International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 3 Issue 5, May-2014

Removal of Heavy Metals from Wastewater using Iron Plaque of *Typha-latifolia* & Bacterial Precipitated $Fe(OH)_3$ Rich Soil

(A Natural Biogenic Iron Oxide)

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***Abstract*- This paper examines the use of iron plaque of *Typha-latifolia* & bacterial precipitated $Fe[OH]_3$ rich soil [Natural Biogenic Iron Oxide] for Removal of Chromium [Cr^{+6}] & Copper [Cu^{2+}] from waste water. In this work sorption of Chromium [Cr^{+6}] & Copper [Cu^{2+}] by iron plaque of *Typha-latifolia* & bacterial precipitated $Fe[OH]_3$ rich soil has been studied. The adsorption capacity for Chromium [Cr^{+6}] & Copper [Cu^{2+}] onto iron plaque of *Typha-latifolia* increased with the decreasing of media size and the maximum uptake were 0.0229 mg Cr^{+6}/g, 0.048 mg Cu^{+2}/g at 298.15 K. In the batch system adsorption equilibrium of Chromium [Cr^{+6}] was attained at 180 min, the removal is then almost stabilizing after 180 min. implying that the equilibrium has been reached, and the optimal media size of [Iron plaque of *Typha-latifolia*] was 150 µ. The maximum sorption efficiencies at equilibrium were 70.6 %, 84.2 %, 89.0 %, 91.6 % for Chromium [Cr^{+6}] ion at 1.18 mm, 600 µ, 300 µ, 150 µ media size respectively. The optimal media size of [iron plaque of *Typha-latifolia*] for Copper [Cu^{2+}] ion was 75µ & the maximum sorption efficiencies were 64 %, 84 %, 96.2 % for 300 µ, 150 µ, 75µ media size respectively. And the adsorption capacity of Chromium [Cr^{+6}] on the bacterial precipitated $Fe[OH]_3$ rich soil increased with the increasing of media size and the maximum adsorption capacity for Chromium [Cr^{+6}] ion on the bacterial precipitated $Fe[OH]_3$ rich soil was 0.022 mg Cr^{+6}/g at 298.15 K & the maximum sorption efficiency was 86%. The characterization of iron plaque consists of Scanning Eectron Microscopy [SEM], Energy-dispersive X-ray spectroscopy [EDX].**

Key words- Heavy Metals, Iron Plaque of Typha-latifolia, Bacterial Precipitated $Fe[OH]_3$ Rich Soil, Natural Biogenic Iron Oxide, Adsorption Capacity

I. INTRODUCTION

The presence of heavy metal ions such as Chromium [Cr^{+6}] & Copper [Cu^{2+}] in industrial wastewaters & municipal wastewaters is a potential hazard to aquatic, animal & human life. Hexavalent chromium [Cr^{+6}] is product of various industrial operations, when trivalent chromium, [Cr^{+3}] is heated in the presence of mineral bases and atmospheric oxygen. Chromium compounds are widely used in a number of industries such as leather, textile, chemical printing, dye-ink manufacturing, electroplating industries etc. From all these processes, chromium can escape into the environment through the effluents. Hexavalent chromium is 100-1000 times more toxic than the most common trivalent compounds. The tolerance limit for [Cr^{+6}] for discharge into inland surface waters is 0.1 mg/l and in potable water is 0.05 mg/l [EPA,1990]. It is essential that industries treat their effluents to reduce the [Cr^{+6}] to acceptable levels. A number of treatment methods for the removal of metal ions from aqueous solutions have been reported, mainly reduction, ion exchange, electrodialysis, electrochemical precipitation, evaporation, solvent extraction, reverse osmosis, chemical precipitation and adsorption [Patterson,1985]. Many reports have appeared on the development of low-cost activated carbon adsorbents developed from cheaper and readily available materials [Babel et al., 2003; Bailey et al., 1999; Pollard et al., 1992]. Adsorption of [Cr^{+6}] by a number of materials such as leaf mould [Sharma and Forster, 1994], activated groundnut husk carbon [Srinivasan et al., 1991; Periasamy et al., 1991], coconut husk and palm pressed fibres [Tan et al., 1993], coconut shell activated. As an environmentally friendly technique, biosorption with seaweed was implemented in the recovery and recycling of chromium [Aravindhan et al., 2004]. Copper [Cu^{2+}] is product of various industrial operations. Its concentrations in industrial wastewaters range from 0.5 to 270 mg/l [Patterson, 1985]. Copper [Cu^{2+}] compounds are widely used in a number of industries. [Cu^{2+}] from electronics & related devices, electric motors, architecture & industry, alloys industries, antibiofouling applications, ceramic glazer etc. Copper in industrial wastewaters & municipal wastewaters is a potential hazard to aquatic, animal & human life. Copper is product of various industrial operations. Copper compounds are widely used in a number of industries such as Copper from electronics & related devices, electric motors, architecture & industry, alloys industries, antibiofouling applications, ceramic glazer etc. From all these industries & processes copper can escape into the environment through the effluents. Copper has toxic effect on human because of its non-biodegradability biological magnification and long persistent in the environment & very adverse effect on environment. Copper metabolism leading to severe copper toxicity [i.e.,Wilson disease]. Liver is the primary organ of copper-induced toxicity. Other target organs include bone central nervous & immune systems. Excess copper intake also induces toxicity indirectly by interacting with other nutrients. Copper has toxic effect on aquatic life; it affects public sewers & sewage treatment plants.

II. MATERIALS AND METHODS

A. Natural Biogenic Iron Oxides [Iron Plaque of *Typha-latifolia* & Bacterial precipitated Fe[OH]$_3$ rich soil]

Biogenic iron oxide is the nanocrystals which are produced by metabolic activity of acediophilic & neutrophilic iron oxidizing bacteria in oxic condition that oxidize soluble Fe^{2+} to Fe^{3+} producing a hydroxide precipitate [iron oxide Fe[OH]$_3$] as external/internal precipitate in wetland, lakes, rivers [fresh water to marine], aquifers, soil, mining area [Fortin D., Langley S., (2005)]. Biogenic iron oxides are produced by auto-lithotrophic microbes that oxidize soluble Fe^{2+} to Fe^{3+} creating a hydrated iron hydroxide precipitate in an organic matrix [Weiss, D. E., (2004); Edwards, K. J., Bach, W., McCollom, T. M., & Rogers, D. R., (2004)]. Iron oxidizing bacteria grows at anoxic/oxic interfaces that retain circumneutral pHs [Emerson, D., & Revsbech, N. P., (1994); Emerson, D., & Moyer, C., (1997)]. Iron oxidizing sheath/filament of *Leptothrix-ochracea* is found in lakes in which iron oxidizing bacteria are found. *Gallionella capsiferriformans* found in rhizosphere of *Typha Latifolia*. *Sideroxydans paludicola* oxidizes iron in the rhizosphere of *Typha Latifolia*, *Magnolia* under microaerobic conditions to produce insoluble ferric hydroxide Fe[OH]$_3$. [Weiss, J.V., (2007); Neubauer, S.C., Toledo-Duran, G.E., Emerson, D., (2007); Emerson, D., Weiss, J.V., Megonigal, J.P., (1999)].

At the roots of wetland plant *Typha-latifolia*, it has been found that the iron oxidation has happened in the form of iron plaque because the roots will have a rusty, reddish/orange coating on them. Not all plants will have this iron plaque on them and would not necessarily be distributed all over the roots. Iron plaque is a combination of iron oxide particles, living & dead microbial cells [iron oxidizers and other microbes], and various extracellular substances [organic polymers, etc.]. Iron plaque on roots can be seen as red-orange zones on the roots. On roots, the iron plaque is a solid and Fe^{+2} is oxidized by Fe-oxidizing bacteria at their cell surface, i.e., outside the cell. In wetlands, you can sometimes see orangish zones on the soil surface that presumably reflects sites where iron oxidation has occurred. Microbial mats in aquatic systems tend to be solid or semi solid [i.e., they have high water content and are often unconsolidated].

B. Site Characteristics

Table-1 Presence of biogenic iron oxide can also be confirmed by evaluation of some parameters at the sampling site.

Parameters	Biogenic Iron Oxide	*Leptothrix ochracea*	*Gallionella capsiferriformans*	*Sideroxydans paludicola*
D.O. mg/L	1.41 ± 0.45	Anoxic Groundwater	<10% ambient O_2	Microaerobic condition
pH	7.16 ± 0.16	7.1 - 7.6	7.1	4.5
Dissolved Fe^{+2}	9.32 ± 5.74 mg/L	250µM	3 - 12 µM	453 µM
Temperature	8 - 14°C	8 - 14°C	10°C	10°C
Organic Density	0.90 ± 0.12 mg/L,	Flow < 2 mL/s		High (13%)
Solids Density	1.89 ± 0.63 mg/L			

Source: [Emerson, D., & Revsbech, N. P., (1994); Emerson, D., & Moyer, C., (1997); Weiss, J.V., (2007); Rentz, J. A., Turner, I. P., & Ullma, J. L. (2009)]

C. Material Collection, Preparation & Characterization of Media

Preparation of Natural Biogenic Iron Oxide [Iron Plaque of *Typha-latifolia*] consists of selection of some wetland plants *Typha-latifolia* from wetland area Raebareli India having iron plaque on their roots, on roots the iron plaque is a solid form. After selection of *Typha-latifolia* plants, cut & collect the portion of fresh roots of *Typha-latifolia* having iron plaque. After collection of roots of *Typha latifolia* wash the iron plaque of *Typha-latifolia* with double distilled water & collect the whole eluent in a glass jar containing particles of iron plaque or natural biogenic iron oxide or bacterial precipitation & passing the particle solution through the geometric sieve for retaining 600µm, 300µm, 150µm,75µm size. Stored the material in glass jar for further use. And the bacterial precipitated Fe[OH]$_3$ rich soil collected from wetland area Raebareli India. In wetlands where plants of *Typha-latifolia* occur, you can see orangish/reddish zones on the soil surface that presumably reflects sites where iron oxidation has occurred. After collection of bacterial precipitated Fe[OH]$_3$ rich soil, dry it in sunlight for making it moisture less & then passing the soil through the geometric sieve for retaining 600µm, 300µm, 150µm, size. Stored the material in glass jar for further use.

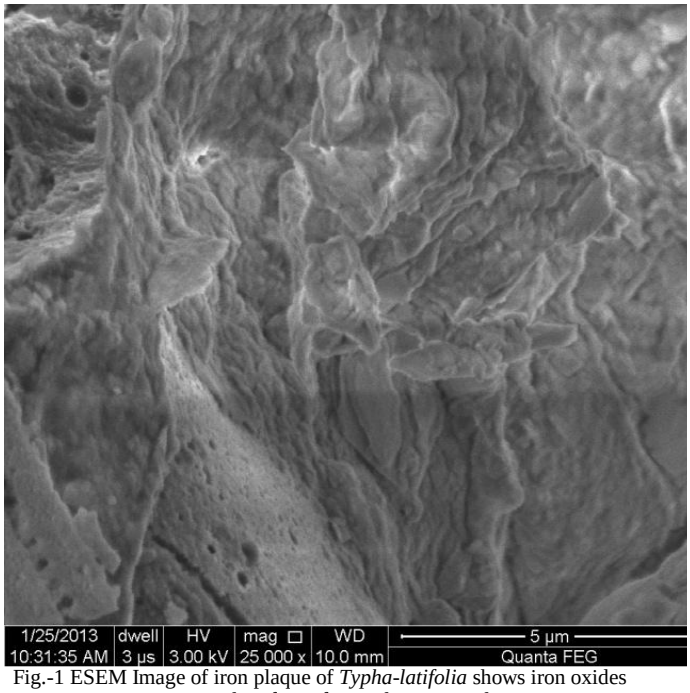

Fig.-1 ESEM Image of iron plaque of *Typha-latifolia* shows iron oxides on a crystal surface of iron plaque particle

Fig.-2 ESEM Image of iron plaque of *Typha-latifolia* shows iron oxides on a crystal surface of iron plaque particle

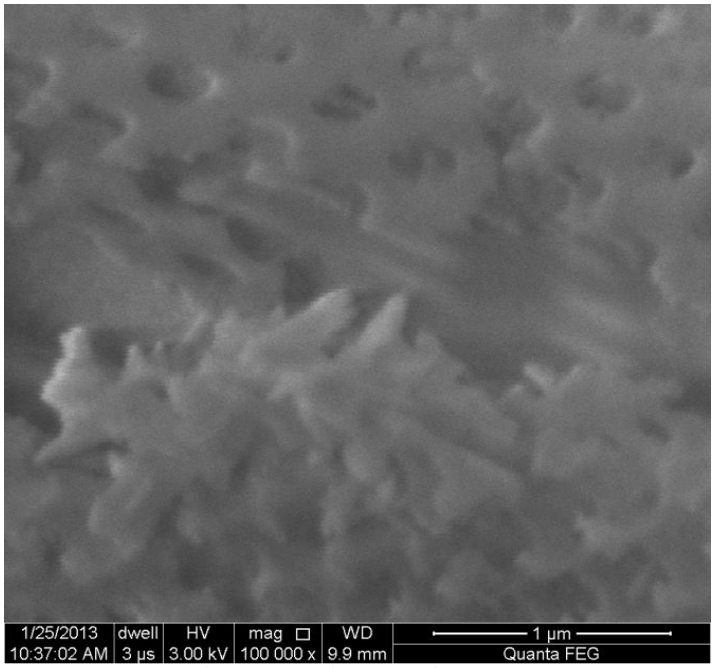

Fig.-3 ESEM Image of iron plaque of *Typha-latifolia* shows iron oxides on a crystal surface of iron plaque particle

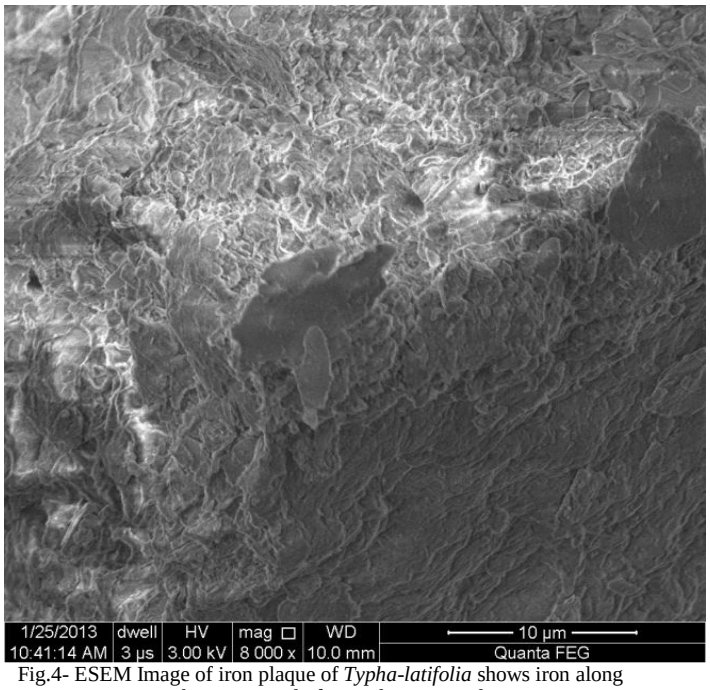

Fig.4- ESEM Image of iron plaque of *Typha-latifolia* shows iron along cracks in a crystal of iron plaque particle

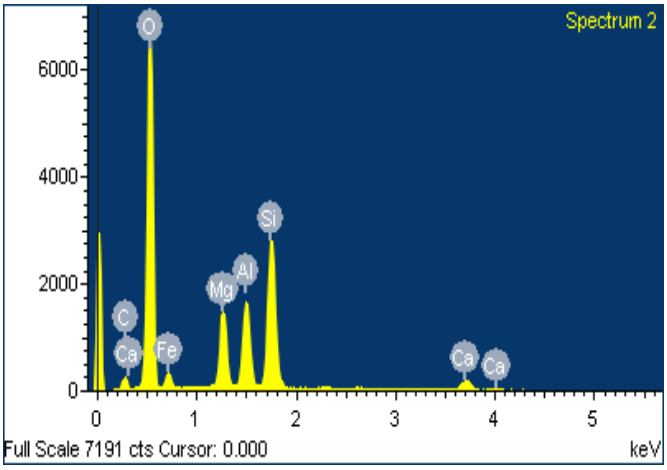

Fig.-5 EDS spectra of iron plaque of *Typha-latifolia*

Table-2 Normalized element composition wt % in iron plaque of *Typha-latifolia*

Element	Weight%	Atomic%
C	4.17	7.15
O	48.56	62.52
Mg	6.67	5.65
Al	7.87	6.01
Si	16.62	12.19
Ca	3.73	1.92
Fe	12.38	4.57
Totals	100.00	

D. Experimental Procedures

The batch sorption studies were carried out at room temperature [25°C ± 2°C] in an orbital shaker incubator at 170 rpm. The reaction mixture consists total volume of 100 ml of [Cr^{+6}] & Copper [Cu^{2+}] in separate conical flasks, containing initial chromium & copper concentrations of 0.5 mg/l and iron plaque of *Typha-latifolia*. To assess the impact of media size on [Cr^{+6}] uptake, experiments were performed with initial [Cr^{+6}] concentration of 0.5 mg/l and 20 g/l adsorbent dose for 1-4 hr. contact time with variation of media size from 1.18 mm to 150 µ. The conical flasks were removed from orbital shaker incubator after desired contact time and the supernatant was separated from adsorbent by 'Whatman' No 42 [ashless] filter paper. A series of batch experiments were conducted for four media size, i.e. 1.18 mm, 600µ, 300µ, 150µ for kinetic uptake of [Cr^{+6}] by iron plaque of *Typha-latifolia*. The effect of contact time was studied with 0.5 mg/l initial [Cr^{+6}] concentration & 20 g/l adsorbent dose; pH was kept 7 at varying contact time from 1-4 hr. In case of [Cu^{2+}] pH was 5.

E. Reagents and Stock Solutions

The potassium dichromate [$K_2Cr_2O_7$] [SRL, India] was used as main stock reagent. Stock Chromium [Cr^{+6}] solution of 500 mg/l was prepared by the dissolution of 141.4 mg potassium dichromate [$K_2Cr_2O_7$] in distilled water & dilute to 100 ml. The secondary standards for further experiments were freshly prepared for each experimental run from the stock solutions. Polished electrolytic copper foil was used as main stock reagent for stock Copper [Cu^{2+}]. Stock Copper [Cu^{2+}] solution of 200 mg/l was prepared by the dissolution of 200 mg polished electrolytic copper foil in a 250 ml conical flask, add 10 ml double distilled water and 5 ml Conc. HNO_3. After the reaction has slowed, warm gently to complete dissolution of the copper and boil to expel oxides of nitrogen, using precaution to avoid loss of copper, cool, add about 50 ml double distilled water, transfer quantitavely to a 1L volumetric flask, and dilute to the mark with water. *Source:* American Public Health Association (APHA).

F. Analytical Method for Aqueous Chromium [Cr^{+6}] & Copper [Cu^{2+}] Ions Determination

The concentrations of Chromium [Cr^{+6}] & Copper [Cu^{2+}] ions present in the solution were measured by a spectrophotometer [model Genesys-20, Thermo Spectronic, USA]. The maximum wavelength at which Chromium [Cr^{+6}] & Copper [Cu^{2+}] determined was 530 & 484 nm respectively. The calibration curves were then prepared by reading absorbance at the maximum wavelength against the known amount of the Chromium [Cr^{+6}] & Copper [Cu^{2+}] present in the solution. The pH values of the aqueous solution of Chromium [Cr^{+6}] & Copper [Cu^{2+}] were monitored by a digital pH meter [model 420A$^+$, Thermo Orion, USA].

III. RESULTS & DISCUSSION

Table-3 Chromium [Cr^{+6}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Media Size	Dose [gm]	C_0 of Cr^{+6} mg/l	Batch Time [hrs.]	C_t of Cr^{+6} mg/l	Removal R[%]	Adsorption Capacity [qe] mg/g
1.18 mm	2 gm	0.5 mg/l	1	0.28 mg/l	44 %	0.011
600 µ	2 gm	0.5 mg/l	1	0.257 mg/l	48.6 %	0.012
300 µ	2 gm	0.5 mg/l	1	0.18 mg/l	64 %	0.016
150 µ	2 gm	0.5 mg/l	1	0.165 mg/l	67 %	0.017

Table-4 Chromium [Cr^{+6}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Media Size	Dose [gm]	C_0 of Cr^{+6}m g/l	Batch Time [hrs.]	C_t of Cr^{+6} mg/l	Removal R[%]	Adsorption Capacity [qe] mg/g
1.18 mm	2 gm	0.5 mg/l	2	0.244 mg/l	51.2 %	0.013 mg/g
600 µ	2 gm	0.5 mg/l	2	0.20 mg/l	60 %	0.015 mg/g
300 µ	2 gm	0.5 mg/l	2	0.159 mg/l	68.2 %	0.017 mg/g
150 µ	2 gm	0.5 mg/l	2	0.134 mg/l	73.2 %	0.018 mg/g

Table-5 Chromium [Cr^{+6}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Media Size	Dose [gm]	C_0 of Cr^{+6} mg/l	Batch Time [hrs.]	C_t of Cr^{+6} mg/l	Removal R[%]	Adsorption Capacity [qe] mg/g
1.18 mm	2 gm	0.5 mg/l	3	0.153 mg/l	69.4 %	0.017 mg/g
600 µ	2 gm	0.5 mg/l	3	0.079 mg/l	84.2 %	0.021 mg/g
300 µ	2 gm	0.5 mg/l	3	0.055 mg/l	89.0 %	0.0222 mg/g
150 µ	2 gm	0.5 mg/l	3	0.042 mg/l	91.6 %	0.0229 mg/g

Table-6 Chromium [Cr^{+6}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Media Size	Dose [gm]	C_0 of Cr^{+6} mg/l	Batch Time [hrs.]	C_t of Cr^{+6} mg/l	Removal R[%]	Adsorption Capacity [qe] mg/g
1.18 mm	2 gm	0.5 mg/l	4	0.147 mg/l	70.6 %	0.017 mg/g
600 µ	2 gm	0.5 mg/l	4	0.079 mg/l	84.2 %	0.021 mg/g
300 µ	2 gm	0.5 mg/l	4	0.055 mg/l	89.0 %	0.0222 mg/g
150 µ	2 gm	0.5 mg/l	4	0.042 mg/l	91.6 %	0.0229 mg/g

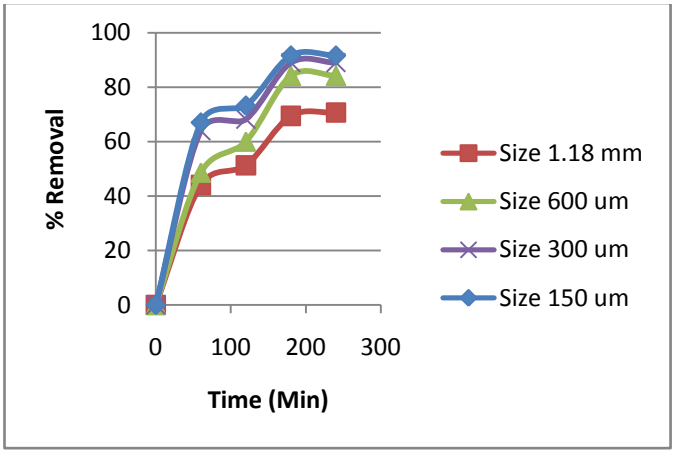

Fig.6 Effect of Media size on Chromium [Cr^{+6}] ion removal by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Table-7 Copper [Cu^{2+}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Iron plaque of *Typha-latifolia*] collected from wetland Raebareli.

Media Size [µ]	Dose [gm]	C_0 of Cu^{2+} mg/l	Batch Time [hrs.]	C_t of Cu^{2+} mg/l	Removal R[%]	Adsorption capacity [qe] mg/g
300 µ	1 gm	0.5 mg/l	3	0.18 mg/l	64 %	0.032 mg/g
150 µ	1 gm	0.5 mg/l	3	0.08 mg/l	84 %	0.042 mg/g
75 µ	1 gm	0.5 mg/l	3	0.019 mg/l	96.2 %	0.048mg/g

Table-8 Chromium [Cr^{+6}] ion removal efficiency in Batch Experiment by Natural Biogenic Iron Oxide [Bacterial precipitated $Fe[OH]_3$ rich soil] collected from wetland Raebareli

Media Size [µ]	Dose [gm]	C_0 of Cr^{+6} mg/l	Batch Time [hrs.]	C_t of Cr^{+6} mg/l	Removal R[%]	Adsorption capacity [qe] mg/g
600 µ	2 gm	0.5 mg/l	3	0.067 mg/l	86%	0.022 mg/g
300 µ	2 gm	0.5 mg/l	3	0.25 mg/l	50%	0.012 mg/g
150 µ	2 gm	0.5 mg/l	3	0.29 mg/l	42%	0.010 mg/g

The percentage of removed [Cr^{+6}] & [Cu^{2+}] ions R [%] in solution was calculated using Eq. [1]:

$$R\ [\%] = [C_0\text{-}C_t/C_0] \times 100 \qquad [1]$$

Where:

C_0, and C_t are the [Cr^{+6}] & [Cu^{2+}] concentrations in mg/l initially and at a given time t, respectively

The adsorption capacity [q_e] mg/g was determined using the mass balance expression Eq. [2] [Horsfall, et al. (2004)].

$$q_e = V(C_0\text{-}C_e)/M \qquad [2]$$

The adsorption capacity (qt) at time t was determined using Equation [3] [Demirbas, et.al. (2004)] given below.

$$q_t = V(C_0\text{-}C_t)/M \qquad [3]$$

Where:

C_0 is the initial metal ions concentration, C_e is the concentration of metal ions in solution [mg/l] at equilibrium, C_t is the concentration of metal ions in solution [mg/l] at time t, V is the volume of initial metal ions solution used [L] and M is mass of adsorbent used [g].

Natural biogenic iron oxide [iron plaque of *Typha-latifolia*] media have shown maximum Chromium [Cr^{+6}] & Copper [Cu^{2+}] uptake potential [91.6%, 96.2%] as compared to bacterial precipitated $Fe[OH]_3$ rich soil media [86%]. From the above results it has been concluded that copper & chromium removal increases as the media size [Iron plaque of *Typha-latifolia*] decreases & vice versa in case of bacterial precipitated $Fe[OH]_3$ rich soil media.

The adsorption capacity for Chromium [Cr^{+6}] & Copper [Cu^{2+}] on the iron plaque of *Typha-latifolia* increased with the decreasing of media size and the maximum uptake were 0.0229 mg Cr^{+6}/g, 0.048 mg Cu^{+2}/g at 298.15 K. In batch system, adsorption equilibrium of Chromium [Cr^{+6}] ion was attained at 180 min, the removal is then almost stabilizing after 180 min. implying that the equilibrium has been reached, and the optimal media size [iron plaque of *Typha-latifolia*] was 150µ for Chromium [Cr^{+6}] ion. The maximum sorption efficiencies at equilibrium was 70.6 %, 84.2 %, 89.0 % & 91.6 % for Chromium [Cr^{+6}] ion at 1.18 mm, 600 µ, 300 µ & 150 µ media size respectively. The optimal media size [iron plaque of *Typha-latifolia*] for Copper [Cu^{2+}] ion was found 75µ & the maximum sorption efficiencies was 64 %, 84 % & 96.2 % for 300µ, 150µ & 75µ media size respectively. And the adsorption capacity of Chromium [Cr^{+6}] ion on the bacterial precipitated $Fe[OH]_3$ rich soil increased with the increasing of media size, and the maximum adsorption

capacity for Chromium [Cr^{+6}] ion onto bacterial precipitated $Fe[OH]_3$ rich soil was 0.022 mgCr^{+6}/g at 298.15 K & the maximum sorption efficiency was 86%.

IV CONCLUSIONS

On the basis of above results it is concluded that a good Chromium [Cr^{+6}] & Copper [Cu^{2+}] ions sorption potential exist in tested natural biogenic iron oxide media. Iron plaque of *Typha-latifolia* can be used as low cost natural biomaterial for removal of heavy metals from water & wastewater. The laboratory experiment suggested that, there is possibility of treatment of contaminated surface water & wastewater, because the observed decrease in contaminants in treated wastewater. The laboratory experiment revealed that the process was pH, and contact time dependent. It is also concluded that iron plaque of *Typha-latifolia* has maximum uptake potential as compared to bacterial precipitated $Fe[OH]_3$ rich soil media. Chromium & copper removal increases as the media size decreases. The results obtained in this study demonstrate that iron plaque of *Typha-latifolia* a form of natural biogenic iron oxide can be utilized for removal heavy metals from wastewaters as a low cost adsorbent.

ACKNOWLEDEMENT

The Author [BK] grateful to Assistant Professor [Dr.N.R.Rawal] for permitting to prepare this paper, and for their valuable suggestion and guidance. Without this suggestion and guidance Author would not able to prepare this paper.

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