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Abstract: An artificial neural network (ANN) model was developed to predict the removal efficiency of chromium (VI) from aqueous solution using a Borasus flabellifer coir powder as adsorbent. The effect of operational parameters such as pH, adsorbent dosage, and initial chromium (VI) concentration are studied to optimize the conditions for the maximum removal of chromium (VI) ions. The ANN model was developed using 54 experimental data points for training and 16 data points for testing by a single layer feed forward back propagation network with 18 neurons to obtain minimum mean squared error (MSE). A tansigmoid was used as transfer function for input and purelin for output layers. The high correlation coefficient ($R^2_{average-ANN}$ =0.992) between the model and the experimental data showed that the model was able to predict the removal of chromium (VI) from aqueous solution using Borasus flabellifer coir powder efficiently. Pattern search method in genetic algorithm was applied to get optimum values of input parameters for the maximum removal of chromium (VI).

Keywords: Biosorption; borasus flabellifer coir powder; chromium (VI).

1. Introduction

Water contamination with heavy metals is a very severe problem all over world [1, 2]. The world production of chromite ore is several millions of tons in a year. Ferrochromite is obtained by direct reduction of the ore while chromium metal is produced either by chemical reduction (the aluminothermic process) or by electrolysis of either CrO_3 or chrome alum solutions. Chromium and its compounds are extensively used in metal finishing, leather tanning, electroplating, textile industries, and chromate preparation [3]. In aqueous phase chromium mostly exists in two oxidation states, namely, trivalent chromium [Cr⁺³ and Cr(OH)²⁺] and hexavalent chromium (HCrO₄⁻, CrO₄²⁻ or Cr₂O₇²⁻, etc). Most of the hexavalent compounds are toxic, carcinogenic and mutagenic. For example, it was reported that Cr₂O₇²⁻ can cause lung cancer [4, 5].

Chromium (III) and Chromium (VI) have major environmental significance because of their stability in the natural environment. Cr (VI) is known to have 100 fold more toxicity than Cr (III) because of its high water solubility and mobility as well as easy reduction [6]. International agency for research on cancer has determined that Cr (VI) is carcinogenic to humans. The

* Corresponding author; e-mail: <u>d_krishna76@rediffmail.com</u>	Received 28 October 2013
© 2014 Chaoyang University of Technology, ISSN 1727-2394	Accepted 28 April 2014

toxicological effect of Cr (VI) originates due to oxidizing nature as well as the formation of free radicals during the reduction of Cr (VI) to Cr (III) occurring inside the cell [8]. Therefore, the World Health Organization (WHO) recommends that the toxic limits of chromium (VI) in waste water at the level of 0.05 mg/l, while total Chromium containing Cr (III), Cr (VI) and other species of chromium is regulated to be discharged below 2 mg/l [7].

Several methods are used to remove chromium from the industrial wastewater. These include reduction followed by chemical precipitation [9], ion exchange [10], reduction [11], electrochemical precipitation [12], solvent extraction [13], membrane separation [14], evaporation [15] and foam separation [16]. Above cited conventional chromium elimination processes are costly or ineffective at small concentrations. In recent years biosorption research is focused on using readily available biomass that can remove heavy metals. This process involves the use of biological materials that form complexes with metal ions using their ligands or functional groups. This process can be applied as a cost effective way of purifying industrial waste water whereby drinking water quality can be attained. A lot of research was focused on bio-adsorbent materials which can efficiently remove heavy metals from aqueous bodies. These materials are identified as biosorbents and the binding of metals by biomass is referred to as biosorption. Since this noble approach is effective and cheap many researchers exploring appropriate biomaterials that effectively remove Cr (VI) from aqueous solutions [17, 18]. A variety of adsorbents like tamarind seeds [19], rice husk [20], azadirachta indica [21], maize bran [22], red saw dust [23], wall nut hull [24], groundnut hull [25], limonia acidissima hull powder [26] were reported in literature for removal of chromium from aqueous solutions or waste waters in a batch or column reactor system. Broad range of biosorbents can collect most of the metal ions from the solution and a certain concentration of a specific metal could be achieved either during the sorption uptake by manipulating the properties of a biosorbent, or upon desorption during the regeration cycle of these biosorbent [27].

The mechanism of biosorption is highly complex and is difficult to model and simulate using conventional mathematical modeling. This is mainly due to interaction of more number of sorption process variables, and hence the resulting relationships are highly non linear [27]. To achieve an optimum management for any control measure, the concept of modeling for an efficient operation and design should be developed. A high quality representative model can provide a favorable solution to optimize the process input parameters. Because of reliable, robust and salient characteristics in capturing the non-linear relationships of variables in complex systems, application of Artificial Neural Network (ANN) has been success fully employed in environmental engineering [28-30]. The most common applications are function approximation, pattern recognition and classification. There is no exact formula available to decide what architecture of ANN and which training algorithm will solve a given problem. The best solution is obtained by trial and error method [39]. One can get an idea by looking at a problem and deciding to start with simple network, trying more complex ones till the solution is within the acceptable limits of error.

The main unit of any ANN is an artificial neuron, which has two parts, one is the summing part and the other is multiplied with a weight parameter and all weighted inputs are summed up. The output of summed value is passed on to the second part, which is the logic part. This logic part is a non-linear function and the power of the ANN lies in this logic part. The intelligent way in which the neurons are prearranged and in which the interconnections are made, results in better problem-solving capability [39]. Once the network had been trained for the satisfactory error goal, the weights and biases were fixed for model. At this stage, the model had been trained and ready for validation using the unseen dataset, i.e., the test data that had not been used for

training was used for this purpose. The simulated network was used to recall the learned pattern on the test data. For this purpose, curve fitted data of the input variables were used [39].

There are numerous known types of neural networks performing various tasks. Based on the feed signals direction the following neural networks types can be distinguished: Feed forward neural network examples single layer perceptron, Multilayer perceptron, Back propagation, Levenberg-Marquardt technique, radial basis function network and fuzzy logic network; recurrent network examples Elman network, Hopfield network and Jordan networks. Most popular is the use of feed forward back propagation in a layered system which was used in current system.

The Levenberg-Marquardt algorithm was designed to approach the second-order training speed. The Levenberg-Marquardt algorithm was designed to approach the second-order training speed and updates weight and bias values. Bayesian regularization backpropagation is a network training function that updates the weight and bias values according to the Levenberg-Marquardt optimization. It minimizes a combination of squared errors and weights and then determines the correct combination so as to produce a network that generalizes well. Examples of supervised learning algorithms include the least-mean-square (LMS) algorithm and its generalization known as the backpropagation (BP) algorithm. The back-propagated through the network on a layer-by-layer basis. Recurrent Networks differ from feed forward network architectures in the sense that there is at least one feedback loop.

The most widely used neural network is back propagation (BP). BP attempts to minimize error by adjusting each value of a network proportional to the derivative of the error with respect to that value. This is called the gradient descent. In the BP learning, the actual outputs are compared with the target values to derive the error signals, which are propagated backward, layer by layer, for the updating of the synaptic weights in all the lower layers [39]. ANN is known for their superior ability to learn and classify data. The inspiration of Neural Network came from studies on the structure as function of the brain and nerve systems as well as the mechanism of learning and responding. The objective of the network is to compute output values from input values by some internal calculations. Table 1 gives the application of artificial neural network for the removal of heavy metals by different bisorbents in separation technology.

S.No.	Type of metal	Adsorbent	Objective of the work	ANN Type	References
1.	Cr (VI)	Brown seaweed, Ecklonia biomass	To study the reduction of Cr (VI) by potential use of the brown seaweed, Ecklonia biomass as a bioreductant in a continuous packed bed column. For process application, a non-parametric model using neural network was used to predict the breakthrough curves of the column.	FFNN ^a -MLP ^b ⁺ -SL ^c -BP ^d	[31]

 Table 1. ANN applications for the removal of heavy metals in separation technology

2.	Cr (VI) & Cr (III)	Zea mays	Artificial neural network was applied to sorption batch studies to develop & validate a model that can predict Cr (III) & Cr (VI)removal efficiency.	FFNN- single layer-SL-BP- LMT ^e	[32]
3.	Cr (VI)	Electrocoag ulation	To study the removal of Cr (VI) from synthetic & real waste water using electrocoagulation. ANN was used for modeling of experimental results.	FFNN-MLP- SL-BP	[33]
4.	Cu (II)	Saw dust	To study the use of saw dust as biosorbent for the removal of Cu (II) from aqueous solution. To apply ANN based on multilayered partial recurrent back-propagation algorithm for the prediction of percentage adsorption efficiency for the removal of Cu (II) ion from aqueous solution by saw dust.	RN ^f -EN ^g -SL- BP	[34]
5.	Cu (II)	Sunflower shells	To study the adsorption potential of shells of sunflower to remove Cu ²⁺ ions from aqueous solution using a fixed bed adsorption column. To study the effects of inlet concentration of Cu (II), feed flow rate, bed height, solution pH & particle size on break through characteristics of adsorption systems.	FFNN-single layer-SL-BP	[35]
6.	Pb (II)	Electrodial ysis	To predict separation percent of Pb ²⁺ ions as a function of concentration, temperature, flow rate & voltage.	FFNN-MLP- SL-BP-LMT	[36]

7.	Pb (II)	Red mud	To develop the prediction models for lead removal from industrial sludge leachate.	FFNN-single layer-SL-BP- LMT	[37]
8.	Zn (II)	Miscellar-e nhanced ultrafiltrati on	To study the removal of zinc ions from waste water efficiently.	FFNN-Single layer-SL-BP- LMT	[38]
9.	Zn (II)	Hazelnut shell	To predict the percentage adsorption efficiency using ANN model based classification technique for the removal of Zn (II) ions from leachate by hazelnut shell.	FFNN-MLP- SL-BP-LMT	[39]
10.	Cd (II)	Shelled moringa oleifera seed (SMOS)	To predict the removal efficiency of Cd (II) ions from aqueous solution using SMOS powder by a two layer artificial neural network model.	FFNN-MLP- SL-BP-LMT	[40]
11.	Cd (II)	Saraca indica leaf powder (SILF)	To predict the removal efficiency of Cd (II) ions from aqueous solution using SILP powder by a single layer ANN model	FFNN-Single layer-SL-BP- LMT	[41]
12.	Pb (II)	Antep pistachio shells	To predict the removal efficiency of Pb(II) ions from aqueous solution by Antep pistachio shells based on 66 experimental sets obtained in a batch study by a three layer artificial neural network model.	FFNN-MLP- SL-BP-LMT	[42]
13.	Ni (II)	Shelled moringa oleifera seed (SMOS)	To predict the removal of efficiency of Ni(II) ions from aqueous solution using SMOS powder by a single layer ANN model.	FNN-Single layer-SL-BP- LMT	[43]
14.	Cd (II & Zn (II)	Sargassum filipendula	To study the use of ANN model to fit the equilibrium experimental data.	Simplex-opti mization-met hod	[44]

15.	Cu (II), Zn (II) & Ni (II)	Clinoptiloli te	To study the estimation of sorptivity of a clinoptilolite & its selectivity towards Cu (II), Zn (II) & Ni (II) ions for multicomponent solution by means of a multilayer perceptron (MLP).	FFNN-MLP- SL-BP-LMT	[45]
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^a Feed forward neural network, ^b Multilayer perceptron, ^c Supervised learning, ^d Back propagation, ^e Levenberg-Marquardt technique, ^f Recurrent network, ^g Elman network

A genetic algorithm (GA) is a search technique mimicking natural selection [46]. The algorithm evolves until it satisfactorily solves the problem, through the fitter solutions in a population surviving and passing their traits to offspring which replace the poorer solutions. Each potential solution is encoded, for example as a binary string, called a chromosome. Successive populations are known as generations. The initial population G (0) is generated randomly. Thereafter G (t) produces G (t+1) through selection and reproduction [46]. A proportion of the population is selected to breed and produce new chromosomes. Selection is according to fitness of individual solutions, i.e., proximity to a perfect solution [47], most often by roulette selection and deterministic sampling.

In the present investigation, batch experimental studies [48] were carried out for the removal of chromium (VI) from aqueous solution using Borasus flabellifer coir powder. The experimental data was used to train ANN model with 18 neurons in hidden layer with feed forward back propagation algorithm. Later the model is tested for validation of the experimental data which is not used for ANN model. The input parameters for the percentage removal of chromium (VI) are initial chromium (VI) concentration, adsorbent dosage and pH. The process optimization has been carried out using genetic algorithm to optimize the input parameters to the process for maximum chromium (VI) removal.

2. Materials and methods

2.1. The diphenyl carbazide method

A 0.25% W/V solution of diphenyl carbozide was prepared in 50% acetone. 15 ml each of the sample solutions containing various concentrations of Cr (VI) were pipette out into 25 ml standard flasks. To this 2 ml of 3M H_2SO_4 was added followed by 1 ml of diphenyl carbozide and total volume was made up to 25 ml using deionised, double distilled water. Chromium (VI) concentrations estimated by the intensity of the red brownish color complex formed, was measured using U-V-Visible spectrophotometer at 540 nm. The absorbance was measured indicating adherence to the Beer Lambert's law (0 to 30 mg/l).

2.2. Preparation of the adsorbent

The Borasus flabellifer coir was obtained from local market; washed, dried, and crushed in primary crusher and air dried in sun for several days until its weight remains constant. After drying, it is crushed in roll crusher and hammer mills. The material obtained through crushing

and grinding is screened through BSS meshes. Finally the products obtained were stored in glass bottles for further use. All the materials were used as such and no pretreatment was given to the materials. The average particle sizes were maintained in the range of 63 to 125 μ m.

2.3. Preparation of chromium stock solution

Potassium dichromate ($K_2Cr_2O_7$) is used as the source for chromium stock solution. All the required solutions are prepared with analytical reagents and double-distilled water. 2.835 g of 99% $K_2Cr_2O_7$ is dissolved in distilled water of 1.0L volumetric flask up to the mark to obtain 1000 ppm (mg/l) of chromium (VI) stock solution. Synthetic samples of different concentrations of chromium (VI) are prepared from this stock solution by appropriate dilutions. 100 mg/l chromium stock solution is prepared by diluting 100 ml of 1000 mg/l chromium stock solution with distilled water in 1000ml volumetric flask up to the mark. Similarly solutions with different metal concentrations such as (5, 10, 15, 20, 25 and 30 mg/l) are prepared.

Crequivalent to
$$l gm = \frac{Molecular Wt. of K_2 Cr_2 O_7 \times 100}{(Atomic Wt. of Cr \times 2) \times purity}$$
(1)

2.4. Batch mode adsorption studies [48]

Batch mode adsorption studies for individual metal compounds were carried out to investigate the effect of different parameters such as adsorbate concentration (5-30 mg/l), adsorbent dosage (0.1-0.7 gm in 50 ml solution), agitation time (0-120 min), pH (1-10), adsorbent size (63 μ m, 89 μ m and 125 μ m) and temperature (303-323K). The solution containing adsorbate and adsorbent was taken in 250 ml capacity conical flasks and agitated at 180 rpm in a mechanical shaker at predetermined time intervals. The adsorbate was decanted and separated from the adsorbent using filter paper (Whatman No-1).

2.5. Metal analysis

Final residual metal concentration after adsorption was measured by UV-Spectrophotometer after sample was complexed with 1-5 Diphenyl carbazide. To estimate the percentage removal of chromium (VI) from aqueous solution, the following equation was used.

Percentage removal of Cr (VI) =
$$\frac{C_0 - C_e}{C_0} \times 100$$
 (2)

The metal uptake (qe) at equilibrium time was calculated from the following equation

$$q_{e} = \frac{(C_{0} - C_{e})v}{1000w}$$
(3)

where $q_e (mg/g)$ is the amount of chromium adsorbed per unit weight of adsorbent, C_0 and C_e are the initial and equilibrium chromium ion concentration (mg/l), v is the volume of aqueous solution (ml), and w is the adsorbent weight (g).

3. Results and discussions

3.1. Characterization of borasus flabellifer coir powder

The scanning electron micrographs (SEM) of Borasus flabellifer coir powder before chromium (VI) adsorption and after chromium (VI) adsorption are shown in Figure 1 and Figure 2. The surface morphology revealed that Borasus flabellifer coir powder was found to be irregular and porous and thus would facilitate the adsorption of metal ions on different parts of the Borasus flabellifer coir powder. The SEM micrographs showed that pores with different sizes and different shapes existed on external surface of Borasus flabellifer coir powder. The micrograph of Borasus flabellifer coir powder after chromium (VI) adsorption shows a reduction of number of pores, pore space and surface area available (refer to Figure 2). Hence it is confirmed that there is metal adsorption on the surface of adsorbent. Furthermore, the EDS spectra of selected zone of Borasus flabellifer coir powder before adsorption and after adsorption was carried out to investigate the chemical constituents in the carbon matrix (refer to Figure 3 (a) and Figure 3 (b)). It has been found from Figure 3 (a) that Borasus flabellifer coir powder having the carbon, oxygen on its surface before interaction with Cr (VI) ions, whereas in Figure 3 (b), new chromium peak was observed with the surface bearing groups of carbon and oxygen, which confirmed the Cr (VI) adsorption on Borasus flabellifer coir powder.



20µm

Figure 1. SEM images of borasus flabellifer coir powder before chromium (VI) adsorption





Figure 2. SEM images of borasus flabellifer coir powder after chromium (VI) adsorption





Figure 3. Energy-disperse spectra of borasus flabellifer coir powder (a) before chromium (VI) adsorption; (b) after chromium (VI) adsorption

3.2. ANN model

In this work, multilayer feed-forward ANN with one hidden layer was used. ANN model optimized structure is shown in Figure 4. For all data sets, sigmoid transfer function in the hidden layer and a linear transfer function in the output node were used. The ANN was trained using backpropagation algorithm. All calculations were carried out with matlab software. Initial concentration of Chromium (VI) (over range of 5-30 ppm), pH (over range 1-3) and biomass dosage (over range of 8-14 g/l) were used as inputs of ANN model. The percentage removal of chromium (VI) was the experimental response or output variable. Total 54 experimental points were split randomly between training and test sets. For training 38 data points were used and 16 data points for testing were used.

The topology of an ANN is determined by the number of layers, the number of nodes in each layer and the nature of transfer functions, correct identification of the set of independent input

D. Krishna and R. Padma Sree

variables and the output variables is the first task in the building ANN model. Optimization of ANN topology is the next important step in the development of a model. The number of neurons (N) in the hidden layer is determined according to the minimum prediction of error of the neural network. Hence it may be considered as a parameter for the neural network design. In order to determine the optimum number of neurons in the hidden layer, different topologies were examined, in which the number of nodes was varied from 2 to 20. Each topology was repeated three times. The MSE was used as the error function. It measures the performance of the network according to the following equation.

$$MSE = \frac{1}{N} \sum_{i=1}^{i=N} (y_{i,pred} - y_{i,exp})^2$$
(4)

where N is the number of data points, $y_{i,pred}$ is the network prediction $y_{i,exp}$ is the experimental response and i is an index of data.

It could be seen that the network mean square error (MSE) is minimum with inclusion of eighteen nodes in the hidden layer. So, based on the approximation of MSE function, a number of hidden neurons equal to eighteen was adopted and a single layer feed forward backpropagation neural network was used for the modeling of the process. Figure 5 illustrates the network error versus the number of neurons in the hidden layer.



Figure 4. ANN optimized structure



Figure 5. Variation of MSE versus number of neurons in hidden layer

The weights and bias values are provided by ANN listed in Table 2. The network was evaluated by comparing its predicted output values with experimental ones using an independent set of data (test data). The plot of experimental results (test data) versus the predicted ones is shown in Figure 6. It shows that the points are well distributed around X=Y line in a narrow area. A correlation coefficient of $R^2=0.992$ for the line plotted using experimental and calculated data, indicates the reliability of the model. Comparison of experimental and estimated removal efficiency on testing data is shown in Table 3. It shows that experimental removal efficiency values and ANN model values are almost equal with low relative error as well low relative percentage error. Finally it is concluded that present ANN model is suited to model for to the removal of chromium (VI) from aqueous solution by using Borasus flabellifer coir powder with 3 input variables (pH, biomass dosage and initial concentration of chromium (VI)).For almost all experiments, the ANN was confirmed to be an adequate interpolation tool, where good prediction was obtained.

ANN modeling technique was found out to have many favorable features such as efficiency, generalization and simplicity, which make it an attractive choice for modeling of complex systems, such as wastewater treatment processes.



Figure 6. Parity plot showing the distribution of experimental versus predicted values of percentage removal of chromium (VI)

W1	(18×3 mat	rix)	W2 (1×18 vector)	b1 (18×1 vector)	b2 (scalar)
-0.0515	0.3188	-0.1708		-7.3284	
0.5949	5.7510	2.4711		-2.9584	
0.3172	0.3154	0.2003		-6.8912	
-5.6896	0.1749	-5.6795		-0.0281	
-3.8895	0.6487	2.4867		1.8634	
1.1195	0.3546	0.5042	[-15.5504, -8.173,	-7.5143	
2.9884	-0.4954	-1.6901	-10.7926, 6.5048,	1.7094	
-0.7398	-4.4804	-2.4238	6.9756, -5.3865,	2.3128	
-0.0093	-3.4553	-0.0278	-2.3501, -4.4462,	-0.4684	18 9716
2.0771	0.4673	-1.1207	-2.2771,-2.1815,	5.0642	10.7710
-0.1146	-0.0655	-4.1055	2.5729, 0.0078,	2.9884	
5.0698	-1.3073	2.1551	-4.2752, -5.2709,	-2.6085	
-5.8688	0.9995	3.8034	-9.9035, 10.3184, 6 5044 12 04951	2.6718	
-1.2704	1.4153	-1.9382	0.5044, 12.0455]	-4.9267	
0.3378	-0.6145	-0.0673		-7.6853	
-0.0702	0.8351	-1.4553		5.6574	
6.3747	-0.0887	6.6508		0.0133	
0.1178	-0.3817	-0.0802		7.2021	

Table 2. Weights and biases of the neural networks

 Table 3.
 Comparison of experimental and estimated removal efficiency on testing data (number of epochs=6000)

Run	Initial concentration of Chromium (VI), mg/l	рН	Biomass loading, g/l	Experimental % efficiency	Simulated % efficiency	Relativ e error	Percentag e relative error
1	15	2	14	92.47	93.23	-0.7592	-0.8143
2	15	3	12	89.25	90.54	-1.2858	-1.4407
3	25	1	12	87.75	87.83	-0.0791	-0.0902
4	25	3	12	85.86	86.14	-0.2846	-0.3261
5	15	2	10	93.86	93.51	0.353	0.3761
6	15	1	12	91.15	90.98	0.1702	0.1867
7	25	2	10	90.45	89.24	1.212	1.3399
8	25	2	14	87.04	88.54	-1.4993	-1.7226
9	20	1	10	88.18	86.71	1.469	1.6659
10	20	1	14	86.79	86.24	0.5461	0.6292
11	20	3	10	86.29	85.75	0.5361	0.6212
12	20	3	14	84.91	84.87	0.0421	0.0496
13	20	2	12	93.43	93.68	-0.2487	0.2662
14	20	2	8	89.74	88.95	0.7916	0.8821
15	15	3	8	85.56	85.87	-0.3137	0.3667
16	25	3	8	82.16	81.82	0.3404	0.4143

3.3. Optimization using genetic algorithm

GA was used to optimize the process parameters for the removal of chromium (VI) from aqueous solution by using Borasus flabellifer coir powder as adsorbent. The Optimum values of variables obtained from pattern search method (Genetic algorithm) for the removal of chromium (VI) are (1) initial concentration of chromium (VI)-5 mg/l (2) biomass dosage-11.2634 g/l (3) pH-2.2621. The residual sum of squares is 0.2553. With these optimum input parameters, the maximum percentage removal of chromium (VI) is 99.0777.

4. Conclusions

A detailed batch experimental study was carried out for the removal of chromium (VI) from aqueous solution by using Borasus flabellifer coir powder. ANN model was employed for modeling of removal of chromium (VI) from aqueous solutions by Borasus flabellifer coir powder. A multilayer network (FFNN-MLP) with one hidden layers (3: 18: 1: 1) was applied to predict percentage removal of chromium (VI) from aqueous solution with the minimum MSE. Back propagation algorithm was used to train the network. The model and the test data are in perfect match with R^2 value of 0.992. ANN successfully tracked the non-linear behavior of percentage removal of chromium (VI) versus the initial concentration of chromium (VI), pH and biomass dosage with low relative percentage error.

A genetic algorithm with pattern search solver is used to optimize the ANN model developed. The optimization results in the following input parameters are pH-2.2621, biomass dosage-11.2634 g/l and initial concentration of chromium (VI)-5 mg/l.

Further, research is to be carried out to make the process economically viable at industrial scale with focus on Cr (VI) removal and regeneration of Borasus flabellifer coir powder by using optimum input parameters for getting maximum yield.

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