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RESEARCH ARTICLE

Biosorption of Chromium (III) from Aqueous Solution by Water Hyacinth Biomass

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ABSTRACT

Release of heavy metals into the environment is a matter of global concern for the past few years and it should be removed from industrial waste waters before discharge for maintaining water quality standards. This study involved the use of water hyacinth biomass as biosorbent for removal of Cr(III) from aqueous solution. The biosorbent was characterized by FTIR, SEM with EDAX. Batch experiments were conducted to determine the sorption isotherms, effect of solution pH, adsorbate concentration, contact time, biosorbent dosage and size. The results confirmed that water hyacinth biomass can remove Cr(III) from aqueous solution. The maximum sorption occurs at pH =5.0, biosorbent size= 0.2mm, biosorbent dosage=2.0g $100ml^{-1}$ of adsorbate, adsorbate / solute concentration= 100 mg L^{-1} Cr(III) at 25ºC at 250 rpm after 24 hrs. The rate of adsorption was rapid and equilibrium attained at 24hrs. Langmuir, Freundlich and Temkin isotherm models were used to analyze the equilibrium adsorption data. Langmuir isotherm fitted the data well with R^2 value of 0.999. Overall, the powdered water hyacinth biomass could be utilized as effective low cost adsorbent for the removal of Cr(III) from waste water.

Keywords: *Adsorption, Cr Removal, Release kinetics, Water hyacinth*

Water is a basic human need which is well emphasized by the saying “A drop of water is worth more than a sack of gold to a thirsty man”. Though it is an essential commodity, disposal of industrial and urban wastes into them beyond its self cleaning capacity causes serious threat. The most prominent presage to the environment through anthropogenic activities is the heavy metal pollution. Heavy metals are trace metals, known to be most persistent and non biodegradable pollutant emanated from wide range of industries. Among that, chromium contamination is a significant obstacle worldwide due to its multifarious use in industrial processes such as plating, alloying, tanning of hides, metallurgy, refractory, textile dyes, paint, chemical manufacturing and petroleum refining etc., (Avudainayagam *et al.,* 2003). This naturally occurring element has a complex electronic and valence shell chemistry with valence states ranging from -2 to +6 (Shahid *et al.,* 2017). From this, the most environmentally significant forms of Cr are trivalent (Cr III) and hexavalent (CrVI). These two oxidation states differ in their mobility and toxicity. Among them, Cr(VI) is mobile and regarded as carcinogen whereas Cr(III) is relatively immobile and less toxic.

The Cr(III) has a significant role in lipid and sugar metabolism and regarded as essential trace element for human and animal health (Eskin, 2016) and not for the plants. Though Cr(III) has its own beneficial role, its uptake in supererogatory amounts causes health effects as well as skin rashes. Since Cr(III) is less toxic and immobile, the environmental conditions and complex of physical, chemical and biological factors have the capability to convert it into Cr(VI). However, there is a statement that, in drinking water all Cr is converted to Cr(VI) by strong oxidants such as chlorine, ozone and permanganate utilized for attaining taste, odour removal and disinfection (Clifford and Chau, 1988). So it needs a necessary relook to sequester Cr(III) before discharge to water bodies.

Conventional methods such as membrane filtration, chemical precipitation, reduction, ion exchange, chelation, reverse osmosis and electrodialysis are available for its removal (Khatoon et al., 2013) but their expensive nature, energy requirements and generation of toxic sludge calls for economically sound and ecologically safe technology. Search for technologies directed attention towards biosorption. Biosorption is a physico- chemical process in which biomass concentrate and bind the contaminant onto their cellular structure

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by various mechanisms like adsorption, absorption, ion exchange, surface complexation and precipitation (Fomina and Gadd, 2014). The present investigation focuses on Cr(III) removal utilizing the biomass of water hyacinth as biosorbent. Among the aquatic plants, water hyacinth has a great potential to remove pollutants due to its polyfunctional metal binding sites (carboxyl, amine imidazole, phosphate, sulphate, sulfhydryl, hydroxyl) and chemical functional groups present in cell proteins and sugars (Mahmood et al., 2010). Water hyacinth (*Eichhornia crassipes*) is the world's most prominent noxious weed grown over a wide range of aquatic environments. This dreaded aquatic weed pose serious challenge to aquatic inhabitants and it is often blamed for its choking nature in water bodies. Presently with the thought of recognizing its role in environmental cleaning, the study has been undertaken to study the effectiveness of water hyacinth biomass in removal of Cr(III) from aqueous solutions.

MATERIAL AND METHODS

Adsorbent and adsorbate

The water hyacinth plants were collected from Vaigai Dam of Theni district, Tamil Nadu, India. The aquaphytes were washed to remove sediments and other small floating freshwater aquatic weed like *Lemna minor.* After washing, the samples were dried, ground with a blender and sieved (desirable). The stock solution of 1000 mg L^{-1} of Cr(III) was prepared by dissolving 4.81g of analytical grade $Cr_2(SO_4)_3.6H_2O$ in 1000ml of double distilled water. Appropriate dilution of the above stock was done to obtain the solutions of required concentration. The pH of the metal solution was adjusted using 1.0 N NaOH or 1.0 N HCl.

Characterization of biosorbent

The biosorbent was tested for various properties with the methodology outlined by Jackson (1973) for pH and EC, USEPA (1979) for total Cr, AOAC (2000) for ash content and AOAC (1940) for crude fibre. The properties of the biosorbent are given in Table1.The morphology of the adsorbent material was analyzed with Scanning Electron microscopy (SEM) (FEI-Quanta 250, Czech republic) at 7.0kV equipped with the back scattered electron detector. Energy Dispersive X- ray (EDAX) spectroscopy was used to determine the elemental composition of the biosorbent. The functional groups present in the biosorbent were identified through Fourier transform infrared spectrometry equipped with diamond tipped ATR accessory (Shimadzu, Japan) at wave number of 4000 to 400cm^{-1}.

Batch experiments

Batch experiments were conducted to study the influence of varied solution pH (1 to 9), biosorbent size (0.2 to 4.0mm), biosorbent dosage (0.5 to 2.5g 100ml^{-1} of adsorbate), solute concentration (100 to 2000 mg L^{-1}) and contact time (6 to 48hrs) on sorption behaviour of Cr(III) and to establish optimum experimental strategies for adsorption process. These batch experiments were carried out at 25°C in 250 ml conical flasks with 100 ml solute volume and agitated on a rotary shaker at 250 rpm. At the end of shaking period, the samples were centrifuged at 8000 rpm for 10 minutes and filtered through whatman no.42 filter. The residual concentration of metal ion (Cr) was determined by atomic absorption spectrophotometer (Perkin Elmer AAnalyst400). The batch experiments were replicated four times.

The amount of Cr(III) ions adsorbed onto the biosorbent at equilibrium (q_e) was calculated using eqn.(1).

where C_0 and C_e are the initial and equilibrium concentration of adsorbate in solution (mg L^{-1}), V is the

$$q_e = \frac{(C_0 - C_e)V}{m} \qquad \text{———} \quad (1)$$

volume of adsorbate solution taken (L) and m is the mass of the adsorbent (g) used.

The percent removal of Cr(III) from aqueous solution was determined using eqn.(2)

$$\%\ \text{removal} = \frac{(C_0 - C_e)}{C_0} * 100 \qquad \text{———} \quad (2)$$

where C_0 and C_e are the initial and equilibrium concentration of adsorbate in solution (mg L^{-1})

Isotherm study

To determine the equilibrium relationship between sorbent and sorbate in solution, solution concentrations of 100 to 2000 mg L^{-1} were prepared with the adjustment of pH to 5. Then the mixture of 100ml of each solution and 2g of adsorbent were agitated in a rotary shaker at 250 rpm and 25°C for 24hrs. The data obtained were fitted to Langmuir, Freundlich and Temkin isotherms.

Langmuir adsorption isotherm is valid for the formation of a monolayer adsorbate on the biosorbent surface and it is given by eqn. (3)

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \qquad \text{———} \qquad (3)$$

Where q_m is the maximum monolayer coverage capacity (mg g^{-1}), q_e is the amount of metal adsorbed per gram of biosorbent at equilibrium (mg g^{-1}), C_e is the equilibrium concentration of adsorbate (mg L^{-1}) and K_L is the langmuir adsorption constant related to the affinity of binding sites (Lmg^{-1}). The values of q_m and K_L were obtained from slope and intercept of plot between C_e/q_e Vs C_e. This Langmuir equation was further analyzed through the dimensionless constant called separation factor (K_R), which is defined by the following equation (4).

$$K_R = \frac{1}{1+K_L C_0} \qquad \text{———} \qquad (4)$$

Where K_L is the Langmuir adsorption constant and C_o is the initial concentration of the adsorbate. For $K_R > 1$, unfavourable, $K_R=1$, linear, $0< K_R<1$, favourable, $K_R=0$, irreversible.

The equation (5) represents Freundlich isotherm, which is commonly used for describing adsorption on heterogenous surface.

$$q_e = K_F C_e{}^{1/\mathrm{n}} \text{———}$$

Where K_F is the constant related to adsorption capacity (mg g^{-1}) and n is the intensity of biosorbent. The values of n and K_F were obtained from slope and intercept of Freundlich plot of log q_e Vs log C_e. The favourability of adsorption is indicated by the magnitude of the exponent 'n'. It is stated that adsorption characteristics is good (if n=2-10), moderately difficult (if n = 1-2) and poor (if n < 1).

Temkin isotherm is used for heterogenous surface energy systems and this isotherm assumes the heat of adsorption which is the function of temperature. The equation (6) represents the Temkin adsorption equation as furnished below.

$$q_e = \frac{RT}{B}\ln A + \frac{RT}{b}\ln C_e \qquad \text{———} \qquad (6)$$

$$\mathrm{B} = \frac{RT}{b}$$

Where R is the universal gas constant (8.314 J mol^{-1} K^{-1}), T is the temperature (25°C), b is the Temkin isotherm constant, A is the Temkin isotherm equilibrium binding constant (L g^{-1}) and B is the constant related to heat of sorption (Jmol^{-1}). The values of B and A were obtained from slope and intercept of temkin plot between q_e and ln C_e.

RESULTS AND DISCUSSION

Characterization of biosorbent

The surface morphology of the biosorbent material was examined by scanning electron microscope. Fig. 1 showed a clear striated and nebulous texture of biosorbent material which may provide large surface for the adsorption of Cr whereas Fig.3 depicted a rough aggregated surface and irregular microstructures. This difference in surface structure of Cr loaded biosorbent could be due to agglomeration of chromium ions on the surface of the biosorbent (Balarak *et al.,* 2014). The EDAX spectra was also recorded to know the elemental composition of the biosorbent before and after Cr(III) adsorption (Figs. 2 & 4). The peaks

Table 1. Physico chemical properties of water hyacinth biomass (biosorbent)

Parameter	Value
pH	6.48
EC (dSm^{-1})	1.61
Ash content (%)	18.0
Crude fibre (%)	19.0
Total Cr (mg kg^{-1})	BDL
Total Cd (mg kg^{-1})	BDL
Total Ni (mg kg^{-1})	BDL
Total Pb (mg kg^{-1})	BDL

(BDL- Below Detectable Limit)

of Ca, O, C indicated the presence of functional groups such as –OH and –COOH in the biosorbent (Jain *et al.,* 2013). The EDAX spectra of biomass before Cr adsorption showed no prominent peaks of chromium whereas the spectra after Cr(III) adsorption produced prominent Cr peaks with the reduction in Ca peaks.

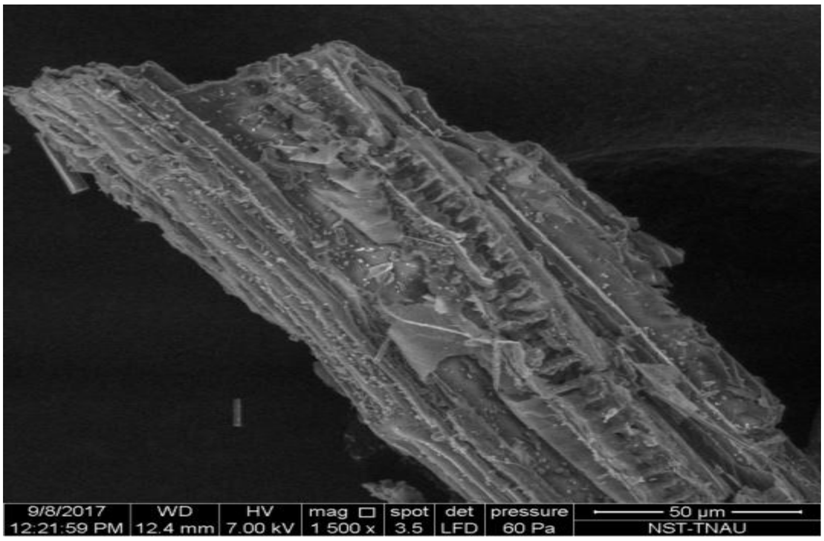
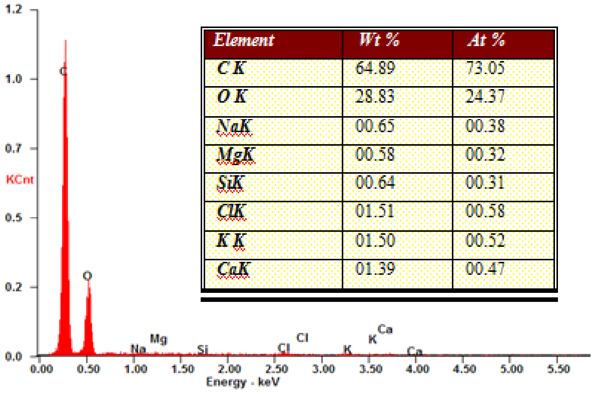


Figure 1. Scanning electron micrograph of water hyacinth biomass

The FTIR spectra of water hyacinth biomass before and after Cr (III) adsorption are shown in Figs.5 & 6. The FTIR spectra of water hyacinth biomass before Cr adsorption showed intense peaks at 2700-3500 cm^{-1} which represent -OH group stretching and –NH bonds with the broad peak at 3332.39cm^{-1}.



Element	*Wt %*	*At %*
C K	64.89	73.05
O K	28.83	24.37
NaK	00.65	00.38
MgK	00.58	00.32
SiK	00.64	00.31
ClK	01.51	00.58
K K	01.50	00.52
CaK	01.39	00.47

Figure 2. EDAX spectra of water hyacinth biomass

The strong spectral peaks at 1024.98 cm^{-1} showed C-O bonds stretching. The FTIR spectra of Cr(III) loaded biosorbent had broad peaks at 3284.18 cm^{-1} and 1019.19cm^{-1} showing the presence of hydroxyl, amine and C-O bonds stretching. The distinct changes were recorded in spectra of biomass after Cr(III) adsorption compared to spectra before Cr adsorption. The chemical shift in the positions of –OH, N-H and C-O group peaks indicated their involvement in the binding of Cr(III).

Table 2. Isotherm constants for adsorption of Cr(III) to water hyacinth biomass

Constants	Cr(III)
Langmuir isotherm	
q_m (mg g^{-1})	76.90
K_L (L mg^{-1})	0.08
R^2	0.99
Freundlich isotherm	
n (L mg^{-1})	2.94
K_F (mg g^{-1})	12.00
R^2	0.86
Temkin isotherm	
B (J mol^{-1})	10.9 0
A (L g^{-1})	2.40
R^2	0.94

Adsorption factors

Solution pH

The solution pH, solute concentration, biosorbent size, dosage and contact time are known to be the factors affecting adsorption of adsorbate. The solution's pH is one of the prominent factor which affects the Cr(III) adsorption onto the adsorbent. The pH dependence for Cr(III) adsorption onto biosorbent was studied at varied pH levels *viz.,* 1,2,3,4,5,6,7,8,9 (Fig.7), where the optima occurred at pH 5.0 (9.98 mg g^{-1} of adsorbent) which was on par with pH 4.0 (9.97 mg g^{-1} of adsorbent). The least adsorption occurs

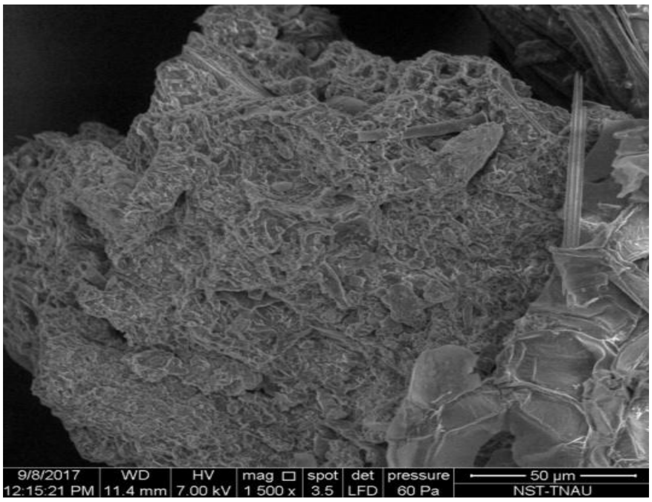
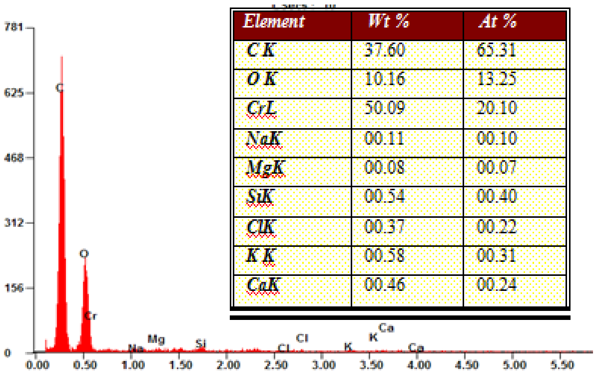


Figure 3. Scanning electron micrograph of water hyacinth biomass loaded with Cr(III)



Element	*Wt %*	*At %*
C K	37.60	65.31
O K	10.16	13.25
CrL	50.09	20.10
NaK	00.11	00.10
MgK	00.08	00.07
SiK	00.54	00.40
ClK	00.37	00.22
K K	00.58	00.31
CaK	00.46	00.24

Figure 4. EDX spectra of water hyacinth biomass loaded with Cr (III)

at pH 9.0 (6.07 mg g^{-1} of adsorbent) with the percent removal of 60.7%. The changes in percent removal of Cr(III) at different pH had been observed, which might be attributed by factors like charge of the metal ion in aqueous solution and charge on biosorbent surface at particular pH.

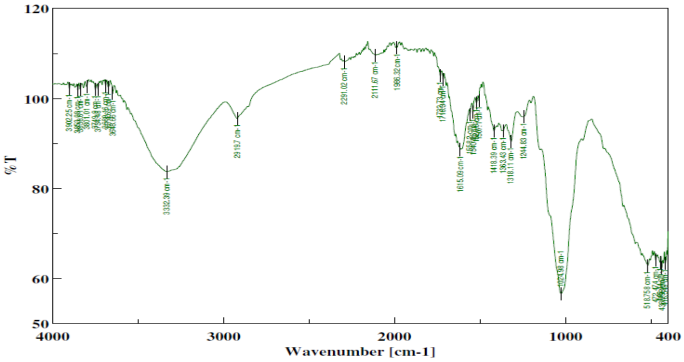


Figure 5. FTIR spectra of water hyacinth biomass

In aqueous solution, Cr(III) exists as cation (Bedemo *et al.,* 2016). The maximum adsorption at pH 5 might be due to the fact that at higher pH, the surface charge of the biosorbent becomes negative increasing % removal (Chathuranga *et al.,* 2012). At lower pH, the adsorption of Cr(III) onto the biosorbent was minimum due to the competition of H^+ and metal ions and electrostatic repulsion (Jeon and Holl, 2003).

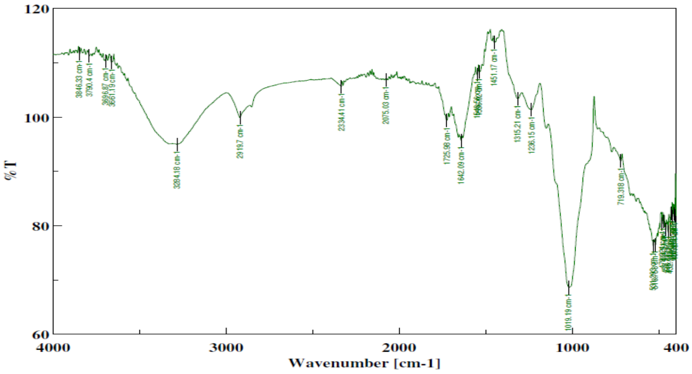


Figure 6. FTIR spectra of water hyacinth biomass loaded with Cr(III)

At moderate pH (3 to 6), the H^+ ions linked with active sites of biosorbent will be released favouring the adsorption of metal cations but at higher pH (>6), precipitation of metals and hydroxide formation dominates restricting the metal adsorption (Salehzadeh, 2013).

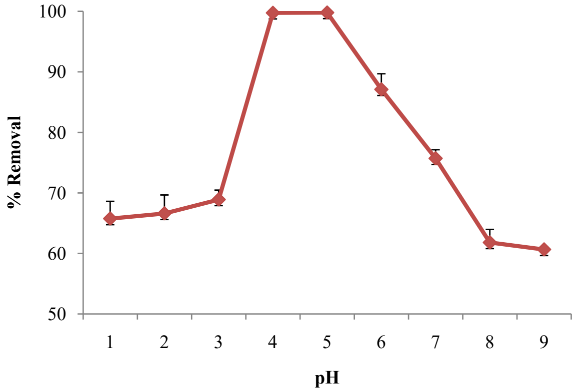


Figure7. Effect of solution pH on Cr(III) adsorption

Biosorbent size

To analyze the effect of biosorbent size on adsorption process, batch experiments were conducted at fixed solute concentration of 200 mg L^{-1} Cr(III) with varying biosorbent size of 0.2, 0.5, 1.0, 2.0, 4.0 mm. Among different sizes of biosorbent, 0.2 mm sorbed the highest quantity of 9.98 mg Cr(III) g^{-1} (99.8% removal)

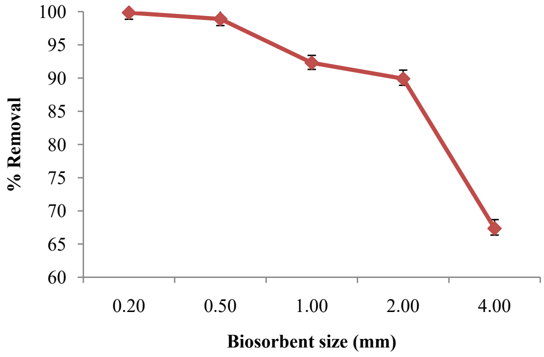


Figure 8. Effect of biosorbent size on Cr(III) adsorption

followed by 0.5 mm which sorbed 9.89 mg (98.9%). Higher adsorption by smaller size of the biosorbent might be due to the fact that, decrease in particle size increases the surface area and the number of binding sites for adsorption increases with increases in surface area (Muthulakshmi and Anuradha, 2015).

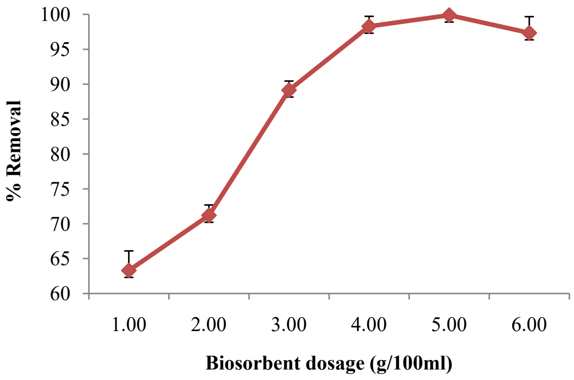


Figure 9. Effect of biosorbent dosage on Cr(III) adsorption

Biosorbent dosage

The effect of biosorbent dosage on the removal of Cr(III) from aqueous solution is presented in Fig.9. The batch experiment were conducted at fixed adsorbate concentration of 200 mg L^{-1}, pH = 5, 0.2 mm biosorbent size with dosage of 0.5 to 2.5 g 100 ml^{-1} of adsorbate with 0.5g intervals.

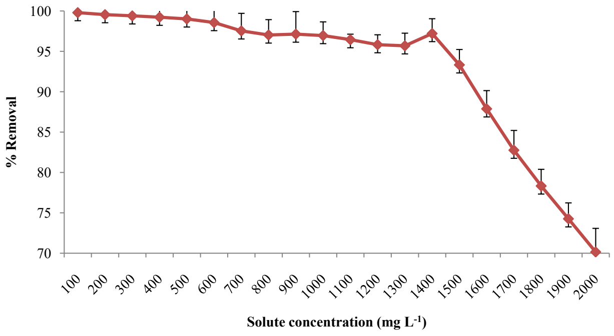


Figure 10. Effect of solute concentration on Cr(III) adsorption

Unit adsorption of Cr(III) decreased from 50.6 to 7.79 mg g^{-1} on increasing the biomass dosage from 0.25 g to 2.5 g for 100 ml of adsorbate but the percent removal of Cr(III) increased from 63.3% to 99.9% on increasing the biomass dosage from 0.5 to 2.0 g. This may be due to the fact that, increasing dosage of biosorbent in the aqueous solution increased the active sites and surface area available for adsorption (Mohammed *et al.,* 2013). At biosorbent dosage of 2.5g, there was a decrease (97.4 %) in adsorption of Cr(III) which may be due to overlapping and aggregation of potential adsorption sites as the result of overcrowding of adsorbent particles (Shanthi and Selvarajan, 2012).

Solute concentration

Adsorption of Cr(III) with regard to the effect of initial metal concentration was studied at the range of 100 to 2000 mg L^{-1} Cr(III) with 100 mg L^{-1} intervals and the experiment was conducted in batch process with fixed parameters of pH 5.0, 0.2 mm biosorbent size, 2.0g of biosorbent dosage. Fig.10 illustrates the adsorption of Cr(III) ions by water hyacinth biomass as a function of initial adsorbate concentration. The amount of Cr(III) sorbed ranged between 4.99 to 70.6 mg g^{-1} for biomass. The sorption capacity increased with increasing the solute concentration but the percent removal gets decreased.

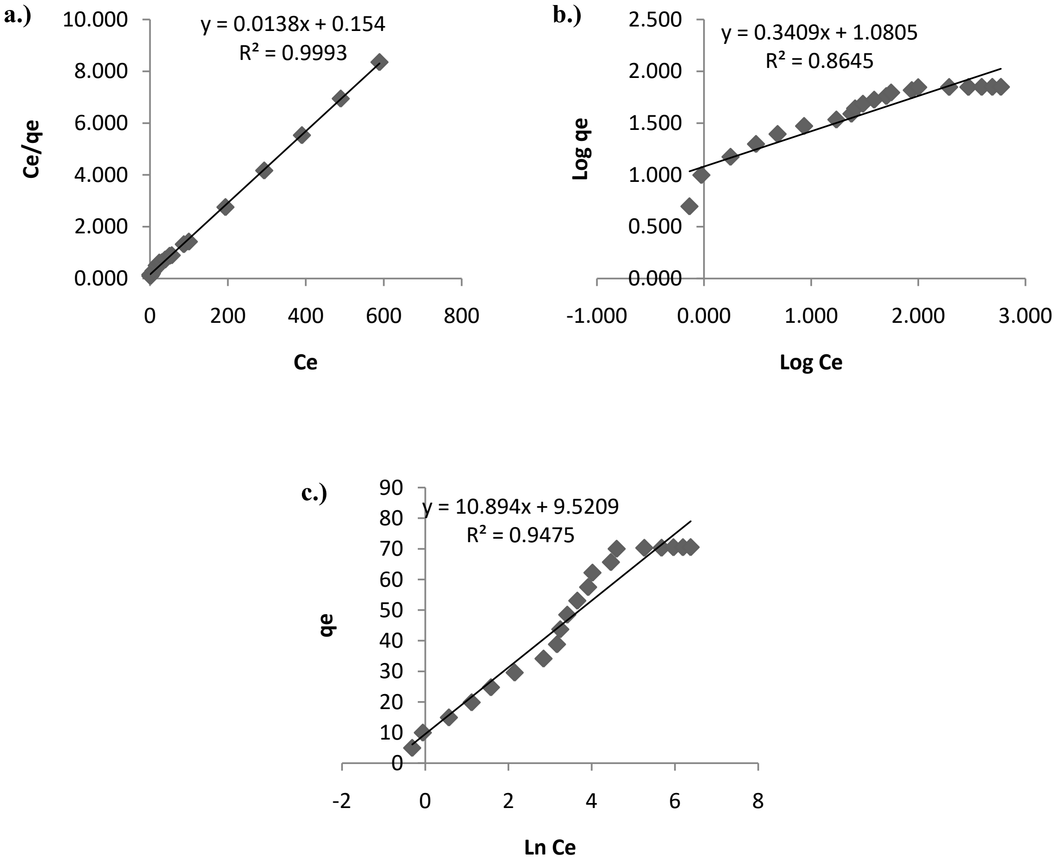


Figure 11. Isotherm plots for adsorption of Cr (III) onto water hyacinth biomass. a.) Langmuir isotherm, b.) Freundlich isotherm, c.) Temkin isotherm

The results showed that, the highest removal of 99.8% was recorded with 100 mg L^{-1} solute concentration beyond that the metal removal went down to 70.2% (2000 mg L^{-1}). This decrease in % removal with increase in solute concentration might be due to the lack of available active binding sites required for high solute concentration of aqueous solution (Freitas *et al.,* 2008).

Isotherm study

To understand the adsorption mechanism at solid liquid interface, the data obtained were fitted to models like Langmuir, Freundlich and Temkin isotherm models were used in the present work. The isotherm plots for the biosorption of Cr(III) was shown in Fig.11. The parameters of isotherm models calculated from experimental data were shown in Table 2. The maximum monolayer adsorption capacity (q_m) of 76.9 mg g^{-1} was reported for Cr(III). The Freundlich, Langmuir and Temkin isotherm model showed R^2 values of 0.864, 0.999 and 0.947, respectively which confirmed the suitability of Langmuir model. It confirms that biosorption of Cr(III) onto biosorbent occurred onto the active sites as monolayer (Adbel- Aty *et al.,* 2013).The K_R values for each of the solute concentrations were found to be between 0 and 1, indicating favourable adsorption of Cr(III) onto water hyacinth biomass. Considering the Freundlich isotherm, the R^2 value was less compared to langmuir isotherm and the 'n' value was in the range of 2–10 (2.94), which showed a good adsorption characteristics.

Contact time

The amount of Cr(III) adsorbed with respect to the contact time was studied within the range from 6hrs to 48hrs (Fig.12). The initial concentration of Cr(III) was taken as 200 mg L^{-1}, biosorbent size of 0.2mm, biosorbent dosage of 2.0g and solution pH of 5. Almost 90% of Cr(III) removal occurred at initial contact time of 6hrs and after 24hrs, equilibrium was obtained. The gradation in amount of Cr(III) adsorbed at initial periods was due to the increased concentration gradient between Cr(III) in solution and Cr(III) in biosorbent as that would increase the number of vacant sites available for adsorption (Singh *et al.,* 2016).

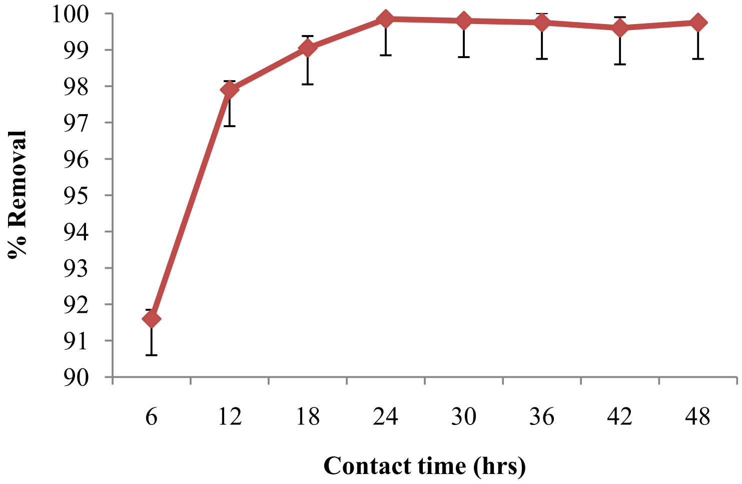


Figure 12. Effect of Contact time on Cr(III) adsorption

Once the surface sorption sites got exhausted, the rate of Cr(III) removal becomes slower and is controlled by rate of its transport from exterior to interior sites of biosorbent particles (Anandkumar and Mandal, 2009).

CONCLUSION

This study shows that water hyacinth biomass adsorbs Cr(III) ions to the tune of 76.9 mg g^{-1}. The sorption of Cr(III) onto water hyacinth biomass was influenced by factors like solution pH, solute concentration, biosorbent size, dosage and contact time. From the data obtained, it is clear that adsorption process follows Langmuir model. The optimum pH for the removal of Cr(III) was 5.0, biosorbent size= 0.2mm, biosorbent dosage 2.0g $100ml^{-1}$ of adsorbate, adsorbate concentration= 100 mg L^{-1} Cr(III) at 25°C at 250 rpm after 24 hrs. Further, the water hyacinth shall be utilized for the removal of Cr from industrial waste water, where there may be competition between different metal ions for adsorption sites onto the adsorbent.

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