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Column study for chloride removal from waste water by a low cost adsorbent (bio adsorbent)

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Abstract: - In this article, adsorption process has been found effective for removal of chloride. Water pollution and its control has become an important issue to deal with. The other filtration method of water treatment is expensive and not feasible. The requirement of low cost natural bio filter is being studied for the removal of water pollutants. Specifically for chloride removal the use of Bio adsorption is found to be very effective. The Parthenium has been extensively used for removal of chloride either by using batch mode adsorption or by column study. Here in this article, the removal of chloride has been studied by using column method.

Key words: - **Chloride, Bio-adsorption, Parthenium, Column Study.**

I. INTRODUCTION

The removal of chloride from water by Parthenium using fixed-bed column adsorption techniques was investigated. Fixed-bed column experiments were carried out for different influent chloride concentrations, bed depths, and various flow rates. The breakthrough time and exhaustion time decreased with increasing flow rate, decreasing bed depth and increasing influent chloride concentration. The bed depth service time model and the Thomas model were applied to the experimental results. Both model predictions were in good agreement with the experimental data for all the process parameters studied, indicating that the models were suitable up to some extent for parthenium fix-bed column design. Chloride is a salt compound resulting from the combination of chloride salts includes Sodium Chloride (NaCl), and Magnesium Chloride ($MgCl_2$). The chloride ion is negatively charge anion (Cl^-). It is formed when the elements chlorine gains an electrons or when a compound such as hydrogen chloride is dissolved in water or other polar solvent. Chloride salts such as NaCl are very soluble in water. It is an essential electrolyte located in all body fluids responsible for maintaining acid/base balance, transmitting nerve impulses and regulating fluid in and out of cells. The word chloride can also form part of the name of chemical compounds in which one or more chlorine atoms are covalently bonded. For example, methyl chloride, more commonly called chloromethane, (CH_3Cl) is an organic compound with a covalent C-Cl bond. It is not a source of chloride ion. Chloride in concentration above 600mg/l tends to give water a salty taste. The concentration of chloride content above 200mg/l is considered objectionable. Presence of high quality of chloride content in water resources indicates pollution due to human and industrial wastes and also from the earthen rocks in the subsurface. High chloride concentrations in freshwater can harm aquatic organisms by interfering with osmoregulation, the biological process by which they maintain the proper concentration of salt and other solutes in their bodily fluids. Difficulty with osmo regulation can hinder survival, growth, and reproduction.

II. LITERATURE REVIEW

The Bio adsorbents cover small plants, shrubs and trees etc. Low cost materials prepared from Bio- wastes such as grass, plants, shrubs, fruits and vegetable peels, nuts, shells, pulps, stones, barks, roots, leaves, fruit wastes and sawdust of certain timber trees have gained immense popularity because of their good metal removal efficiency and ease of operation in low cost. Bio- waste was studied in removal of Hg by chemically modified cotton (Roberts and Rowland, 1973), removal of lead ions using soymida fabrifuga bark (Banker and Dara 1985). Cr removal by rice husk carbon was studied by Srinivasan et al. (1988), Manju and Aniradhan (1990) used coconut fibre pith based pseudo activated carbon for Cr (VI) removal. Removal of Ni from aqueous solutions was studied using an agricultural waste peanut hulls (Periasamy and Namasivayam, 1995). The use of

Bio waste based adsorbents became common in late 1990s and lot of materials have been studied so far, for example, sunflower plant dry powder for removal of heavy metals (Bhalke et al., 1999), eucalyptus globulus bark for removal of Ni and Cu (Dolultani et al., 1999), orange peel for Ni removal (Ajmal et al.,2000), coirpith activated carbon in Cd, Ni and Cu removal (Kadirvelu, 2001), hazelnut shell activated carbon in Ni removal (Demirbas et al., 2002), rice husk in Cr removal (Tang et al., 2003), Jack fruit in Cd removal (Inbaraj and Sulochana, 2004), black gram husk in Pb removal (Saeed et al., 2005), Coconut copra meal in Cd (Ofomaja and Ho ys, 2006), ficus religiosa leaves in Pb and Cr removal (Qaiser etal., 2007), pretreate arca shell biomass in removal of heavy metals and radionuclie (Dahiya et al.,2008), Rice husk ash in Pb removal (Naiya et al., 2009), Bamboo activated carbon in Pb removal (Lalhruaitluanga et al., 2010), onion skin in Pb removal (Saka et al., 2011), Pinus tree bark in Pb removal (Affonso et al., 2012),Aliathus excels tree bark in Pb removal (Waghmare and Chaudhary, 2013), Neem bark and potato peels in removal of Malachite green (Sharma et al., 2014), Chondruscrispus powder for removal of chromium ions (Elavarasan, 2015). Ashtikar and Parkhi (2014) studied removal of Cu (II) from aqueous solution using mango seed powder as low cost adsorbent. The results showed that the adsorption was rapid during first 45 minutes & equilibrium was reached in 90 minutes. With increase in the adsorbent doses & temperature percent removal of copper increases. Sethu et al. (2010) studied adsorption of Cu(II) on mango leaf bio-sorbent. Results showed that the maximum adsorption capacity was 206.85 mg/g at 100 mg/L Cu(II) ion concentration and 0.4 g/L of adsorbent dosage. Adsorption of Cu(II) on mango leaf biosorbent was found to fit the Freundlich isotherm. Mango leaf was utilized as low-cost adsorbent for the removal of potassium ion from aqueous solution (Chaudhary et al., 2015). A maximum adsorption capacity of 42.5mg/g was obtained at pH 6.5 and Langmuir model fitted the equilibrium data better, giving correlation coefficient of 0.9999.

Removal of Pb(II) ions from aqueous solution by adsorption using bael leaves (Aegle marmelos) was studied by Chakravarty et al. (2009).The maximum Pb removal capacity was reported as 104 mg/ g at 50 mg/ L initial Pb(II) concentration at pH 5.1. The sorption process was described by pseudo second order kinetics model and Langmuir isotherm equations. Bael tree leaf was studied for removal of Pb ions (Kumar and Gayathri, 2009). The maximum adsorption capacity obtained was 90.07 mg/g at pH 5.0 at 30^0 C in for the initial metal ion concentration of 25-100 mg/l and an adsorbent dose of 5-20 g/l. In my earlier research paper it was studied that during the batch study of the chloride by using parthenium a significant adsorption was observed at pH -7. Veried dose shows different adsorption. (Lokesh Kumar, S. K. Sigh ,2015, Removal of chloride from Ground Water by Bio-adsorption, Int. J of advance research.

III. MATERIAL AND METHODS

A. *CHLORIDE SAMPLE & GAJARGHAS (Parthenium) AS BIOADSORBENT*

Natural Wastewater Sample with Chloride Concentration 1000 ppm was taken and the preliminary experiments were done with this sample. A plant known as grass / Gajarghas / Parthenium sps. / Congress (Belongs to the family asteraceae) was used as a bio-adsorbent. Before using, the Plant, it was dried & powdered (containing leaves and stem). The word 'Parthenium' has been derived from the Latin word 'Parthenice', meaning reputed medicinal merits. It has been speculated by scientists that the unique properties of this weed can be exploited for different purposes like activated carbon making, biomass generation, pesticide use etc. Parthenium is considered to be one of the ten worst weeds in the world. Parthenium is an herbaceous annual or ephemeral member of the family Asteraceae. It can reach heights of up to 2 m in good soil, and attain flowering in less than 4– 6 weeks of germination. Seeds produced per plant can go up to 25000. Dispersion of the seeds occurs through various vectors like water, muddy surfaces, small animals, vehicles, machinery etc. Partheniumhas a variety of vernacular names, like Congress grass, White top, Star Weed, Carrot Weed, Gajar Ghaas (Hindi), Ramphool, feverfew etc. The pollen and seeds of these plants are known to be a major cause of asthma, and bronchitis. The weed shows the presence of toxins known as Sesquiterpene lactones, parthenin, phenolic acids such as caffeic acid, vanillic acid,anisic acid, panisic acid, and parahydroxy benzoic acid which are known to be lethal to humans as well as animals.

B. *METHOD OF ACTIVATION OF BIO-ADSORBENT*

Sample collection was done on the basis of the health of the plant through visual observations. Young plants having a fresh green shoot and sizable stem thickness were selected. On collection, the specimens were washed with tap water and then distilled water. Further, for biomass collection, the specimens were subjected to drying in an oven at 100C for three days. Drying was followed by careful crushing of the specimen, and then sieving the mixture through a 500 micron sieve. The obtained biomass was used for the study. The biomass was added to the test solutions in the ratio of 0.10 g: 100 ml test solution. The pH of the sample was maintained at 7, and the chloride concentration at 100% of the initial wastewater solution. The mixture was then incubated in a rotary shaker incubator at 150 rpm and 300 C for 60 minutes. On the termination of contact time, the solution was filtered using an ordinary filter paper. The sample was titrated for chloride content using the Argentometric titration method specified in the APHA handbook for water and wastewater analysis. The tests were repeated by carrying out variations in pH, by using different chloride concentration levels, and with variable contact times.

IV. MODEL ON COLUMN PERFORMANCE

Optimization of parameters in the continuous adsorption process by experimental methods is an expensive and time consuming. It would be much cheaper and faster to use mathematical modeling to predict the duration of the column before regeneration become necessary (Warchol and Petrus 2006). Moreover, it is hard to generalized operational design parameters, therefore, theoretical models taking in to account of chemical and physical conditions become important (chen et al. 2003). Some of the well known mathematical models applied for column mode operation are Bed Depth Service Time (BDST) model, Thomas Model, Theoretical breakthrough curve etc.

A. BED DEPTH SERVICE TIME (BDST) MODEL

The Bohart-Adams equation can be represented as (Bohart and Adams 1920):

$$Ln\left(\frac{Co}{Cb} - 1\right) = Ln\left(e^{kadsN0(z/u)} - 1\right) - kadsC0ts \qquad (1)$$

Further, Hutchins (1973) modified the Bohart Adams equation and presented a linear relationship between the bed depth and service time which required only three fixed bed tests to collect the necessary data.

$$t_s = \frac{N_0 Z}{C_0 u} - \frac{1}{k_{ads} C_0} Ln\left(\frac{C_0}{C_b} \mathrm{I} 1\right) \qquad (2)$$

Where,

t_s is the service time at breakthrough point (h),
N_0 the dynamic bed capacity (mg L^{-1}) ,
Z the packed- bed column depth (cm),
u the linear flow rate (cm/h) defined as the ration of volumetric flow rate Q(cm^3/h) to the cross section area of the bed A (cm2) ,
C0 and Cb are respectively the influent and the breakthrough adsorbate concentration (mg/L) and
Kads the adsorption rate constant (L/mg.h).

Plotting service time (tss) versus bed depth (Z) will generate a straight line equation having slope of (N_0/C_0.u) and the intercept of (-)$\frac{1}{k_{ads} C_0} Ln\left(\frac{C_0}{C_b} - 1\right)$. Critical bed depth ($Z_0$) represents the theoretical minimum depth of column that would be able to prevent the adsorbate concentration from exceeding C_b. It is obtained when breakthrough is immediate and it can be calculated by substituting t_s = 0 in equation as below

$$Z_0 = \frac{u}{K_{ads} N}\left(\frac{C_0}{C_b} - 1\right) \qquad (3)$$

According to BDST model equation, the data collected from one flow rate experiment can predict the system with different flow rate. When an experiment conducted at flow rate Q_1, yields an equation of the form

$$t_s = a_1 z + b_1 \qquad (4)$$

The predicted equation for new flow rate Q_2 is given by :

$$t_s = a_2 z + b_1 \tag{5}$$

and $$a_2 = a_1 \left(\frac{Q_1}{Q_2}\right) \tag{6}$$

where a_1 and a_2 are the slop at flow rate Q_1 and Q_2 respectively. However the intercept b_1 remained same since it is independent of flow rate in line arized BDST equation. BDST equation can also be used to design system for treating other influent solute concentration using the data of a previous laboratory experiment of one influent solute concentration. When an experiment conducted at initial concentration C_1, yield an equation of the form

$$t = r_1 \; X + S_1 \tag{7}$$

The predicted equation for new flow rate Q_2 , is given by:

$$t = r_2 \; X + S_2 \quad \text{and} \tag{8}$$

The new slop and intercept values can be determined as :

$$r_2 = r_1 \left(\frac{C_1}{C_2}\right) \tag{9}$$

$$s_2 = s_1 \frac{C_1}{C_2} \left(\frac{Ln\left[{}^{C_2}/_{C_F} - 1\right]}{Ln\left[{}^{C_1}/_{C_b} - 1\right]} \right) \tag{10}$$

Where,

r_1 and r_2 is slopes at influent concentration
C_1 and C_2 respectively, S_1 and S_2 are intercept at influent concentration C_1 and C_2 respectively ,
C_F is effluent concentration at influent concentration C_2 and
C_b is effluent concentration at influent concentration C_1.

V. COLUMN ADSORPTION EXPERIMENTS

Continuous flow adsorption experiments were conducted in PVC columns of 4.0 cm inside diameter. At the top of the column, the influent fluoride solution (1000 mg /L) was pumped through the packed column (22, 44 and 66 cm), at flow rates of 5, 10 and 15 mL/min, using a peristaltic pump. Samples were collected from the exit of the column at regular time intervals and analyzed for residual chloride concentration at pH 7. The parthenium first activated and then it was used as bio adsorbent .The saturation capacity for the Parthenium in these column studies was calculated from the following equation where qe is the chloride adsorbed (mg/g), C_0 is the influent chloride concentration (mg/L), C is the effluent chloride concentration (mg/L), VE is the volume of solution required to reach the exhaustion point (L), and m is the mass of adsorbent (g).

A. EFFECT OF FLOW RATE

The adsorption columns were operated with different flow rates (5, 10, and 15 mL/min) until no further chloride removal was observed. The breakthrough curve for a column was determined by plotting the ratio of the Ce/C_0 (Ce and C_0 are the chloride concentration of effluent and influent, respectively) against time. The column performed well at the lowest flow rate (5 mL/min). Earlier breakthrough and exhaustion times were achieved, when the flow rate was increased from 5 to 10 mL/min. The column breakthrough time (Ce/C_0=0.05) was reduced from 65 to 35 min, with an increase in flow rate from 5 to 10 mL/min. This was due to a decrease in the residence time, which restricted the contact of chloride solution to the Parthenium. At higher flow rates the chloride ions did not have enough time to diffuse into the pores of the Parthenium and they exited the column before equilibrium occurred.

Successful design of a column chloride adsorption process requires a description of the dynamic behavior of chloride in a fixed bed. Various simple mathematical models have been developed to describe and possibly predict the dynamic behavior of the bed in column performance. One model used for continuous flow conditions is the Thomas model, which can be written as:

$$C_e/C_0 = 1/1 + exp\,((k_{th}/Q)(q_0 M - C_0 V_{eff})) \qquad (11)$$

Eq. can be expressed in linear form as:

$$ln[C_0/C_e - 1] = k_{th} q_0 M/Q - k_{th} C_0 t \qquad (12)$$

Where

V_{eff} is the volume of effluent (L),
k_{th} is the Thomas model constant (L/mg h),
$q_0$is the adsorption capacity (mg/g),
Q is the volumetric flow rate through column (L/h),
M is the mass of adsorbent in the column (g),
C_0 is the initial chloride concentration (mg/L) and
Ce is the effluent chloride concentration (mg/L) at any time t (h).

The Thomas model constants k_{th} and q_0 were determined from a plot of ln [C_0/Ce-1] versus t at a given flow rate. The model parameters are given in Table. The Thomas model gave a good fit of the experimental data, at all the flow rates examined, with correlation coefficients greater than 0.97, which would indicate the external and internal diffusions were not the rate limiting step. The rate constant (k_{th}) increased with increasing flow rate which indicates that the mass transport resistance decreases. The reason is that the driving force for adsorption is the chloride concentration difference between Parthenon and solution.

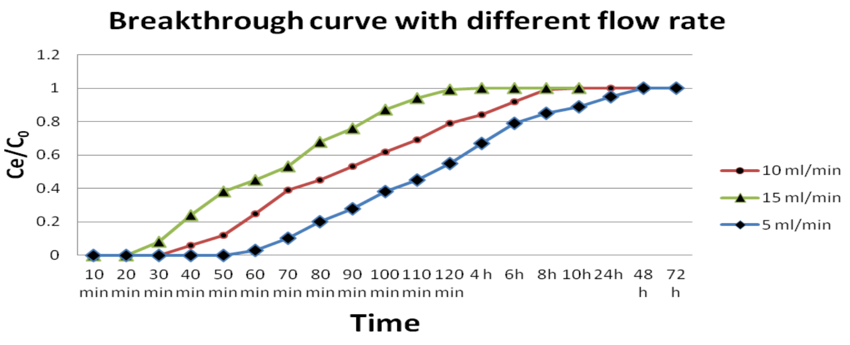

Fig 1 Breakthrough curves expressed as Ce/C_0 versus time at different flow rates (initial chloride Concentration 1000 mg/L, initial pH 7, bed depth 66 cm and temperature 30 ± 1°C)

B. EFFECT OF BED HEIGHT

The accumulation of chloride in a fixed-bed column is dependent on the quantity of adsorbent inside the column. In order to study the effect of bed height on chloride retention dry activated parthenium of three different bed heights, viz. 22, 44, and 66 cm, were used. A chloride solution of fixed concentration (1000 mg/L) was passed through the fixed-bed column at a constant flow rate of 5 mL/min. The breakthrough time varied with bed height. Steeper breakthrough curves were achieved with a decrease in bed depth. The breakthrough time decreased with a decreasing bed depth from 44 to 22 cm, as binding sites were restricted at low bed depths. At low bed depth, the chloride ions do not have enough time to diffuse into the surface of the Parthenium and a reduction in breakthrough time occurs. Conversely, with an increase in bed depth, the residence time of chloride solution inside the column was increased, allowing the chloride ions to diffuse deeper into the Parthenium. The breakthrough service time (BDST) model is based on physically measuring the capacity of the bed at various percentage breakthrough values. The BDST model constants can be helpful to scale up the process for other flow rates and concentrations without further experimentation. It is used to predict the column performance for any bed length, if data for some depths are known. It states that the bed depth, Z and service time, t of a column bears a linear relationship. The rate of adsorption is controlled by the surface reaction between adsorbate and the unused capacity of the adsorbent. The BDST equation can be expressed as follows

$$t_s = \frac{N_0 Z}{C_0 u} - \frac{1}{k_{ads} C_0} Ln\left(\frac{C_0}{C_b} - 1\right) \quad (13)$$

Where C_b is the breakthrough chloride concentration (mg/L),
N_0 is the adsorption capacity of bed (mg/L),
Z is depth of column bed (cm),
u is the linear flow velocity of chloride solution through the bed (mL/cm^{2}h),
Ka is the rate constant (L/mg h).

The column service time was selected as the time when the normalized concentration, Ce/C_0 reached 0.05. A plot of service time versus bed depth, at a flow rate of 5 mL/min was linear. The high correlation coefficient value (R^2= 0.988) indicated the validity of the BDST model for the present system. The values of N and Ka were evaluated from the slope (N/C_0u) and intercept ((1/KaC_0)ln[(C_0/C_b)-1]) of the BDST plot. The values of BDST model parameters are presented in Table . The value of Ka characterizes the rate of transfer from the fluid phase to the solid phase. If Ka is large, even a short bed will avoid breakthrough, but as Ka decreases a progressively deeper bed is required to avoid breakthrough

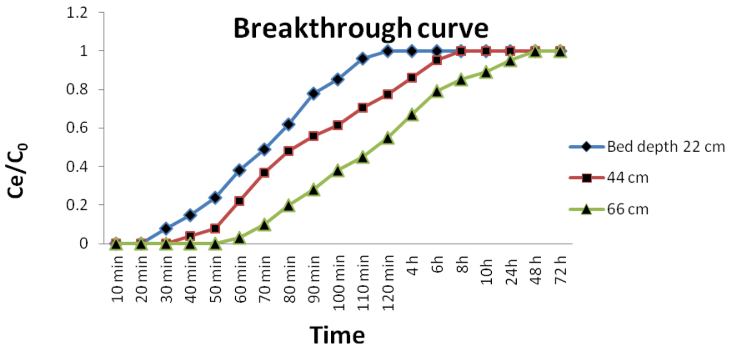

Fig 2 Breakthrough curves expressed as Ce/C_0 versus time at different bed depth (initial chloride Concentration 1000 mg/L, initial pH 7, flow rate 5 mL/min and temperature 30 ± 1°C)

C. THOMAS MODEL AND BDST MODEL PARAMETERS FOR THE ADSORPTION OF CHLORIDE ON PARTHENIUM

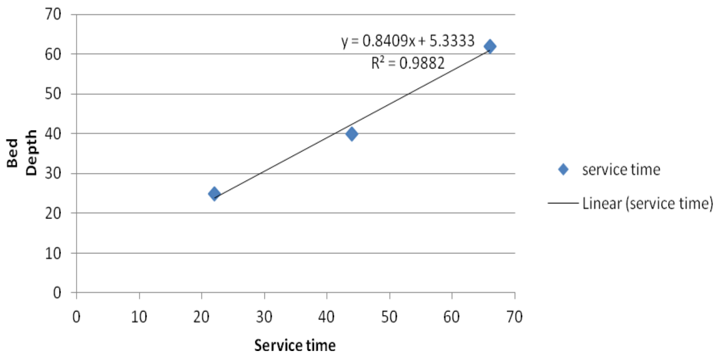

Fig 3 Parameters for BDST Models

Table 1 BDST MODEL coefficient

N (mg/L)	Ka (L/mg h)	R^2
20063	5.52X 10-4	0.998

D. PARAMETERS FOR THOMAS MODEL

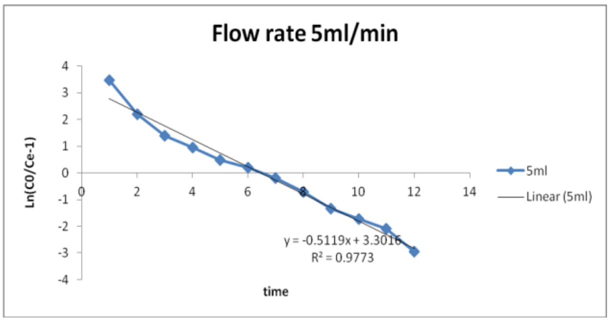

Fig 4 Parameters for Thomas model at flow rate 5ml/min

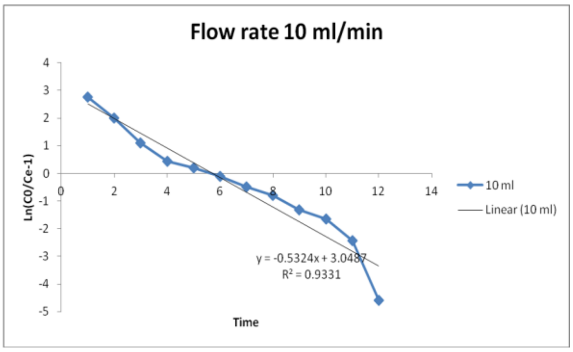

Fig 5 Parameters for Thomas model at flow rate 10 ml/min

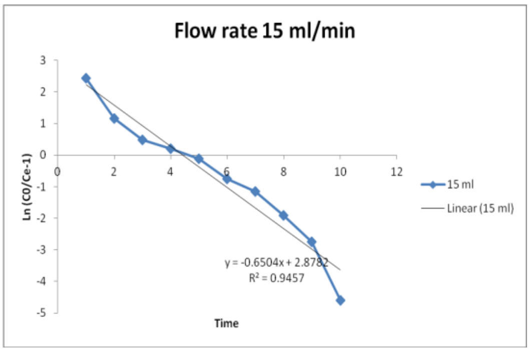

Fig 6 Parameters for Thomas model at flow rate 15 ml/min

Table 2 THOMAS MODEL COEFFICIENT

Flow Rate (ml/min)	**q0 (mg/g)**	**Kth (L/mg h)**	**R^2**
5	**3.87**	**0.000511**	**0.977**

10	**6.85**	**0.000532**	**0.933**
15	**7.95**	**0.00065**	**0.945**

VI. RESULTS AND DISCUSSION

BDST model is best suited to the observation and adsorbent performance compare to Thomas model. The high correlation coefficient value (R^2= 0.998) indicated the validity of the BDST model for the present system. Thomas model also shows good results. The correlation coefficient value (R^2= 0.977 with flow rate of 5 ml/min.). In the Thomas model as the flow rate increases from 5 to 15 ml/min the efficiency of the adsorption reduces. The column breakthrough time (Ce/C_0=0.05) was reduced from 65 to 35 min, with an increase in flow rate from 5 to 10 mL/min. This was due to a decrease in the residence time, which restricted the contact of chloride solution to the Parthenium. At higher flow rates the chloride ions did not have enough time to diffuse into the pores of the Parthenium and they exited the column before equilibrium occurred. The concentrations of chloride in the treated waste water are expected to be well below the permissible limits for discharge of effluents as per Indian standards. Regeneration of adsorbents is expected to allow reuse of the adsorbents. A good recovery of metal ions is expected on desorption. Studied waste material is expected to be of significant use in water treatment.

VII. CONCLUSION

Parthenium can be utilized as a good bio-adsorbent for the removal of Chloride salts. It can also be used for the removal of heavy metals and other effects on the waste water treatments. The different column models have been effective for study of maximum efficiency of parthenium towards removals of different salts.

VIII. FUTURE SCOPE

The column study can be conducted with some different bio adsorbents for removal of pollutants in water and waste water according to the affinity of the bio adsorbent. Desorption can also be studied for recovery of heavy metals.

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