

Hexavalent chromium removal in a tannery industry wastewater using rice husk silica

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ABSTRACT: Present study dealt the removal of Cr(VI) in a tannery industry wastewater using rice husk silica powder as an adsorbent. The experimental investigations have been carried out by using rice husk silica powder for different adsorption dosage, different contact time and different pH against the initial Cr(VI) concentration of 292 mg/L. The maximum percentage removal of Cr(VI) in a tannery industrial wastewater (88.3 %) was found at an optimum adsorbent dosage of 15 g, contact time of 150 min., and pH of 4. Further, the experimental data on removal of Cr(VI) from tannery industry wastewater was validated with the Cr(VI) aqueous solution of same initial concentration of tannery industry wastewater against the optimum process parameters. The results of the validation experiment showed that the experiments conducted for the removal of Cr(VI) in a tannery industry wastewater may be reproducing capability for analyzing various parameters along with Cr(VI) based water and industry wastewater. The experimental data were fitted to Langmuir and Freundlich isotherm models. Isotherm models result indicated that the equilibrium data fitted well with the Langmuir isotherm than Freundlich isotherm, because of higher correlation created between dependent and independent variables. Thus, the adsorption method using rice husk silica powder was used effectively for removing Cr(VI) in the tannery industrial wastewater, seems to be an economical and worthwhile alternative over other conventional methods, because of their abundant source, low price, multi-purposes and antimicrobial properties.

Keywords: Adsorption, Isotherm models, Magnetic stirrer, Tannery industry wastewater, UV/VIS spectrophotometer

INTRODUCTION

Development of rapidly increasing industry and population growth, utilization of water for various purposes increases tremendously, as a result, sources of water are polluted by discharging wastewater from both industry and domestic sectors (Sivakumar Durairaj, 2013c). When compared to water pollution caused by domestic sector, pollution caused by industry sector contributes more (Olayinka *et al.*, 2007). Heavy metal contamination is a serious environmental issue of global concern, particularly in developing countries (Kleckerova and Doeekalova, 2014; Akoto *et al.*, 2014).

In the developing countries, heavy metals containing effluent are continuously released from thousands of small and large-scale industries such as metallurgy, battery manufacturing, mine drainage, chemical manufacturing, leather tanning and

electroplating industries etc. (Oladoja *et al.*, 2013; Rameshraj and Suresh, 2011) directly or indirectly into the natural water resources, mostly without proper treatment, posing a major threat to the environment.

Today, the heavy metals like Cu, Zn, Ni, Pb, Cd, and Cr into various sources of drinking water pose a great threat to public health. Among these heavy metals, pollution by chromium is of major concern as the metal is used in electroplating, leather tanning, metal finishing, and chromate preparation. Chromium is one of the heavy metals used by modern industries like plastic, pigment, wood preservative, electroplating, leather tanning, cement, mining, dyeing and fertilizer (Oladoja *et al.*, 2013). Among them, chrome tanning industry contributes more chromium pollution in water and land environment (Sudipta *et al.*, 2012). Chromium occurs in aqueous systems as trivalent and hexavalent forms. However, the latter form is of particular concern due to its carcinogenic and mutagenic effects (Oladoja

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et al., 2013). Human is facing the problems like gastric system, epigastric pain, nausea, vomiting, severe diarrhea, corrosion of the skin, respiratory tract and lung carcinoma when consumed the chromium polluted water. In addition to chromium, wastewater generated from these industries contains much higher concentrations of properties like colour, total dissolved solids, suspended solids, phenols, chlorides, ammonia, and other heavy metals (Sivakumar and Shankar, 2012a, 2012b). The discharged wastewater has generally contaminated the surface water and groundwater, results increased water pollution also soil is contaminated due to discharge wastewater (Sivakumar, 2011). The maximum permissible limit of chromium content in drinking water is 0.05 mg/L (WHO, 1996). Recently, increasing awareness of water and wastewater pollution and its far reaching effects has prompted concerted efforts towards pollution abatement.

Removal of Cr(VI) ions from industrial wastewater is achieved principally by the application of several conventional processes such as reduction followed by chemical precipitation (Karale *et al.*, 2007; Sarin and Pant, 2006), absorption (Sivakumar, 2013 a), activated carbon (Ghosh, 2009), electrochemical processes (Golder *et al.*, 2011), ion exchange (Rafati *et al.*, 2010; Balan *et al.*, 2013), biological operations (Sivakumar Durairaj, 2013c), catalytic oxidation (Kennedy *et al.*, 2004) and membrane processes (Sarin and Pant, 2006) etc. These methods require large amounts of chemical substances and energy, generation of toxic sludge, fouling, high capital and operational costs, expensive equipment requirement and efficient monitoring system (Demirbas *et al.*, 2004; Demiral *et al.*, 2008).

Adsorption is a simple and low cost method used for removing Cr(VI) from any aqueous solution (Chuah *et al.*, 2005; Gholami *et al.*, 2006; Sarin and Pant, 2006). The main advantages of the adsorption method are that single or multiple ions present in the any aqueous solution adhered onto porous solid surface and can be separately and quantitatively determined (Jal *et al.*, 2004; Oladoja *et al.*, 2013). The other advantages include low cost, high efficiency of metal removal from dilute solutions, minimization of chemical, biological sludge, no additional nutrient requirement and regeneration of absorbent and possibility of metal recovery (Sivakumar, 2013b). The various adsorbents have been used for removal of Cr(VI) in aqueous solution and industrial wastewater like silica gel (Katsumata *et al.*, 2003), river sand (Sharma and Weng, 2007), manganese-coated

sand (Lee *et al.*, 2010), fly ash (Sharma *et al.*, 2008), coconut shell (Pino *et al.*, 2006), wheat bran (Nameni *et al.*, 2008), rice bran (Oliveira *et al.*, 2005), etc.

This study mainly concentrated on removal of Cr(VI) from tannery industry waster than aqueous solution using rice husk silica. Rice husk silica is derived from the rice husk. Rice husk is the by-product of the rice milling industry. RHA is light in weight and has approximately 15-20 % of paddy weight (Liou and Wu, 2009). Rice husk ash is produced from the combustion of rice husk. The colour of RHA varied from white to gray (Muhammad *et al.*, 2013). Rice husk is commonly used as a low-value energy resource, burned in the field, and or discarded into the environment (Chen *et al.*, 2011; Lin *et al.*, 2013). Rice husk mainly consists of ash and organic matter, which comprises an organic cellulose, hemi-cellulose, lignin (Liou and Wu, 2010; Wang *et al.*, 2010) and inorganic silicon dioxide (Wang *et al.*, 2010). Further, Farook, *et al.*, 2006 reported that rice husk is composed of 20 % ash, 32.2-38 % cellulose (Mahvi *et al.*, 2004; Chuah *et al.*, 2005), 21.3-22 % lignin (Mahvi *et al.*, 2004; Chuah *et al.*, 2005), 21.4 % hemi-cellose (Mahvi *et al.*, 2004; Chuah *et al.*, 2005), 18 % pentose, and 2 % of other organic components (Adam *et al.*, 2006). As rice husk is insoluble in water, having good chemical stability, structural strength due to the high silica content (Lee *et al.*, 1994).

RHA, produced after the burning of Rice husks (RH) has high reactivity and pozzolanic property (Tomas, 2013). Rice husk ash is used as supplemental material for cement and concrete cube manufacturing industries (Della *et al.*, 2002). It is also used as additives for soil stabilization, as adsorbent for removal of contaminants from contaminated water and wastewater. Preparation of useful products from rice husk eliminates the disposal problem. Rice husk ash contains nearly 90-96 % of silica (Thankappan *et al.*, 2006), which may be used as one of the ingredients for making ceramic (Farook *et al.*, 2006; Liou and Wu, 2009), polymers and electronic products, it has highly porous and light in weight, with a very high external surface area (Kumar *et al.*, 2013). Silica was extracted from rice husk ash (RHA) and used to produce a corrosion inhibitor for carbon steel (Denni *et al.*, 2013). Because of easy to convert organic components into carbonaceous substances from rice husk (Kennedy *et al.*, 2004; Chen *et al.*, 2013), a greater attention has been made nowadays by many researchers. There are several studies used rice husk derived adsorbent (rice husk, rice husk carbon, rice husk ash and rice husk silica) for

removal of Cr (VI) from aqueous solution (Srinivasan *et al.*, 1988; Della *et al.*, 2002; Bhattacharya *et al.*, 2006; Singha *et al.*, 2011; Singh and Singh, 2012; Anand *et al.*, 2014; Apeksha and Mote, 2014).

Thus, this study focused to investigate the effect of adsorption dosage, contact time and pH against the initial concentration of Cr(VI) in a tannery industry wastewater using rice husk silica powder. The experimental results of Cr(VI) in a tannery industry wastewater using rice husk silica powder were validated with the Cr(VI) in an aqueous solution and other parameters in a tannery industry wastewater. Further, the experimental data were fitted to Langmuir and Freundlich isotherm models for determining the suitability of rice husk silica for removing Cr(VI) in a tannery industry wastewater. All experiments were conducted in the Environmental Engineering Laboratory, Department of Civil Engineering, Vel Tech High Tech Dr.Rangarajan Dr.Sakunthala Engineering College, Avadi, Chennai, Tamil Nadu, INDIA, during December 2013.

MATERIALS AND METHODS

Study Area

The selected study area of this present study is Nagalkeni, which is situated in Kanchipuram District, Tamil Nadu with 12.96 Latitude and 80.14 Longitude (Fig. 1).The groundwater of Nagalkeni is polluted by untreated sewage and wastewater from tannery

industry. Furthermore, the porous media (the soil) in Nagalkeni are also contaminated, when untreated sewage and wastewater from tannery industry is discharged on the land. The tannery industry wastewater contained heavy metals, particularly Cr (VI), which is carcinogenic to human beings when the concentration of Cr(VI) exceed the tolerance limit of 0.05 mg/L (BIS drinking water quality standard - IS 10500:1991).

Adsorbent Preparation

Rice husk collected from rice mill was mixed together to form a homogeneous mixture. Then, a homogeneous mixture of rice husk was thoroughly washed with tap water to clean dirt and mud. Silica may be obtained from rice husk through different methods (Nittaya and Apinon, 2008; Tzong-Horng Liou, 2004; Della *et al.*, 2002). In this paper, the cleaned rice husk was mixed with 1.0 M HCl in the ratio of 100 g rice husk per 1 litre acid and heated to the temperature of 100 °C for 2 hours. At the end of 2 hours reaction, the trace of HCl acid within the treated rice husk was removed by washing with tap water. Then, it was dried in an oven at 130 °C for 24 hours. During, this period, rice husk ash was obtained from the rice husk and the colour of rice husk ash is in black. Further, the rice husk ash was heated to a temperature of 650 °C for 6 hours to obtain silica.

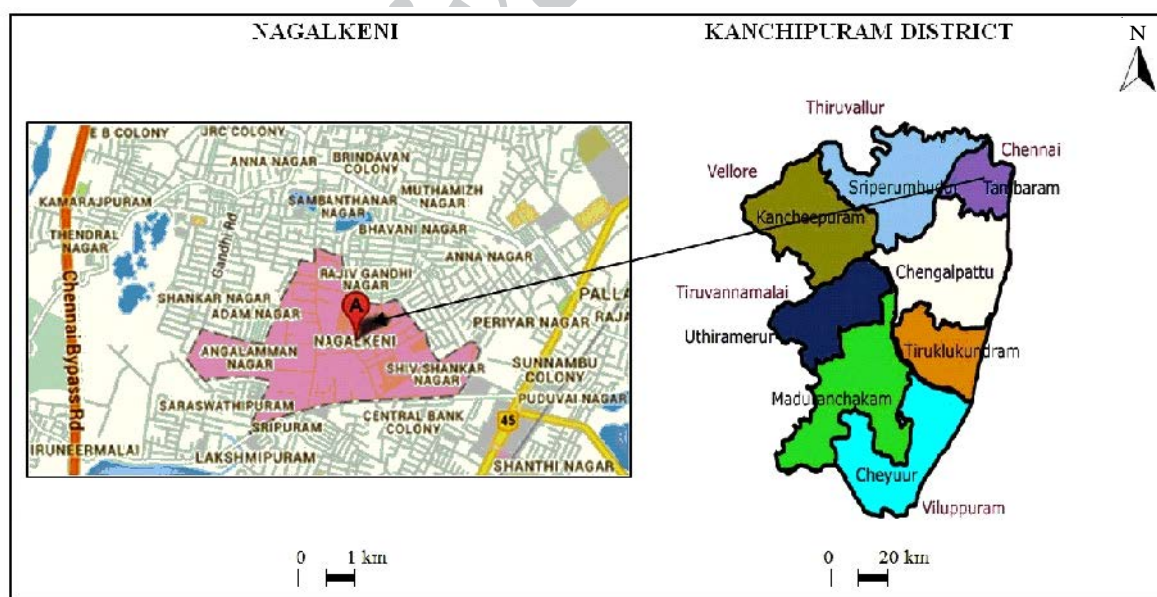


Fig.1: Study area of Nagalkeni

Rice husk silica was produced by a sol-gel method (Qu *et al.*, 2010). Oladoja *et al.*, (2013) and Farook *et al.*, (2006) obtained the silica from rice husk by thermal treatment with the temperature of 700 °C for 5 hours. In this study, silica is prepared by physical activation, i.e., gasification of chars in an oxidizing atmosphere (Swarna latha *et al.*, 2013) followed by chemical activation, i.e., carbonization of carbonaceous materials impregnated with chemical reagents (Oladoja *et al.*, 2013; Farook *et al.*, 2006). The obtained silica from the rice husk was white in colour. Since the heating temperature of 300 °C for 1 h, the ash produced from the rice husk found to be black in colour (Fig. 2). The colour of the rice husk ash turned from black to blackish white, gray and white respectively, for the temperature 400 °C (Fig. 3), 500 °C

(Fig. 4) and 650 °C (Fig. 5). The colour changes from blackish white, gray and white was achieved for the time period of 2, 4 and 6 h respectively. The black colour of rice husk ash contains carbon content of 64.8 % (300 °C for 1 h), the blackish white colour of rice husk ash contains carbon content of 37.3 % (400 °C for 2 h), gray colour of rice husk ash contains carbon content of 15.9 % (500 °C for 4 h), and white colour of rice husk ash contains carbon content of 0.26 % (650 °C for 6 h). The same result was observed by Della *et al.*, (2002) and Oladoja *et al.*, (2013). The rice husk silica was further grained to fine particles of uniform geometrical size of 0.10 mm, which were used for treating removal of Cr(VI) from tannery industry wastewater.



Fig. 2: Rice husk carbon (300 °C for 1 h)



Fig. 3: Rice husk ash (400 °C for 2 h)



Fig. 4: Rice husk ash (500 °C for 4 h)



Fig. 5: Rice husk silica (650 °C for 6 h)

The physical and chemical properties of rice husk silica were presented in Table 1. The specific surface area was determined by the nitrogen adsorption according to B.E.T. equation (Della *et al.*, 2002; Nittaya and Apinon, 2008). The pH was measured using a pH meter (Sivakumar, 2012). The moisture content was determined by the oven dry weight method and density was determined using Pycnometer (Punmia, 2005). APHA, (2005) procedures were followed for the determination of chemical properties in a tannery industry wastewater.

From Table 1, it may be observed that silica contributes more fraction than other constituents of the rice husk ash. Della *et al.*, (2002) reported that the silica presented in the rice husk ash is about 94.95 %, which was achieved when the rice husk was calcined at 700 °C for 5 h. Nittaya and Apinon, (2008) produced the silica of 90.3 % from rice husk, when rice husk being treated by 2.5 N sodium hydroxide with 700 °C for 3 h and 98.14% silica was achieved with the heat treatment of 700 °C for 6 h.

Collection of Wastewater Sample

Totally five wastewater samples were collected without the presence of bubbles using cleaned air tight plastic bottles, took to the laboratory and then they were stored for analyzing Cr(VI) and other basic parameters. Various physico-chemical parameters such as pH, total dissolved solids (TDS), total solids (TS), chemical oxygen demand (COD), biochemical oxygen demand (BOD), and sulphate (SO_4^{2-}) in a homogenized wastewater samples were analyzed before and after

treating with rice husk silica by using standard procedures prescribed in APHA, 2005. The Cr(VI) was also measured in a homogenized wastewater sample before and after treating with rice husk silica using UV/VIS spectrophotometer (APHA, 2005). In this method, first prepared stock solution by dissolving 141.4 mg of dried potassium dichromate in distilled water of 100 mL and kept in a volumetric flask and by dissolving 250 mg 1,5 Diphenyl carbazide in 50 mL acetone in a dark brown coloured bottle to prepare a stock solution. Prepared a series of reference solutions by pipetting known quantity of chromium stock solution (0.02, 0.04, 0.06, 0.08, 0.1, 0.12, 0.14, 0.16 mg/L) into 100 mL volumetric flasks. Adjust pH of the solution to 2.0 ± 0.5 with 0.25 mL phosphoric acid and 0.2 N sulfuric acid, mix well and dilute with distilled water. In order to develop colour, added 2.0 mL of Diphenyl carbazide to the solutions, mix well and allow the solutions to stand for 10 min. Then the absorbance measurements were performed at the wavelength of 540 nm using the Perkin Elmer UV/VIS spectrophotometer. The physico-chemical characteristics and Cr(VI) in a tannery industry wastewater are given in Table 2.

Experimental Arrangement

The adsorption capacity of the rice husk silica powder on removal of Cr(VI) in a tannery industry wastewater was performed in this present study. The size silica particle used in this study was 0.10 mm. The experiments were conducted for different adsorbent dosage of 5, 10, 15, 20 g, contact time of 30, 60, 90, 120, 150, 180 min., agitator speed of 250, 500, 750, 1000 rpm, dilution ratio of 0, 1, 2, 3, 4, 5 and pH of 2, 3, 4, 5, 6, 7, 8 and 9 (different process parameters). Tannery industry wastewater was taken in 4 glass beakers of 250 ml capacity and was kept on the magnetic stirrer

Table 1: Physical and chemical properties of rice husk silica

Properties	Values
Physical Properties	
Specific surface, m^2/g	186
pH	6.0
Moisture content, %	0.7
Density, g/cm^3	2.20
Chemical Properties	
SiO_2	95.3 %
Al_2O_3	0.3 %
CaO	1.3 %
NaO	0.45 %
K_2O	1.4 %
Fe_2O_3	0.5 %
MgO	0.8 %
Loss on Ignition	0.3 %
Other Impurities	0.15 %

Table 2: Characteristics of tannery industry wastewater

Sl.No.	Characteristics	Values
1	pH	7.6
2	TDS	12350 mg/L
3	TS	16625 mg/L
4	COD	8256 mg/L
5	BOD	5689 mg/L
6	Sulphate	852 mg/L
7	Chromium	292 mg/L

apparatus for different adsorbent dosage, different contact time, different agitations speeds, different concentration dilutions and different pH. The treated tannery industry wastewater with rice husk silica against various process parameters was allowed to settle for 24 h, and then it was filtered. Further, the reduction in concentrations of Cr(VI) in a tannery industry wastewater was analyzed by a digital UV/VIS spectrophotometer. The other physico-chemical parameters also determined by using the standard procedure stipulated by APHA, 2005. The adsorption removal percentage of various parameters such as Cr(VI), pH, TDS, TS, COD, BOD, and SO_4^{2-} in a tannery industry wastewater by rice husk silica powder was calculated by using the following formula:

$$\text{Percentage Removal} = \frac{(c_0 - c_t)}{c_0} * 100 \quad (1)$$

in which C_0 is the concentration of Cr(VI), pH, TDS, TS, COD, BOD, and SO_4^{2-} in a tannery industry wastewater before treatment (time $t = 0$ min.) with rice husk silica powder and C_t is the concentration of Cr(VI), pH, TDS, TS, COD, BOD, and SO_4^{2-} in a tannery industry wastewater after treatment with rice husk silica powder ($t = t$ min.). This study mainly focuses on removal of Cr(VI) in a tannery industry wastewater rather than other physico-chemical parameters. But, the other physico-chemical parameters are used to check the validity of rice husk silica powder for the removal of Cr(VI) in a tannery industry wastewater. Thus, the equilibrium experimental data of Cr(VI) are used to fit the isotherm models. Using a mass balance, the concentrations of Cr(VI) at different time adsorbed by rice husk silica powder was calculated as

$$q_t = \frac{(c_i - c_t) V}{M} \quad (2)$$

in which, q_t is the amount of Cr(VI) adsorbed by the rice husk silica powder at time t , C_i is the initial concentration of Cr(VI), V is the volume of the aqueous phase, M is the weight of rice husk silica powder.

RESULTS AND DISCUSSION

In the present study, Cr(VI) in a tannery industrial wastewater was reduced using the method called adsorption. The selected process parameters of this adsorption method are different adsorption dosage, different contact time and different pH. The results are presented below.

Effect of adsorption dosage

The Fig. 6 shows the effect of adsorption dosage in the removal of Cr(VI) from tannery industry wastewater. The selected adsorption dosage was 5, 10, 15 and 20 g for the contact time of 90 min., and pH of 7 against an initial Cr(VI) concentration of 292 mg/L ('0' dilution ratio). From Fig. 6, it may be observed that the percentage removal of concentration of Cr(VI) by rice husk silica powder with the adsorption dosage of 5, 10, 15 and 20 g was found to be 47.4, 56.3 and 64.5 and 60.8 % respectively. From Fig. 7, it may be observed that till the adsorbent dosage reaches to 15.0 g, the removal percentage of Cr(VI) in a tannery industry wastewater increased, beyond which removal percentage got decreased. The less removal rate of Cr(VI) in a tannery industry wastewater for the dosage of 5.0 g, 10.0 g is due to less availability of specific surface area of rice husk silica. Further, the less removal rate of Cr(VI) in a tannery industry wastewater for the dosage of 20.0 g is due to the agglomeration of rice husk silica, results, which is not exposing the surface area to adhere the Cr(VI) from tannery industry wastewater (Sivakumar and Shankar, 2012). Thus, optimum adsorption dosage for which, maximum removal of Cr(VI) in tannery industry wastewater (64.5 %) is found to be 15 g (Fig. 6).

Effect of contact time

The Fig. 6 shows the effect of contact time of removal of Cr(VI) in a tannery industry wastewater. The selected contact time was 30, 60, 90, 120, 150, and 180 min. for an optimum adsorption dosage of 15 g, and pH of 7 against an initial Cr(VI) concentration of 292 mg/L ('0' dilution ratio). From Fig. 7, it may be observed that the percentage removal of concentration of Cr(VI) by rice husk silica powder with the contact time of 30, 60, 90, 120, 150, and 180 min. was found to be 34.5, 46.5, 54.6, 62.8, 68.4 and 64.6 % respectively. From Fig. 3, it may be observed that till the contact time reaches to 150 min., the removal percentage of Cr(VI) in a tannery industry wastewater increased, beyond which removal percentage got decreased. The less removal rate of Cr(VI) in a tannery industry wastewater for the contact time of 30, 60, 90, 120 min. is due to the larger surface area of rice husk silica was not contacted properly with the tannery industry wastewater. Further, the less removal rate of Cr(VI) in a tannery industry wastewater for the contact time 180 min. is due to presence of exhausted sites on the surface of rice husk silica, results,

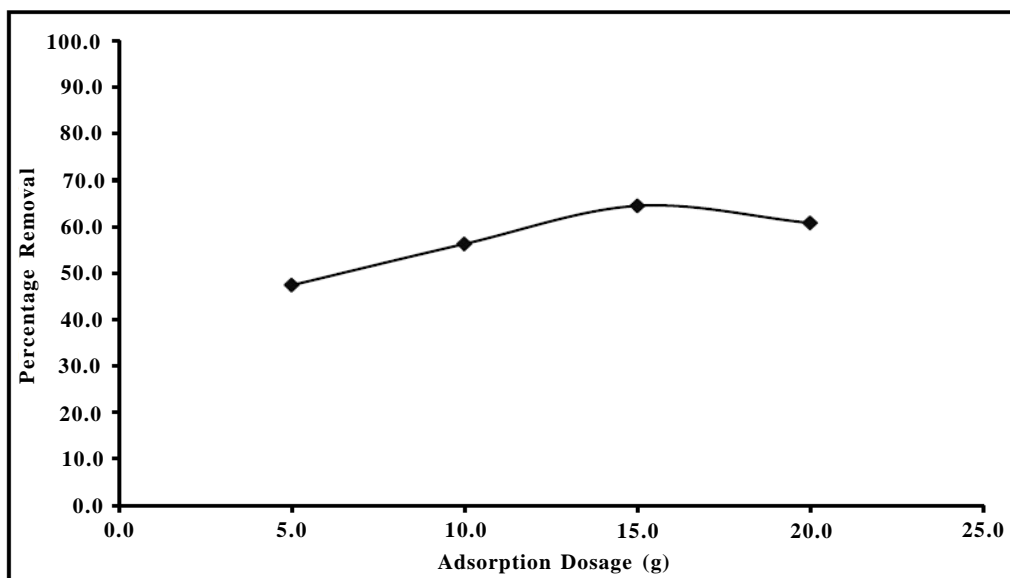


Fig. 6: Effect of adsorption dosage of removal of Cr(VI) in a tannery industry wastewater

the adsorbent is transported from the exterior to the interior sites of the adsorbent particles (Sivakumar and Shankar, 2012). Thus, optimum contact time for which, maximum removal of Cr(VI) in tannery industry wastewater (68.4 %) is found to be 150 min. (Fig. 7).

Effect of pH

The Fig. 8 shows the effect of pH on removal of Cr(VI) in a tannery industry wastewater. The selected pH ranges was 2, 3, 4, 5, 6, 7, 8, and 9 for an optimum adsorption dosage of 15 g and optimum contact time of 150 min. against an initial Cr(VI) concentration of 292 mg/L ('0' dilution ratio). From Fig. 8, it may be observed that the percentage removal of concentration of Cr(VI) by rice husk silica powder with the pH range of 2, 3, 4, 5, 6, 7, 8 and 9 was found to be 62.3, 79.6, 88.3, 84.6, 80.3, 76.4, 73.1 and 69.2 % respectively. From the Fig. 4, it may be observed that at low pH, hydrogen ions in the tannery industry wastewater were not allowing the Cr(VI) ion to adhere with the binding sites of rice husk silica. At high pH, a tannery industry wastewater becomes basic and rice husk silica released more hydroxyl groups making the tannery industry wastewater more basic (Sivakumar, 2013); as a result, Cr(VI) ion was not adhering into the binding sites of adsorbents. Thus, an optimum pH value, for which, maximum removal of Cr(VI) in a tannery industry wastewater (88.3 %) is found to be 4 (Fig. 8).

Validation of Experiment

Validation of experiment is required to verify the finding of similarities against the observed values of an experimental investigation. This validation experiment is used to check the degree of similarity between the adsorption experiments conducted at an optimum adsorbent dosage of 15 g, optimum contact time of 150 min. and optimum pH of 4 against an initial Cr(VI) concentration of 292 mg/L in a tannery industry wastewater using rice husk silica powder with the new experiments (separate experiments) conducted against the same optimum values of adsorbent dosage, contact time, pH against an initial Cr(VI) concentration of 292 mg/L in an aqueous solution using rice husk silica powder. The maximum removal of Cr(VI) in a tannery industry wastewater and an aqueous solution using rice husk silica powder at optimum values of selected parameters is shown in Fig. 9. The results of validation experiments showed that the maximum removal percentage of Cr(VI) in an aqueous solution using rice husk silica powder at the optimum conditions is greater than (93.5 %) the results of Cr(VI) removal in a tannery industry wastewater (88.3 %) by rice husk silica powder. From Fig. 5, it may be noted that the high values observed in a Cr(VI) aqueous solution than in a tannery industry wastewater is due to there are no competitive ions present in aqueous solution than in a

Hexavalent chromium removal

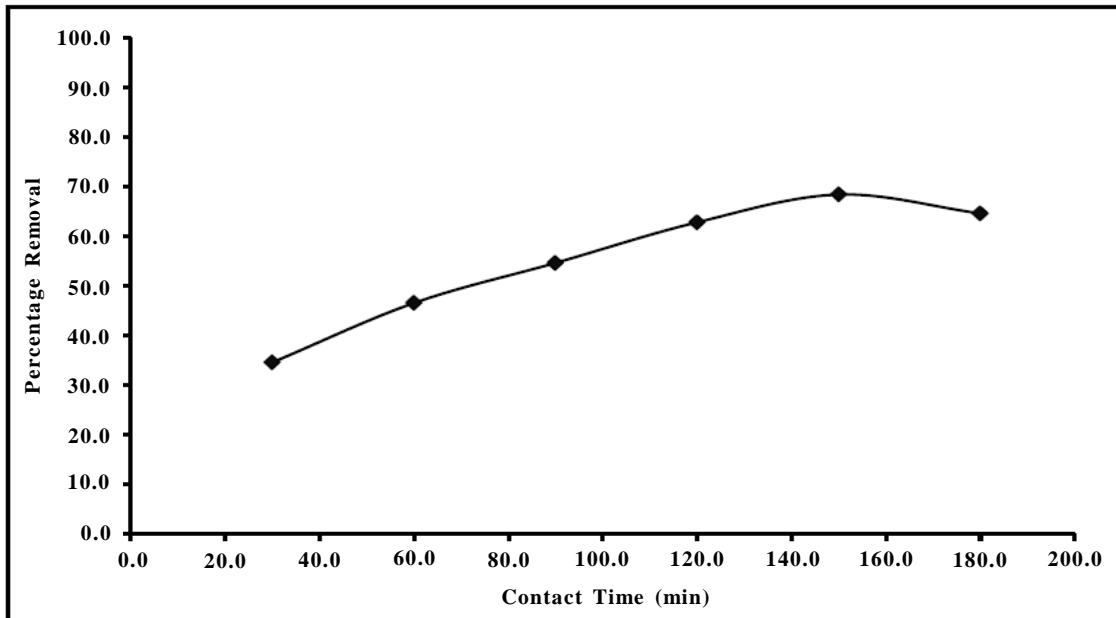


Fig. 7: Effect of contact time on removal of Cr(VI) in a tannery industry wastewater

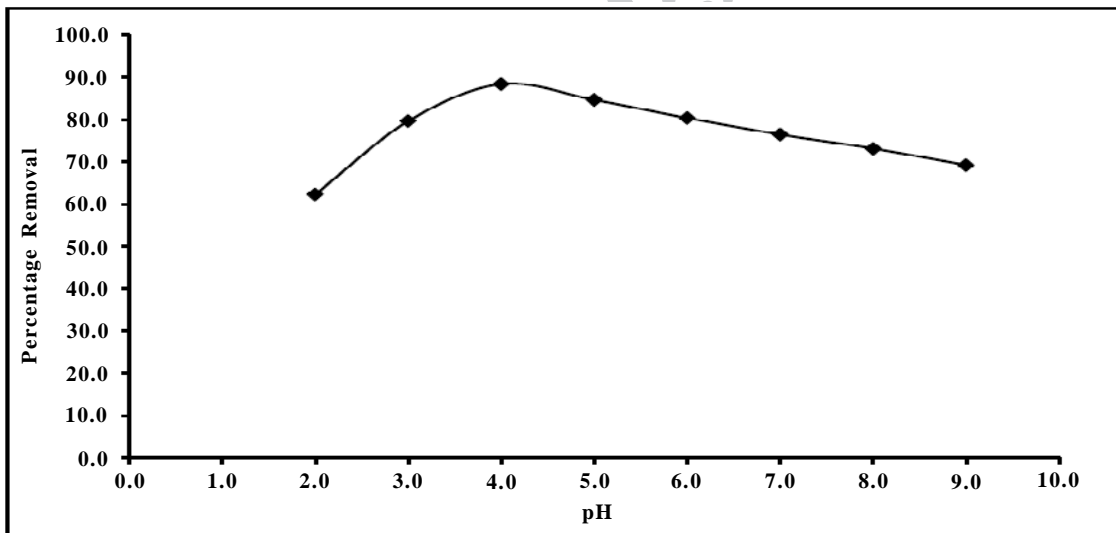


Fig. 8: Effect of pH on removal of Cr(VI) in a tannery industry wastewater

tannery industry wastewater, where the competitive ions like chloride, sulphate, acids, alkaline and other trace metals are presented.

Further, the obtained maximum removal percentage of Cr(VI) in a tannery industry wastewater using rice

husk silica powder with an optimum adsorbent dosage of 15 g, an optimum contact time of 150 min. and optimum pH of 4 against an initial Cr(VI) concentration of 292 mg/L was verified with the other physico-chemical parameters TDS, TS, COD, BOD, and SO_4^{2-} in

a tannery industry wastewater. The results of maximum reduction of various parameters TDS, TS, COD, BOD, and SO_4^{2-} along with reduction of Cr(VI) in a tannery industry wastewater are presented in Table 3. The other values of adsorbent dosage, contact time and pH were not presented in Table 3.

From Table 3, it may be noted that the maximum removal of various parameters TDS, TS, COD, BOD, and SO_4^{2-} in a tannery industry wastewater was obtained at the same an optimum adsorbent dosage of 15 g, an optimum contact time of 150 min. and optimum pH of 4 observed for Cr(VI) removal in a tannery industry wastewater. Thus, the validation results (Fig.

5 and Table 3) indicated that the optimum values found from this study were reproducing capability for analyzing various parameters in an aqueous solution and in any type of industrial wastewaters. Swathi *et al.*, (2014) found that COD reduction of 68 %, sulphate reduction of 67 %, BOD reduction of 73% and iron reduction of 81% in a tannery industry wastewater using rice husk as an adsorbent. The reduction of COD, BOD, and SO_4^{2-} in a tannery industry wastewater using rice husk ash is found to be 81.5 %, 86.7 % and 93.4 % respectively (Table 3). The results of the present study show a maximum reduction (Table 3) than the previous study (Swathi *et al.*, 2014).

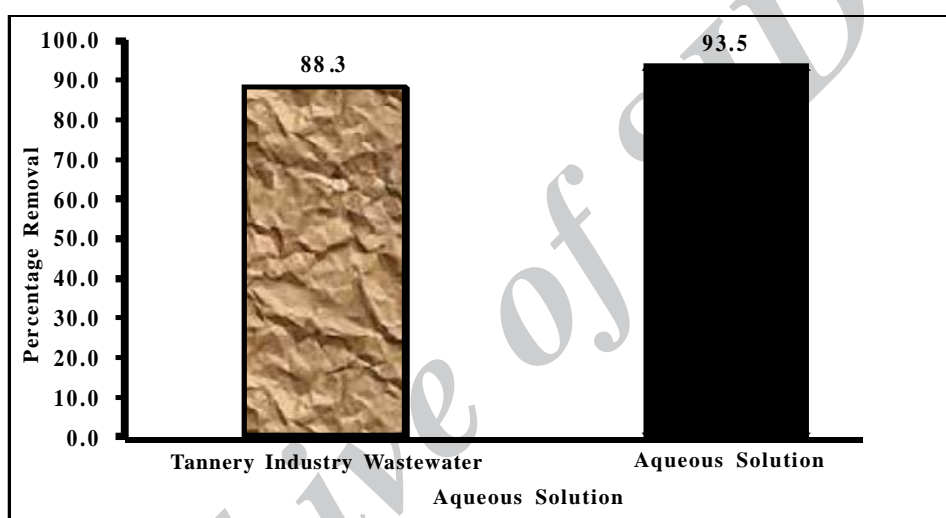


Fig. 9: The Maximum Removal Percentage of Cr(VI) in a tannery industry wastewater and Cr(VI) in an aqueous solution using rice husk silica powder with an optimum adsorbent dosage of 15 g, an optimum contact time of 150 min. and optimum pH of 4 against an initial Cr(VI) concentration of 292 mg/L

Table 3: The maximum removal percentage of Cr(VI) and other physico-chemical parameters in a tannery industry wastewater by rice husk silica powder with an optimum adsorbent dosage of 15 g, an optimum contact time of 150 min. and optimum pH of 4

Parameters	Initial	Final	Percentage Removal
Cr(VI)	292 mg/L	34 mg/L	88.3
TDS	12350 mg/L	1074mg/L	91.3
TS	16625 mg/L	1031mg/L	93.8
COD	8256 mg/L	1362mg/L	81.5
BOD	5689 mg/L	757mg/L	86.7
SO_4^{2-}	852 mg/L	56mg/L	93.4

Isotherm Study

Adsorption isotherms are describing the distribution of the adsorbate species among liquid phase and adsorbed phase, based on heterogeneity or homogeneity nature of adsorbents, type of coverage and an interaction between the adsorbate species. Adsorption data are usually described by adsorption isotherms, such as Langmuir and Freundlich isotherms. These isotherms relate metal uptake per unit mass of adsorbent, q_e , to the equilibrium adsorbate concentration in the bulk fluid phase C_e .

The Langmuir model is based on the assumption that the maximum adsorption occurs when a saturated monolayer of solute molecules is present on the adsorbent surface, the energy of adsorption is constant and there is no migration of adsorbate molecules in the surface plane (Bhattacharya *et al.*, 2006). The Linear form of Langmuir isotherm is written as;

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \quad (3)$$

The Eqn. 3, may be rewritten as

$$\frac{1}{q_e} = \frac{1}{q_m b C_e} + \frac{1}{q_m} \quad (4)$$

where, C_e , q_e , q_m and b are representing the equilibrium concentration of Cr(VI), adsorption capacity of rice husk silica at equilibrium, maximum adsorption capacity of rice husk silica and energy constant related to the heat of adsorption (Langmuir

constant) respectively. The q_m and b in the Langmuir isotherm can be determined by plotting (C_e/q_e) versus (C_e) .

It can be seen from Fig. 10 that the equilibrium data fits the Langmuir equation well for Cr(VI) removal in a tannery industry wastewater. From Fig. 6, the values of the q_m and b were found to be 78.125 mg/g and 0.0634 L/mg for the parameter Cr(VI) in a tannery industry wastewater. The separation factor or equilibrium parameter R_L is the essential characteristics (Bhattacharya *et al.*, 2006), which is dimensionless constant, represented as;

$$R_L = \frac{1}{(1+b C_o)} \quad (5)$$

In which, C_o is the highest initial concentration of Cr(VI) (mg/L) and b (L/mg) is Langmuir constant. The R_L value indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). From Fig. 6, it was found that the R_L value is 0.05125, the results present study was found to be favorable for adsorption by rice husk silica powder for the removal of Cr(VI) in a tannery industry wastewater.

The Freundlich isotherm model is describing the adsorption of solutes from an aqueous phase to a solid surface. The Freundlich isotherm assumes several adsorption energies are involved (Bhattacharya *et al.*, 2006) in the adsorption process. Freundlich adsorption isotherm is the relationship between the amounts adsorbed per unit mass of adsorbent, q_e , and the

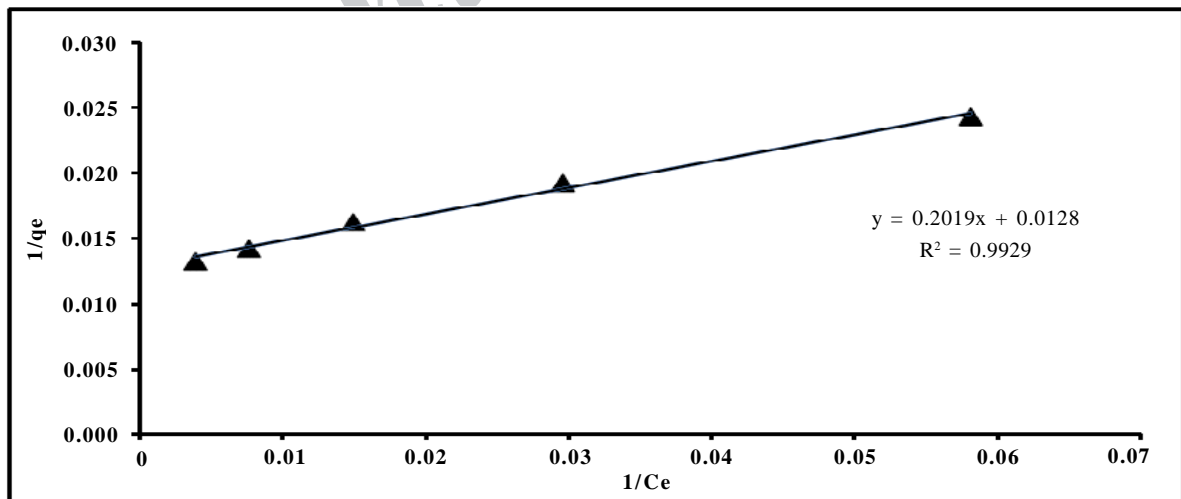


Fig. 10: Langmuir adsorption isotherm for removal of Cr(VI) in a tannery industry wastewater

concentration of Cr(VI) at an equilibrium, C_e . The linear form of logarithmic equation for Freundlich isotherm is written as;

$$\log(q_e) = \log(k_f) + \frac{1}{n} \log(c_e) \quad (6)$$

where, K_f and n are the Freundlich constants, K_f and n are the indicators of the adsorption capacity and adsorption intensity respectively. In this case, the plot of $\log(C_e)$ vs $\log(q_e)$ was employed to generate the intercept value of K_f and the slope of n .

The value of n indicates how the adsorption process is favourable for the removal of Cr(VI) in a tannery industry wastewater. The value of $n > 1$ represents the favourable adsorption condition. From Fig. 11, it may be noted that the equilibrium data obtained for the Cr(VI) removal in a tannery industry wastewater fitted with Freundlich isotherm. The values of K_f and n were found to be 3.8963 and 4.4603 respectively, for the removal of Cr(VI) in a tannery industry wastewater.

In order to measure the degree of relationship between two or more variables, the correlation and regression analysis were performed for all parameters (Sivakumar, 2012). From Fig. 11, it may be found that n value is greater than 1, indicates favourable adsorption process by rice husk silica powder. From Figs. 10 and 11, it may be observed that the correlation coefficient R^2 found to be 0.9929 and 0.9615 respectively for the Langmuir and Freundlich isotherms. The results of R^2 indicated that the equilibrium data of Cr(VI) removal in a tannery industry wastewater by rice husk silica fitted well with the Langmuir isotherm than Freundlich

isotherm. The same observations were made by Khadka and Mishra, (2014). The previous studies also indicated the same results as that of the present study. Apeksha and Mote, (2014) also obtained the R^2 value around 0.99 for Langmuir isotherm than Freundlich isotherm (0.98). Similarly, Singha *et al.*, (2011) also achieved the R^2 value of 0.9869 for the Langmuir isotherm.

The maximum removal of Cr(VI) was achieved at the pH of 1.5 using rice husk (Singha *et al.*, 2011). The maximum removal of Cr(VI) from an aqueous solution using rice husk ash occurred at a pH of 3 (Bhattacharya *et al.*, 2006) and 4 (Anand *et al.*, 2014). Whereas, this study removed the maximum Cr(VI) at the pH of 4 using rice husk silica powder in a tannery industry wastewater.

The maximum removal of Cr(VI) from an aqueous solution by rice husk ash was found to be 25.64 mg/g (Bhattacharya *et al.*, 2006). Nearly 45.6 mg/g removal was achieved by Srinivasan *et al.*, (1988), when rice husk carbon was used for the removal Cr(VI) in an aqueous solution, but Singh and Singh, (2012) 38.8 mg/g removal of Cr(VI) from an aqueous solution using rice husk carbon. Apeksha and Mote, (2014) obtained 16.94 mg/g removal of Cr(VI) in an aqueous solution using rice husk and 11.11 mg/g of Cr(VI) removal was achieved using rice husk ash (Apeksha and Mote, 2014). The maximum of 11.39 mg/g removal of Cr(VI) in an aqueous solution using rice husk was achieved by Singha *et al.*, (2011). Whereas this study removed the maximum Cr(VI) of 78.125 mg/g using rice husk silica powder in a tannery industry wastewater.

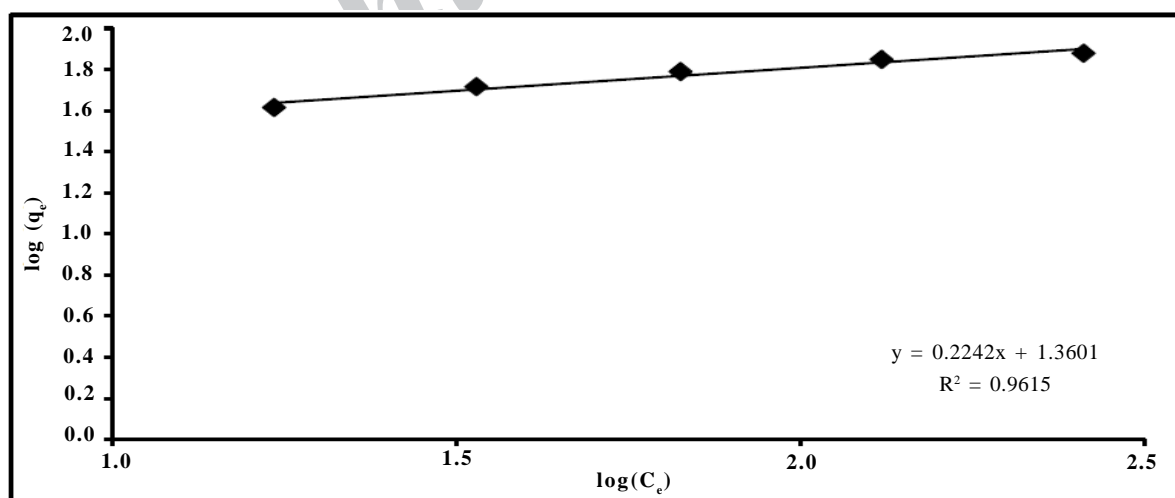


Fig. 11: Freundlich adsorption isotherm for removal of Cr(VI) in a tannery industry wastewater

About 90 % removal of Cr(VI) in an aqueous solution using rice husk ash was achieved at the pH of 4 by Anand *et al.*, (2014). Whereas, Singh and Singh, (2012) found that the maximum removal (94 %) of Cr(VI) using rice husk carbon. Nearly 79.94 % removal of Cr(VI) in a tannery industry wastewater was only achieved using rice husk (Swathi *et al.*, 2014) and this study achieved the removal percentage of 88.3 % in a tannery industry using rice husk silica powder. The maximum removal of Cr(VI) in an aqueous solution achieved with the contact time of 180 min. (Singha *et al.*, 2011) and 100 min. (Anand, *et al.*, 2014). This study removed the maximum of Cr(VI) in a tannery industry wastewater rather than the aqueous solution with the contact time of 150 min.

The maximum removal of Cr(VI) in an aqueous solution and in a tannery industry wastewater depends on the surface area presented in a rice husk, rice husk carbon, rice husk ash and rice husk silica. The availability of surface area depends on the method of heat treatment, chemical treatment and contact time. Della *et al.*, (2002) obtained RHA specific surface area around 54 m²/g at 700 °C for 6 h and after wet grinding for 80 min, the particle's specific area increased to 81 m²/g from 54 m²/g. In this study, the specific area of gray colour RHA (500°C for 4 h) is about 242 m²/g, whereas specific areas of white colour RHS (650°C for 6 h) are about 128 m²/g. This decrease in the specific area is proportional to heating temperature and time; as a result, there is an agglomeration effect and diminishing porosity on rice husk ash (Della *et al.*, 2002). Further, increased specific area from 128 m²/g to 186 m²/g for RHS was obtained, when RHS was milled with milling machine for about 60 min. for this present study. The surface area found in the rice husk ash is about 57.5 m²/g (Bhattacharya *et al.*, 2006) and 105.42 m²/g (Singh and Singh, 2012). Singha *et al.*, (2011), found that the surface area for rice husk is about 0.54 m²/g. The increased surface area is due to increase in porosity, which was obtained through pre-thermal treatment. Pretreatment of rice husks can remove lignin, hemicellulose, and reduce cellulose crystallinity (Kumar *et al.*, 2013).

The variation of removal of Cr(VI) in an aqueous solution and in a tannery industry wastewater of this present study and in previous studies is due to type of aqueous solution, the type of adsorbent, availability of surface area, thermal treatment, chemical treatment, contact time, initial concentration, pH and adsorbent dosage. The important point is to be noted that all

previous studies were removed the Cr(VI) in an aqueous solutions (Srinivasan *et al.*, 1988; Della *et al.*, 2002; Bhattacharya *et al.*, 2006; Singha *et al.*, 2011; Singh and Singh, 2012; Anand *et al.*, 2014; Apeksha and Mote, 2014), whereas Swathi *et al.*, (2014) removed the Cr(VI) from a tannery industry wastewater. This study differs from the Swathi *et al.*, (2014), because this study removed the Cr(VI) in a tannery industry wastewater using rice husk silica powder, but Swathi *et al.*, (2014) used rice husk for the removal of Cr(VI) in a tannery industry wastewater.

CONCLUSION

In the present study, experiments have been conducted for the removal of Cr(VI) in tannery industrial wastewater using rice husk silica powder as an adsorbent. To know the ability of rice husk silica powder for removing Cr(VI) in the tannery industrial wastewater, the experiments were conducted with varying adsorbent dosage, contact time and pH against the initial concentration of 292 mg/L. The results showed that the maximum percentage removal of Cr(VI) in the tannery industrial wastewater at an optimum adsorbent dosage of 15 g, contact time of 150 min., pH of 4 respectively, using rice husk silica powder was 88.3 %. The experimental data on removal of Cr(VI) from tannery industry wastewater is validated with the Cr(VI) in an aqueous solution of same initial concentration of tannery industry waster. Also, the obtained maximum removal percentage of Cr(VI) in a tannery industry wastewater using rice husk silica powder with an optimum process parameters was verified with the other physico-chemical parameters TDS, TS, COD, BOD, and SO₄²⁻ in a tannery industry wastewater. The Langmuir and Freundlich adsorption isotherms were used to check the favourable nature of the adsorption process, but, Langmuir isotherm was found to be in good agreement with experimental data. This study concluded that the experimental investigation done for this study may be reproduced for removing Cr(VI) from tannery wastewater or from any chromium based water and industrial wastewater.

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