Alternative adsorbent to remove Cr (VI) ions from industrial wastewater

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Abstract. Heavy metals which are discharged from industries in the environment with wastewater causes serious problems for the living organisms. To save the water bodies from the heavy metal contamination, many treatment processes are there. But most used is Adsorption processes where Activated Carbon is used as Adsorbent. As the activated carbon is costly, the treatment is expensive. So, low-cost alternate adsorbents are to be identified in the present scenario. The locally available agro waste material namely Teak Leaves (botanical name is Tectona Grandis) is used as low-cost adsorbent Teak leaves (TL) with pretreatment have been used to study for their potential use to remove chromium (VI) from wastewater by Adsorption. Contact time, Adsorbent dose, pH, initial Cr (VI) ions concentration and particle size have been studied for batch process. The optimum pH, agitation time, Cr (VI) ions conc. adsorbent dose and particle size were found 2.0, 75 minutes, 175 milligrams/l, 25 gm/l and 150 – 300 microns respectively at the room temperature (30 ± 2 °C) and achieved 69.1 % to remove Cr (VI). Whereas, the powdered activated carbon (PAC), the removal was 78% at optimum conditions 2.0, 60 min, 125 mg/l, 15 gm/l and <75micron respectively. The adsorption equilibrium data was used to fit into, Freundlich, Langmuir and modified Freundlich isotherm. But Freundlich isotherm fitted well compared to other two. A distinct advantage of TL as bio-sorbent is that these are locally and easily available agricultural waste and can be used with minor chemical treatment. This process is cost effective and eco-friendly.

Keywords. Adsorption, Adsorbent, Chromium (VI), Isotherm, Langmuir, Teak Leaves

1. Introduction

Global water resource management is one of the crucial challenges in the present situation. Heavy metals are entering into human body through contaminated water and also other living organisms through food chain. These heavy metals are considered as potentially carcinogenic to living cells [1] Mining, electroplating, tanning, metallurgical operations, etc are the processes to discharge heavy metals into the environment [2]. Heavy metals like Ni (II), Cr (VI), Cu (II), etc are non-biodegradable [3]. Through bio-accumulating of heavy metals in living organisms, it reaches the toxicological level [4]. Unless water bodies and land are managed properly, there is a risk for human health, agricultural development and the ecosystems [5]. Cr (III) is an essential trace element for plants and animal metabolism. Whereas Cr (VI) is toxic, carcinogenic and mutagenic [6]. According to EPA, Cr (VI) is the one of the causes of cancer in human being when ingested over life span. Due to the toxicological effects of this metal, Cr (VI) ions should be removed from wastewater before it’s disposed.
Ion-exchange, precipitation, reverse osmosis, evaporation, chemical reduction, electro dialysis, etc are used processes for removal of metals from water [7]. Due to the operational and maintenance difficulties and treatment expenditure for removal of toxic metal ions is high, so uses of these methods for large scale treatment of wastewater is expensive.

Various investigators have used different low-cost adsorbents have been used to remove some abiotic absorbents include brick kiln ash, fly ash etc. had been already used[8]. Biotic absorbents namely peanut hull[9], teak leaves [10], neem leaves [11], cashew nut shell [12] etc. were also applied to remove heavy metals from wastewater.

Cellulose, hemicellulose, pectin, lignin, carotene, anthocyanin and tannin are the chemical components of a tree leaves which are the most important sorption sites. All these chemical components contain hydroxyl, carboxylic, carbonyl, amino and nitro groups which play a vital role for metal sorption [2]. The prime objective of the study is the optimization of pH of the solution, agitation time, initial Cr (VI) ions concentration, adsorbent dose and particle size to remove Cr (VI) from wastewater using TL and PAC as adsorbents. The best fit isotherm should be identified among Langmuir, Freundlich and Modified Freundlich adsorption isotherms based on regression coefficient.

2. Literature Review

Ajmal Mohammed et al (2008) had applied teak leaves for the separation of Pb (II) ions from aqueous solution. Maximum adsorption occurred at pH 5.0 [10]. Shivami and Prince (2008) had removal Cr(VI) by rude Pongamia Leaf Powder(CPLP) from wastewater. The adsorbent was most effective at pH 2.0 and contact time 165 minutes [30]. Adsorption of Cr (VI) from aqueous solution by Areca nut shell was conducted by Geetha et.al (2009). It was reported that maximum Cr (VI) removal was 100% at pH 4.0[31]. Mise and Shivarajappa (2010) studied the adsorption of Cr (VI) by the activated carbon of date palm seeds. Maximum Cr (VI) removal was at pH 2.0 [32]. From the above studies, it can be concluded that pretrreated teak leaves has not applied for the removal of Cr (VI) from wastewater. As it is not converted into activated carbon so the operating cost for the wastewater treatment will be low. And it will be ecofriendly for the users.

3. Materials and Methodology

3.1. Materials

3.1.1. Stock Solution preparation for Cr (VI)
Stock Solution was prepared by dissolving 2.828 gms of Potassium dichromate salt in 1000 ml of double distilled water to get 1000 mg/L or 1000 ppm [14]. The stock solution was further diluted in double distilled water to get the desired strength of Cr (VI) solution.

3.1.2. Adsorbent Development
In the present studies, locally available dry biomass namely dried teak leaves (Tectona Grandis) were used. Teak leaves were initially washed thoroughly with distilled water to remove the dirt and other impurities. It was dried in the Sun dried to remove the moisture completely. The dried teak leaves were dipped in 5 % ethanol for 3 – 4 hrs to remove the chlorophyll content. And, also it was washed again to remove the traces of ethanol with distilled water. These leaves were again sun dried at 42°C for two days. The leaves were dipped for overnight into 0.1 N NaOH solution to remove the lignin content and some humic acid from the leaves. These were Sun dried for three days to remove moisture content completely. These dried leaves were crushed to smaller particle sizes (1.14 mm to 75 micron) and stored.
in desiccators. This material hence for this referred as “TL” (Treated Teak Leaves). Powdered activated carbon (PAC) had procured from the local chemical supplier, was also used as alternative adsorbent to compare the results.

3.1.3. Reagents
All used chemicals were analytical reagent grade. pH of the solution was adjusted by using low conc. HCl or NaOH.

3.1.4. Equipment
The major equipment used include
i) Motorized rotary shaker with speed controller
ii) UV Spectrophotometer
iii) Scanning electron microscope (SEM)
iv) Digital pH meter and Digital weighing balance

3.2. Methods

3.2.1. Characterization of TL adsorbent
The prepared TL adsorbent material is in greenish brown colour. The surface characteristics of TL was studied using SEM. It can be seen from Figure 1& 2. These large pores are formed by the elimination of dirt, dust and chlorophyll. The FTIR of the TL showed the probable functional groups present in the adsorbent. The FTIR spectrum of TL (Figure 3) indicates the presence of predominant peaks at 3422.70, 3390.50 cm⁻¹ (–OH and –NH stretching vibrations), 2932.80 cm⁻¹ (- CH stretching vibrations), 1731.50, 1637.20 cm⁻¹ (-NH bending vibration in –NH₂), 1367.80, 1310.60 cm⁻¹(-NH deformation vibration in –NH₂).

3.2.2. Adsorption Studies
Adsorption studies were conducted with 100 ml of synthetic solution containing metal ions in glass bottles, placed on a rotary shaking machine. These glass bottles were agitated at a speed of 150 rpm to a required contact time after adjusting the pH and adding known amount of adsorbent dose. The filtrate was analysed for the estimation of residual chromium (VI) in the solution after adsorption using UV-spectrophotometer at 540 nm as per standard methods [15].

The specific amount of chromium adsorbed was determined by the given equation

\[
q_e = (C_0 - C_e) \times \frac{v}{w} \quad (1)
\]

\[
\% \text{ Removal} = \frac{(C_0 - C_e) \times 100}{C_0} \quad (2)
\]

Where \(q_e\) is the adsorbate amount (mg/g) at the equilibrium condition adsorbed on the surface of adsorbent. Where \(C_0\) and \(C_e\) are the initial and equilibrium concentrations of chromium (mg/l) respectively. Where, \(v\) is the volume (ml) of the aqueous solution and \(w\) is the mass of adsorbent added.

3.2.3. Adsorption isotherms
Adsorption isotherms were studied to model the adsorption process and calculate the adsorption capacity of TL. Langmuir, Freundlich and Modified Freundlich isotherms were applied in the present studies.

**Figure 1.** SEM (Pre adsorption)  **Figure 2.** SEM (Post adsorption)

**Figure 3.** FTIR of Teak Leaves

4. Results and discussion

In the earlier studies, teak leaves with the operating conditions of initial Cr (VI) ions conc. of 50 mg/l and pH 6.0, were efficient to remove 44 % of Cr (VI) ions from the aqueous solution [14]

4.1. Role of pH on metal uptake
The effect of pH on TL and PAC was investigated using two sets of glass flasks orderly arranged one for TL and the other for PAC adsorbent materials. Each glass flask contains 100 ml of synthetic solution. pH of solution in each flask of each set was adjusted to 2, 4, 6, 8 & 10 respectively using either low conc. HCl or NaOH. 0.5 g of TL or PAC was added separately to each flask and agitated for 45 mins. The samples were filtered using filter paper. The residual Cr (VI) in filtrate was estimated using UV spectrophotometer. The percentage of Cr (VI) removal with TL and PAC were estimated using equation 2. The outcomes are shown in Figure 4. It is found that the maximum Cr (VI) removal of 69.1 % and 78 % at pH 2.0 for TL & PAC respectively. This may be due to electro negativity of chromium in comparison with hydrogen. So, H+ ions can easily substitute metal ions from the soln. Due to the presence of more H+ ions at low pH, a greater number of metal ions will be replaced by available H+ ions. Hence, the value of pH is 2.0 to be considered for both TL and PAC for further analysis.

![Figure 4. Role of pH on Cr (VI) ions uptake](image)

4.2. Role of Contact Time on the Removal of Cr (VI) ions

Agitation time or contact time is the crucial parameter to remove adsorbate. It is obvious; more adsorption takes place with high contact time till equilibrium reaches. The role of contact time on TL and PAC was studied with synthetic soln at pH 2.0 containing 50 mg/l of Cr (VI) collected in the glass flasks. A series of such glass flasks collected and added 500 mg of TL or PAC and placed in the rotary shaker at the room temperature. One glass flask each containing TL or PAC was being withdrawn from the rotary shaker after the agitation periods of 2, 5, 10, 15, 20, 30, 45, 60, 75, 90, 120, 150 and 180 mins respectively and filtrated through the filter paper. The filtrate was analysed to estimate the residual Cr (VI). The percentage of Cr (VI) removal using TL and PAC is shown in figure 5.

From the Figure 5, it has been observed that rapid rate of adsorption up to almost 75 min and hence considered the optimum contact time for TL was 75 mins and for PAC 60 mins. The optimum contact time for TL was 75 mins indicates that the adsorption process attained the equilibrium state. In the equilibrium state, the rate of adsorbate transfer into the inter phase of the adsorbent surface and the solution remains constant.
To remove metal ions with concentration variation using TL & PAC was investigated with contact time 75 & 60 mins respectively. 0.5 gms of TL or PAC adsorbent was mixed to each glass flask containing 100 ml stock solution with pH 2.0 and metal conc. 50, 75, 100, 125, 150, 175, 200, 300 and 500 mg/l. After the completion of required period of agitation, the filtrate was analysed using UV spectrometer. The optimum metal ions concentration for TL and PAC are 125 mg/l and 175 ppm respectively. The metal removal percentages against metal ion concentration are shown in figure 6.

Figure 6 depicts that the metal removal efficiency decreases with the rising of initial conc. to a certain point. Thereafter, the metal removal efficiency remains same with the rising initial conc. As the number active sites on the adsorbent surface becomes less so the metal ions conc. Increases in the soln.

The explanation is that the free energy of the active surface of the adsorbent is more for the low initial conc. So the metal removal efficiency from the solution is more at the low initial concentration. But the magnitude of free energy of the activated surface reduces with the initial concentration, as the free energy makes an electrovalent bond with metal ions in the adsorbent and soln inter phase. The initial metal ions concentrations for TL and PAC are found 125 ppm and 175 ppm respectively.

Figure 6. Role of initial metal ions conc. on the removal of Cr (VI)
4.4. Role of Adsorbent Concentration to remove the metal

The role of adsorbent dose to remove metal was investigated with synthetic solution with pH value 2.0 with agitation time 75 mins & 60 mins for TL and Activated carbon respectively. The metal ion concentration for TL and PAC materials was maintained as 125 ppm and 175 ppm respectively. The adsorbent doses of 0.5, 1, 2, 3, 5, 7.5, 10, 15, 20, 25, 30 and 40 g/l for both TL and PAC were selected and added to glass flasks arranged in order. After the required time of agitation, the solutions were filtered and estimated metal ions remaining in filtrate. The metal removal percentages are shown in figure 7. The conclusion from the Figure7, Cr (VI) removal efficiency increases with the amount of adsorbent. The optimal adsorbent doses are 25 gm/l and 15 g/l for TL and PAC respectively. As the metal ions dose in the stock solution is constant, the removal efficiency of metal varies with the adsorbent dose to a certain extent. The more amount of adsorbent indicates much available active sites for removal. As the available Cr (VI) ion in the aqueous solution is constant, so the requirement of active sites of the adsorbent is also fixed. Hence, there is no effect for adsorption by increasing the active sites of the adsorbent. Conclusion is that the requirement for the adsorbent dose is fixed with a given metal ions dose and no effect on increasing the adsorbent dose to separate the metal ions from the solution after attaining equilibrium state.

![Figure 7](image_url)

Figure 7. Role of adsorbent dose to remove metal ions

4.5. Role of Particle Size on the separation of metal

Adsorbent particle size is an important factor in adsorption process. The role of particle size was analysed using TL particle size range between 2.36 mm and 75 microns. The separation of metal ions with different particle size implies that the separation of metal ions increases with the decrease of particle size due to more surface area and also reduces the mass transfer resistance which is shown in figure 8. From figure 8, it can be concluded that the optimal metal ions separation takes place with the particle size 150 – 300 micron for TL and 75 micron for PAC respectively. The uptake percentages of metal ions from the solution are 85.3 % for TL particle and 92.8 % for PAC. Lower mesh particles have more active surface area compared to the bigger mesh the particles with the same mass quantity. The metal ions removal efficiency increases with the lower mesh particle sizes.
4.6. Adsorption Models

Three models namely Langmuir, Freundlich and modified Freundlich isotherms have been applied to study the different adsorption parameters. The Langmuir adsorption model can be applied in the linear form as [16].

\[ \frac{1}{q_e} = \frac{1}{q_{\text{max}} b} \times \frac{1}{C_e} + \frac{1}{q_{\text{max}}} \]  

\( q_{\text{max}} \) is the maximum adsorbed metal per unit weight of adsorbent with equilibrium concentration \( C_e \). While \( b \) is a constant for affinity of the binding sites. \( Q_e \) is an adsorption capacity when the adsorbent’s surface is being deposited with Cr (VI) ions. To predict the adsorption efficiency of the process, the dimensionless constant \( R \) can be calculated by the following formula.

\[ R = \frac{1}{1 + bC_0} \]  

\( C_0 \) is initial concentration of chromium (VI) and \( b \) is Langmuir isotherm constant respectively. If \( r < 1 \), it is favourable isotherm, \( r > 1 \) the unfavourable isotherm, if \( r = 1 \), it is a linear isotherm [17] and when, \( r = 0 \), an irreversible type of isotherm [18].

The Freundlich model (Eq. 5) describes about the heterogeneity and monolayer adsorption.

\[ Q_e = K_f C_e^{1/n} \]  

The Freundlich adsorption model is written in eq. 6 [19].

\[ \log (q_e) = \log K_f + \frac{1}{n} \log (C_e) \]  

\( K_f \) is Freundlich constant and \( 1/n \) is adsorption intensity (heterogeneity factor) respectively. \( K_f \) and \( 1/n \) may be obtained from the intercept and slope of the linear Freundlich plot.

Modified Freundlich isotherm. The following equation is derived based on the assumptions that the exchanger surface is heterogeneous nature and each exchange site adsors individually as per Langmuir isotherm [20].
\[ S_1 = S_{\text{max}} \alpha \left( \frac{a_1}{a_2} \right)^\beta \left[ 1 + \alpha \left( \frac{a_1}{a_2} \right)^\beta \right] \] (7)

\[ \alpha = \frac{A a_2^\beta}{S_{\text{max}}} \]

\[ a_1 = \text{activity for solute 1} \]
\[ a_2 = \text{activity for solute 2} \]
\[ S_{\text{max}} = \text{available maximum exchange sites} \]
\[ S_1 = \text{adsorbed amount of solute 1} \]

By equation (7), the Freundlich isotherm may be written when \((a_1 / a_2)^\beta \ll 1\), trace quantities of solute 1. However, without limiting the sorbing solute to trace quantities, equation (7) is used as modified Freundlich isotherm, can be written as follows,

\[ \log \left[ \frac{S_1}{(S_{\text{max}} - S_1)} \right] = \beta \log C + \log \frac{A}{S_{\text{max}}} \] (8)

Where, activities are used as concentration \((a_1 / a_2 \text{ for } C)\). The constants \(A\) and \(\beta\) are measured by linear regression. The sorption process can be analysed from these constants based on the distribution of sorptive energies of the exchange surface.

**Figure 9.** Langmuir Model with PAC

**Figure 10.** Freundlich Model with PAC
Figure 11. Modified Freundlich Model with PAC

Figure 12. Langmuir Model with TL

Figure 13. Freundlich Model with TL
Figure 14. Modified Freundlich model with TL

Table 1. Linear equations of isotherms for PAC and TL

| Model          | Equations               | PAC          | TL           |
|----------------|-------------------------|--------------|--------------|
| Model          | Linear equation         | $R^2$        | Linear equation | $R^2$        |
| Langmuir       | $Y = 0.029 X + 0.035$   | 0.707        | $Y = 1.108 X + 0.060$ | 0.878        |
| Freundlich     | $Y = 0.294 X + 1.042$   | 0.962        | $Y = 0.671 X + 0.011$ | 0.96         |
| Modified Freundlich | $Y = 0.628 X - 0.578$   | 0.936        | $Y = 1.171 X - 1.866$ | 0.818        |

Table 2. Constants of Freundlich and Modified Freundlich Isotherms

| Adsorbent | Freundlich constants | Modified constants | Freundlich constants | Recommended Isotherm          |
|-----------|----------------------|--------------------|----------------------|-------------------------------|
|           | $K_f$                | $1/n$              | $A$                  | $\beta$ | $K_d$ |
| PAC       | 11.015               | 0.294              | 12.894               | 0.628              | 0.421               | Freundlich and Modified Freundlich |
| TL        | 1.0256               | 0.671              | -                    | -                  | -                  | Freundlich |

Table 3. Optimized conditions to remove the metal ions by PAC and TL.

| Sr. No. | Parameter                          | PAC       | TL       |
|---------|------------------------------------|-----------|----------|
| 1       | pH                                 | 2.0       | 2.0      |
| 2       | Contact time                        | 60 mins.  | 75 mins. |
| 3       | Initial Conc. Of metal ions         | 175 ppm   | 125 ppm  |
| 4       | Adsorbent dose                      | 15 g/l    | 25 g/l   |
| 5       | Particle size of the adsorbent      | 75 micron | 150 – 300 micron |
| 6       | % Removal of Cr (VI) ions at Sl. No. 1 to 5 optimized conditions | 78        | 69.1     |

5. Comparison between different Adsorbents
The performances also have been analysed from the cost perspective. The preparation cost for TL and PAC adsorbents are Rs. 190.00 and Rs. 1400.00 per kg respectively. The optimal TL and PAC doses are 1.0 kg in 40 litres of wastewater with metal ions concentration 125 ppm and 1.0 kg in 66.67 litres of wastewater containing metal ions 175 ppm. The cost for TL and PAC adsorbents are Rs. 4.75 and Rs. 20.90 per litre wastewater treatment respectively. Hence, the cost of wastewater treatment with TL bio-sorbent appears very low.

Table 4. Statement about performance of various Low-Cost Adsorbents.

| Adsorbent                        | Adsorbate | Operating parameters                                                                 | Maximum % removal | Reference |
|---------------------------------|-----------|---------------------------------------------------------------------------------------|-------------------|-----------|
| Eucalyptus bark                 | Cr (VI)   | At value of pH 2.0, Initial metal conc. 250 ppm. Adsorption capacity 45 mg/g of adsorbent, | Maximum removal of Cr (VI) 87.4% | [21]      |
| Micelle-Clay                    | Cr (VI)   | At value of pH 2.0, Initial metal conc. 50 ppm, agitation time 3 h with Temp. 25.0 ± 0.2°C, Adsorbent conc. 5.0 g/l, pH 4.0, | Removal efficiency was 100% at optimal conditions. | [22]      |
| Modified Coconut husk           | Cr (VI)   | Cr (VI) ions conc. 20 mg/L, Contact time 90 mins. Adsorbent dose 0.4 g/l, At the value of pH 2.0, | 96% ± 3.6          | [1]       |
| Maize Bran                      | Cr (VI)   | Initial metal conc. 200 ppm with Temp. 40°C.                                          | Maximum metal removal 312.52 mg/g | [23]      |
| Activated carbon from coconut tree | Cr (VI) | At the value of pH 3.0 and Particle dimension 125-250 µm.                               | Adsorption capacity Was reported 3.46 mg/g | [24]      |
| Teak Leaves                     | Cr (VI)   | At the value of pH, agitation time, Cr (VI) ions conc., adsorbent dose and particle size are 2.0, 75 minutes, 175 milligrams/l, 25 gm./l and 150 – 300 microns | 69.1 %            | Mandal & Rao |

6. Conclusion
Wastewater treatment by adsorption using TL as adsorbent material is a low-cost process and also eco-friendly process. The metal separation depends on the various parameters like pH value of the solution, agitation time, initial metal ion concentration, etc. The observed equilibrium data is the best fitted to the Freundlich isotherm which confirms the monolayer adsorption. Studies reveal that the agricultural waste (TL) can be effectively utilized to remove metal ions from the industrial wastewater. The small-scale industries may adopt this technique to treat the wastewater at a low cost.
7. References

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