewage treatment plant, optimize the experiment and to understand the effect of various operating parameters and their interactions on K removal. To optimise and evaluate interactive effects of independent factors in numerous chemical and biochemical processes response surface methodology (RSM) has been applied (Arulkumar *et al.*, 2011). It is essential that experimental design methodology is an economic way for extracting the maximum amount of complex information, a significant experimental time saving factor and moreover, saves the material used for analyses and personal costs as well (Kincl *et al.*, 2005). In the present study, the investigation proposes to use phytorid sewage treatment plant which is a modified engineered wetland to enhance pollutant removal by physical, chemical and biological processes. Phytorid bed is a scientifically developed, sustainable constructed wet land treatment methodology for domestic sewage.

## MATERIALS AND METHODS

### Treatment plant

The average flow rate of sewage water in Nag river was 426 m<sup>3</sup> hr<sup>-1</sup>. The scientific study to convert sewage into

water resource for irrigating the agricultural crops and gardening was proposed. A pilot project in collaboration with NEERI technology was undertaken as a model, and the treatment plant was constructed at Maharaj bag campus of Agriculture College, Nagpur. (21° 09' 0" N latitude and 79° 09' 0" E longitude). The designed capacity of the plant was 100 m<sup>3</sup> d<sup>-1</sup> of treated water. As sewage water flow in the Nag river was 426 m<sup>3</sup> hr<sup>-1</sup> in the driest month of May, it was not possible to construct the phytorid constructed wet land directly across the flowing river, requiring huge amount of funding and space. Therefore, as suggested by Massuod *et al.* (2009), the intake well was designed and constructed at the bank of the river.

### Experimental design

The Phytorid sewage treatment plant was designed for 100 m<sup>3</sup> d<sup>-1</sup> capacity. However, for maximum removal of K from the sewage treatment plant, the independent variables ranges selected were 50-150 m<sup>3</sup> d<sup>-1</sup> for sewage water flow (hydraulic loading), 10 - 80% for dilution and 16-100% for spatial length of the sewage treatment plant. The surface response modelling with Box-Behenken experimental design widely used for controlling the effects of parameters in any processes was used for the pre-treatment and optimization (Bhanarkar *et al.*, 2011). Its usage decreases number of experiments, using time and material resources. Statistical methods measures the effects of change in operating variables and their mutual interactions on process through experimental design way (Moghadhan *et al.*, 2011.)

The experimental plan consisted of base run 15, and independent variables were studied at three different levels of low (-1), medium (0) and high (+1). Box-Behenken experimental design has the advantage of fewer trials (15 basic run) than that would be required in full factorial design (27 runs). The removal of K concentration was taken as response (Y) of the experimental design. Furthermore, the analysis performed on the results is easily realized and experimental errors are minimized due to 3D response. Statistical methods measured the effects of change in operating variables and their mutual interactions on process.

### Response surface methodology

Statistical designs are powerful tools used to study the main as well as the interactive effects of different process variables on a process. Among them, response surface methodology (RSM) is a collection of certain statistical techniques for designing experiments, building models, evaluating the effects of factors and searching for optimal conditions of desirable responses. The advantages of adopting RSM: (1) it provides more information on the experiment than unplanned approaches; (2) it reduces number and cost of experiments; (3) it makes possible to study the interactions among experimental variables within the range studied, leading to a better understanding of the process; (4) it facilitates to determine operating conditions necessary for the scale-up of the process. Its greatest applications have been in industrial research, particularly

in situations where most of variables influencing the system feature (Myers and Monngomery, 2002).

### Experimental design for absorption studies

In present study, the Box–Behnken experimental design was chosen for finding out the relationship between the response functions (K removal) and independent variables (Hydraulic loading, dilution indicating initial concentration of sewage and spatial length of the sewage treatment plant for three different size fractions of 50 - 150 m<sup>3</sup>d<sup>-1</sup>, 10 - 80%, 16-100% respectively). In order to study the effects of three independent variables on pollutant concentration removal of the K; batch runs were conducted at different combinations of the process parameters using Box-Behnken designed experiments. The hydraulic loading i.e. flow range studied was between 50 to 150 m<sup>3</sup>d<sup>-1</sup>, dilution (initial concentration of sewage) was kept between 10 to 80% and the spatial length was varied between 16 and 100% (Table 1).. The operating ranges for hydraulic loading i.e. flow ( $X_1$ ), dilution ( $X_2$ ) and spatial length ( $X_3$ ) were determined by an iterative method. The relationship between the parameters and responses were determined using Box-Behnken design under RSM. In this study, the experimental plan consisted of 15 base run, and the independent variables were studied at three different levels of low (-1), medium (0) and high (+1). Box-Behnken design presents an approximately rotatable design with only three levels per variable and combines a fractional factorial with incomplete block design excluding the extreme vertices (Aslan and Cebeci, 2007). The Box-Behnken design has good performance with less error.

The percentage of K removal was taken as a response (Y) of the experimental design. If all variables are assumed to be measurable, the response surface can be expressed as follows:

$$Y = f(x_1; x_2; x_3; \dots; x_k) \dots (1)$$

Where Y is the answer of the system, and xi are the variables of action called factors.

The goal is to optimize the response variable Y. It was assumed that the independent variables are continuous and controllable by experiments with negligible errors. It was required to find a suitable approximation (equation) for the true functional relationship between independent variables and the response surface (dependable variable). Usually, a second-order model is utilized in response surface methodology. In the optimisation process, the responses can be simply related to chosen variables by linear or quadratic models. A quadratic model, which also includes the linear model, is given below:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} x_i x_j + \epsilon$$

Where,

Y = Response,

$x_1, x_2, \dots, x_k$  = Coded independent variables,

$\beta_i, \beta_{ii}$  and  $\beta_{ij}$  = Linear, quadratic and interaction coefficients, respectively,

$\beta_0$  = Constant, and

$\epsilon$  = Random error.

Trials were performed in triplicate. Minitab 16 Free trial version software package for regression and graphical analysis were used for analyses of the data. In all calculation, spreadsheets of Microsoft Excel 2007 were used as ODBC (Open Database Connectivity) data source running under windows.

### Statistical analysis

The significance of the independent variables and their interactions was tested by the analysis of variance (ANOVA). Results were assessed with various descriptive statistics such as t-ratio, p-value, F-value, degrees of freedom (df), coefficient of variation (CV), coefficient of determination ( $R^2$ ), adjusted coefficient of determination ( $R^2_{adj}$ ), sum of squares (SS), mean sum of squares (MSS) statistic test to reflect the statistical significance of the quadratic model. The tabulated value of F statistic corresponding to df was obtained at desired probability level (i.e. 0.05 significance level or 95% confidence).

### Validation experiments

The mathematical model generated during RSM implementation was validated by conducting additional experiments for different combination of the three independent variables in random fashion, each within its respective experimental range.

## RESULTS AND DISCUSSION

### K removal

The results of K removal along with experimental conditions are given in table 2. By applying multiple regression analysis on the design matrix and responses are given in table 2, approximate uncoded function for K concentration removal applicable for the treatment plant under study is given in following equation (Bhanarkar *et al.*, 2011):

$$Y = 11.8803 - 0.0288228 X_1 - 0.0405383 X_2 - 0.149455 X_3 + 0.000203617 X_1^2 + 0.000400748 X_2^2 + 0.000821310 X_3^2 - 3.69214 \times 10^{-04} X_1 X_2 + 7.14286 \times 10^{-05} X_1 X_3 + 0.000728316 X_2 X_3 \quad (2)$$

Where, Y is the pollutant concentration removal; and  $X_1, X_2, X_3$  are corresponding uncoded variable of flow (hydraulic loading), dilution and spatial length, respectively.

### Model statistical tests

The ANOVA was conducted as the analysis of variance, to test the significance of the developed model (Sen and Swaminathan, 2004). The summary of ANOVA of the regression model presented in table 3 indicated that the model equation could be used adequately to describe the concentration removal of K under a wide range of operating conditions. F-value of 169.73 being greater than the tabulated value ( $F_{tab} = 4.1$ ) implied that the model was significant. The probability value ( $p\text{-model} < F = 0.000$ , or below 0.0001) was less than 0.05, indicating that the quadratic

model was highly significant. The goodness of fit of the model was checked by calculating the regression coefficient ( $R^2$ ). A fairly high value of  $R^2$  (**98.07%**) suggests that most of the data variation was explained by the regression model. Moreover, a closely high value of the adjusted regression coefficient  $R^2_{adj}$  (**97.20%**) indicates the capability of the developed model to satisfactorily describe the system

behaviour within the studied range of operating parameters, Similar results were reported by Zhang *et al.* (2010) . According to them,  $R^2_{adj}$  corrects  $R^2$  for the sample size and the number of terms in the model; e.g. many terms in the model and small sample size might cause that  $R^2_{adj} \ll R^2$ , which is not obtained in our study.

**Table 1. Experimental range and levels of variables**

Variables	Unit	Code	Range and Levels		
			Low level	Centre	High level
Hydraulic loading (flow)	( $\text{m}^3 \text{d}^{-1}$ )	$X_1$	50	100	150
Dilution (Initial concentration of sewage)	(%)	$X_2$	10	45	80
Spatial length	(%)	$X_3$	16	58	100

**Table 2. Box-Behnken Experimental Design Matrix with variable and pollutant removal**

Factors: 3 Replicates: 2 Base runs: 15 Total runs: 30 Base blocks: 1  
 Total blocks: 1 Center points: 6 Design Table (randomized)  
 Run Blk A B C

Run order	Coded variable			Uncoded variable			Response
	X1	X2	X3	Flow( $\text{m}^3/\text{day}$ )	Dilution (%)	Spatial (%)	K
1	+	-	0	150	10	58	6.420
2	-	-	0	50	10	58	4.830
3	+	0	-	150	45	16	7.420
4	0	+	-	100	80	16	6.351
5	-	+	0	50	80	58	6.780
6	0	-	+	100	10	100	4.980
7	-	0	-	50	45	16	7.470
8	0	0	0	100	45	58	4.480
9	0	-	+	100	10	100	5.114
10	0	+	+	100	80	100	7.240
11	+	-	0	150	10	58	6.220
12	-	0	-	50	45	16	7.540
13	0	0	0	100	45	58	4.720
14	+	0	-	150	45	16	6.890
15	0	0	0	100	45	58	4.828
16	0	-	-	100	10	16	8.520
17	0	0	0	100	45	58	4.720
18	0	0	0	100	45	58	4.980
19	-	-	0	50	10	58	5.354
20	-	+	0	50	80	58	6.243
21	+	0	+	150	45	100	6.180
22	0	+	+	100	80	100	7.120
23	0	0	0	100	45	58	4.890
24	-	0	+	50	45	100	6.000
25	0	-	-	100	10	16	8.240
26	-	0	+	50	45	100	6.000
27	+	0	+	150	45	100	6.320
28	+	+	0	150	80	58	5.120
29	0	+	-	100	80	16	6.110
30	0	+	0	100	80	58	5.190



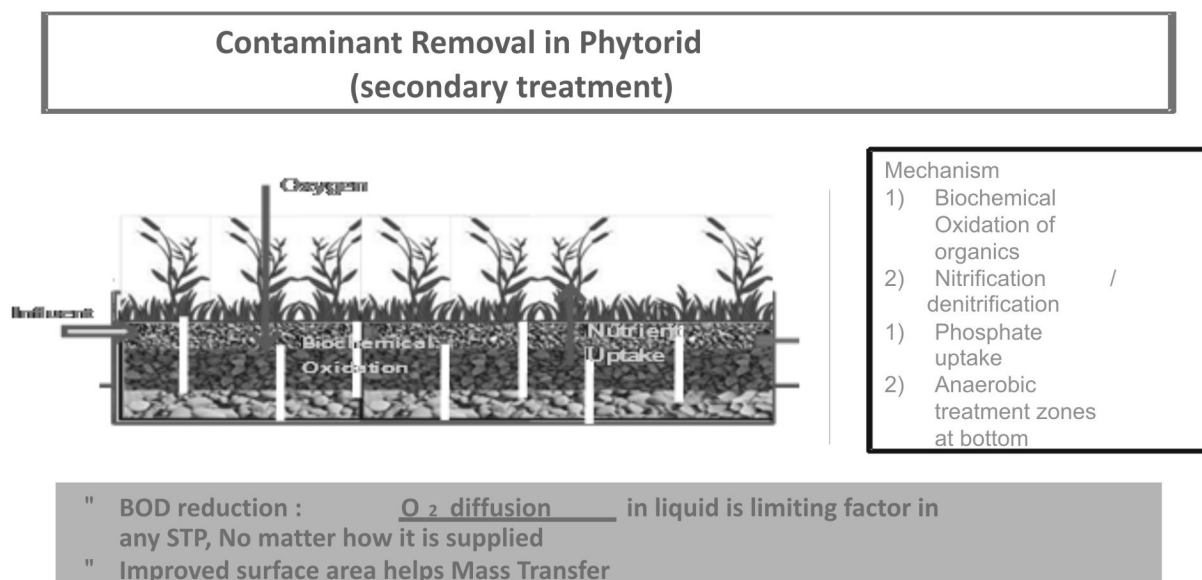
**Table 3. Analysis of variance (ANOVA) of the response surface quadratic model for the prediction of K**

Factor (Coded)	DF	Sum of	Mean square	F-	p-	Remark
Model	9	36.0562	4.0062	112.73	0.000	Significant
X <sub>1</sub>	1	0.0131	0.0131	0.37	0.551	Nonsignificant
X <sub>2</sub>	1	0.0142	0.0142	0.40	0.535	Nonsignificant
X <sub>3</sub>	1	5.7444	5.7444	161.65	0.000	Significant
X <sub>12</sub>	1	1.0249	1.9135	53.85	0.000	Significant
X <sub>22</sub>	1	1.0697	1.7797	50.08	0.000	Significant
X <sub>32</sub>	1	15.5003	15.5003	436.17	0.000	Significant
X <sub>1</sub> X <sub>2</sub>	1	3.3398	3.3398	93.98	0.000	Significant
X <sub>1</sub> X <sub>3</sub>	1	0.1800	0.1800	5.07	0.036	Significant
X <sub>2</sub> X <sub>3</sub>	1	9.1699	9.1699	258.04	0.000	Significant
Residual Error	20	0.7107	0.0355			
Lack-of-Fit	3	0.0187	0.0062	0.15	0.926	Nonsignificant
Pure Error	17	0.6920	0.0407			
Total	29	36.7670				

R-Sq = 98.07(%)      R-Sq (pred) = 95.51(%)      R-Sq(adj)=97.20%      CV =10.03 (%)

**Table 4. Experimental condition for model validation with corresponding predicted and observed responses**

Additional experiment	Flow( m <sup>3</sup> d <sup>-1</sup> )	Dilution (%)	Spatial (%)	Predicted K removal	Experimental K removal
1	150	45	16	7.16	7.42
2	100	70	16	6.32	6.451
3	50	80	40	6.60	6.788
4	90	10	100	4.88	4.96
5	140	10	58	6.06	6.16



**Fig. 1. Contaminant removal process in the phytorid sewage treatment plant (Source: NEERI)**

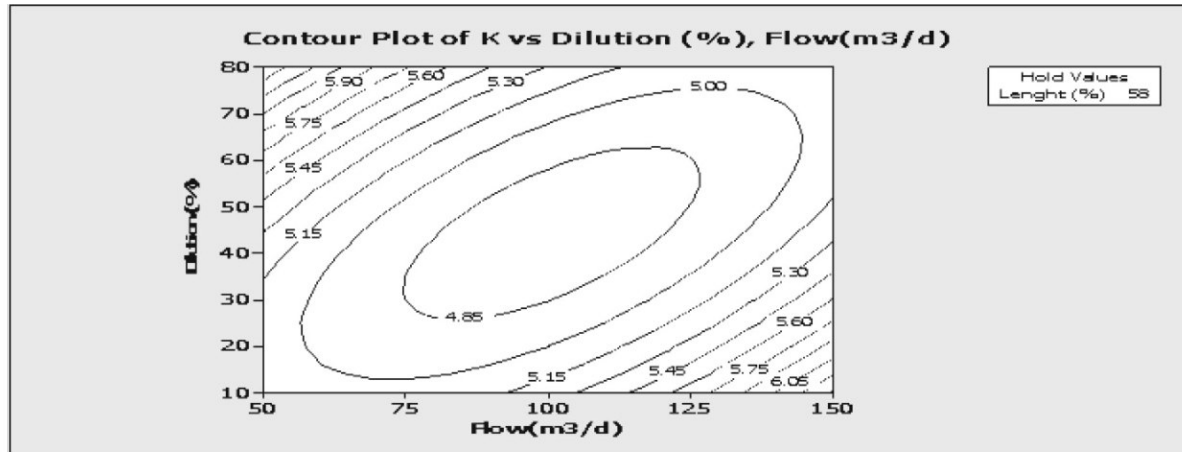


Fig.2(a). Contour plot showing effect of two independent variables  
(Length (%) was held at their respective (centre level)  
Dilution (%) and flow (m<sup>3</sup> d<sup>-1</sup>)

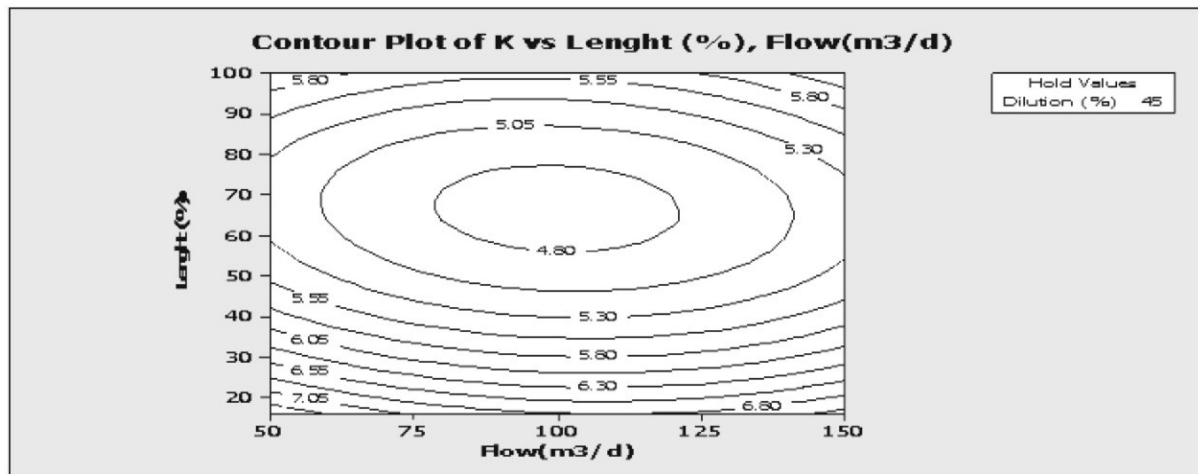


Fig.2(b). Contour plot showing effect of two independent variables  
(Dilution (%) was held at their respective centre level)  
Length (%) and flow (m<sup>3</sup> d<sup>-1</sup>)

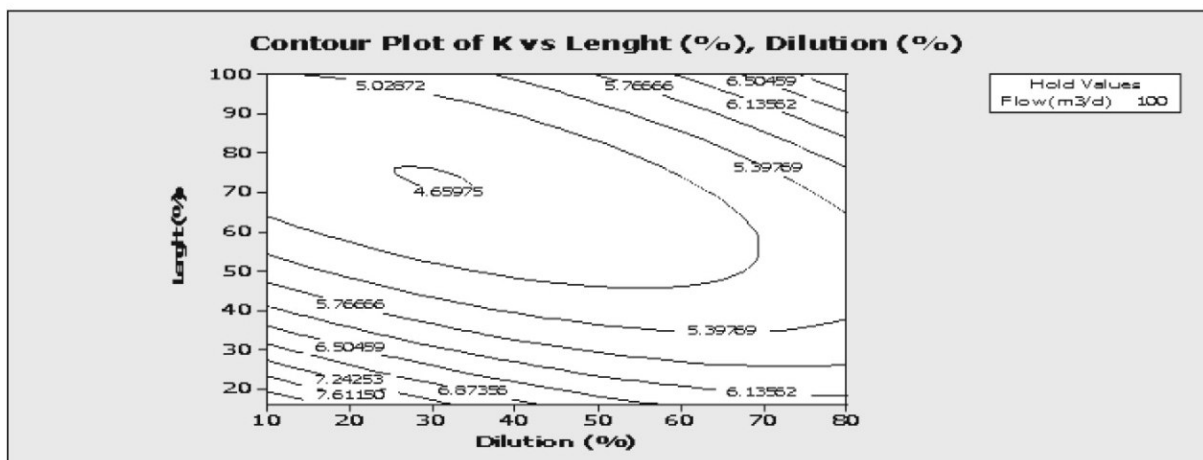


Fig.2(c). Contour plot showing effect of two independent variables  
(Flow was held at their respective centre level)  
Length (%) and dilution (%)

### Parameter

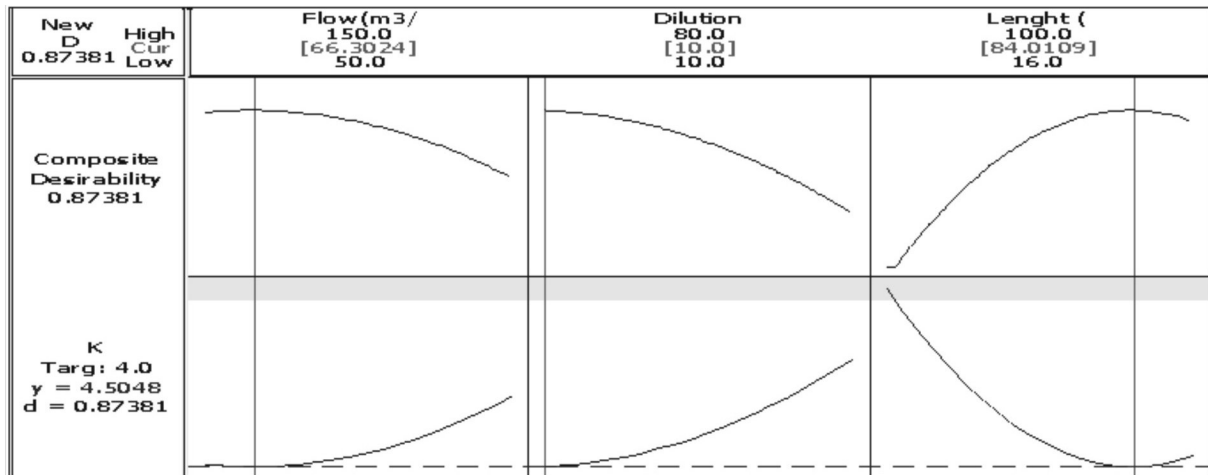


Fig.3. Contour plot showing the optimizing condition of the K

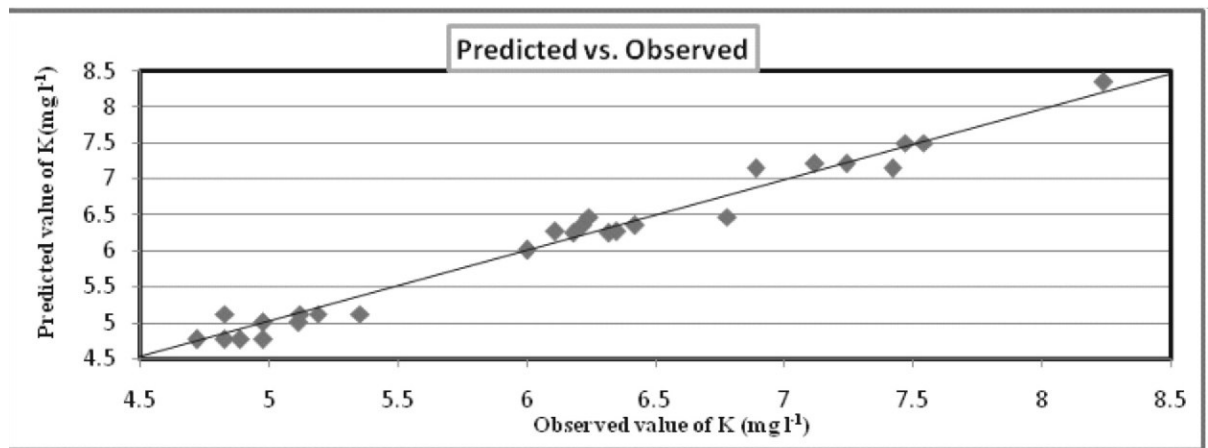


Fig.4. Plot showing the experimental observed values versus predicted values of the K

A similar pattern has been reported by others for the second-order RSM experiments based on Box-Benhken (Aslam and Cebeci, 2010) and central composite (Liu *et al.* 2004) designs. Further, a relatively low value of the coefficient of variation (CV=10.03 %) indicates good precision and reliability of the conducted experiments as similar to earlier reported by Ahmad *et al.* (2005) in optimization of turbidity removal.

#### Optimization of experimental condition K removal

In order to observe the better understanding of the influence of the independent variables and their interactions on the dependent variable, response contour plots for the measured responses were drawn based on the quadratic model as suggested by Yetilmesoy *et al.* (2009). Fig.2 exhibits the response contour plots as the functions of two independent variables keeping other variable fixed at the centre level. It can be seen from Fig. 2(a) that the concentration of K was observed increased with increasing the hydraulic loading of sewage might be due to the higher K concentration in sewage.

Similarly K concentration was found increased due to increase in the dilution which might be due to K bounded in organic matter was released during the decomposition in oxidation process for which oxygen was provided by fresh water. Absolute value increase in 'K' as observed in the contour plot was therefore increased. But overall there is reduction in K concentration.

From Fig. 2(b) it is evident that the concentration of K found decreasing from 7.55 mg l<sup>-1</sup> to 4.80 mg l<sup>-1</sup> with increasing length from 50 to 70 per cent. This might be due to the uptake of the K by the phytoplants during passing of sewage water through the treatment plant after 70 percent length the concentration of K was found slightly increased in the plant due to decomposition of organic matter through oxidation process by addition of oxygen of atmosphere from phyto plants through the roots. There was no significant trend in decrease of concentration of K with increase in flow in the treatment plant from 50 m<sup>3</sup> d<sup>-1</sup> to 100 m<sup>3</sup> d<sup>-1</sup> might be due to higher K concentration in sewage. Whereas the concentration of K was found slightly increased when hydraulic loading increased from 100 mg l<sup>-1</sup> to 150 mg l<sup>-1</sup>.

The release quantity of K was very small therefore did not contributed to overall concentration of P in treated water and thereby the absolute value increase in P as observed in the contour plot.

As depicted in Fig. 2 (c) it is clearly observed that the concentration of K was decreased from 7.61 mg l<sup>-1</sup> to 4.66 mg l<sup>-1</sup> with increasing spatial length from 10 to 80 per cent. This decrease in concentration of K along the length of treatment plant might be due to uptake of K by the phyto plants while passing the sewage water through the treatment plant. Similarly it is also evident that P concentration was decreased slightly due dilution might be due to adding of fresh water of lower concentration of K in sewage water.

Therefore, it is concluded from the Fig 2(a), 2(b) and 2(c) that the range of variable for the significant K removal was dilution > 10 per cent, the spatial length more than 20 per cent and flow at 100 m<sup>3</sup> d<sup>-1</sup> play significant role in removal of K (Mohamed, 2005).

### Response optimization

The optimization of K using the response optimizer was carried out for independent variable for the value of K as 100 m<sup>3</sup> d<sup>-1</sup> as presented in Fig. 3. The optimized global solution for independent variable found was 65.15 m<sup>3</sup> d<sup>-1</sup> of flow, 10.00 per cent dilution and 83.88 per cent spatial length with composite desirability=1.00. Similar results were reported by Khajeh (2011) for extraction time with various parameters such as pressure, temperature and percent of modifier (methanol).

### Global Solution

Goal	Lower	Target	Upper	Weight	Import
K Target	0	4	8	1	1

### Global Solution

Flow (m<sup>3</sup> d<sup>-1</sup>) = 65.15

Dilution (%) = 10.00

Length (%) = 83.88

### Predicted Responses

K = 4.50 ,

desirability = 0.874

Composite Desirability = 0.874

### Predicted vs. Observed

The diagnostic plot shown in Fig.4 indicated the experimental and the predicted K concentration removal values and was used to estimate the adequacy of the regression model. Observed and predicted values of K removal were observed in well agreement. The points cluster around the diagonal line indicated a good fit of the model as earlier reported by Ahmad *et al.* (2005) in optimizing the condition for K parameter.

### Validation of the data

In order to verify the validity of the proposed model, additional five experiment were conducted for different combination of the three independent process variables in a random fashion (Wu *et al.*, 2009) each within its respective experimental range and corresponding response variable (K concentration removal) was generated using the uncoded variables and model equation (2). Table 4 presents the experimental condition along with the model

predicted and experimental results. Experimentally determined response factor values for each of the five sets of process variables were then used along with the model predicted values to compute the R<sup>2</sup> values. A correlation (R<sup>2</sup>= 0.984) among the predicted and measured values of the response values of the response factor suggest for the adequacy of the proposed quadratic model in predicting the response variable for the validation data set comprised of different combination of the process variables (Korbhati and Rouf, 2008).

Application of response surface methodology and Box–Behnken design from the point of view K concentration removal from sewage water is discussed. Three-level three-factorial Box–Behnken experimental design was applied in the study. Predicted values of K concentration removal obtained using model equations were in good agreement with the experimental values (R<sup>2</sup> - 0.98). In order to gain a better understanding of the effect of the variables on K removal, the predicted models were presented as contour graphs. This study proved that Box– Behnken design and response surface methodology could economically and efficiently be applied for modelling of some pollutant concentration removal from the phytoid sewage treatment plant.

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