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Eco-Friendly Chromium Removal from Tannery Wastewater Using Oak Acorn Waste

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Highlights

- Cr (VI) removal was studied using oak acorn waste as a natural adsorbent.
- Optimal adsorption at pH 7, 10 min contact time, and 550 rpm stirring rate.
- 1.00 g of adsorbent in 100 ml gave best Cr (VI) removal results.
- Oak acorn waste reduced COD, SS, BOD, and Cr (VI) in tannery wastewater.
- Maximum removal rates: COD 1%, SS 17%, BOD 12.5%, Cr (VI) 12%.

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Abstract

Chromium (VI) is a heavy metal which is the cause of water pollution and toxic effect on the living structures. In this study, the adsorption of Cr (VI) and the removal of other pollution parameters were investigated using natural adsorbent as waste acorn of *Quercus ithaburensis* (Oak tree) which was produced by valeks industries. The study consists of two experimental parts. In the first part, principal factors affecting the adsorption were investigated by batch experiment methods. Optimum conditions of stirring rate, pH, particular size of adsorbent, starting concentration, amount of adsorbent, mixing time and temperature for effective chromium removal were determined. The best adsorption conditions were found as 10 min contact time at 7 of pH with 550 rpm stirring rate in 100 ml sample volume using 1.00 g of adsorbent.

In the second part, adsorption tests were exercised with wastewater under pre-determined optimum conditions for the adsorbent. Experimental studies were taken place on the removal of COD (Chemical Oxygen Demand), SS (Suspend Solid), BOD (Biological Oxygen Demand) and chromium (VI) from composite sample of the tannery wastewater. The outputs of adsorption using oak acorn waste for COD, SS, BOD and chromium (VI) respectively about 1%, 17%, 12.5%, 12% was obtained.

1. INTRODUCTION

Toxic metals are harmful to many life forms which are exposed to these metals by air, water and especially food. The form of the metallic contamination cog in water may be either in a solution water or at the bottom of the water collection without solving. Metallic contamination is moved from one place to another place through convection, wind, and water [1-3].

In the leather industry, finished leather is made with raw materials. During these operations, many chemicals, auxiliary materials, and water are used. The amount of water and chemicals used is changed in accordance with the type of leather, technology and tannery's tools, and equipment status [1-4]. Disposing the used water contains many pollutants. In this case, this wastewater contains major pollution parameters: chromium (VI), total chromium, COD, BOD, TSS, Sulphur, and oil and grease [2] (Table 1). To reduce its negative impact, treatment of the wastewater has been attempted. Waste removal methods are usually based on sedimentation of hydroxide form after being reduced from Cr (VI) to Cr (III). For degradation chromium to +3, pH should be brought to the range of 2 and 3. After this stage, with a reducing agent (SO_2, $NaHSO_3$, $Na_2S_2O_3$, Na_2SO_3), Cr (VI), becomes reduced to Cr (III). With the neutralization and OH in the second stage, the sedimentation is provided [3-6]. Therefore, reductive conversion of mobile Cr (VI) to immobile

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Cr (III) represents a principal remediation objective for contaminated soils and waters. This approach leverages the fundamental solubility disparity: Cr (VI) compounds dissolve readily, whereas Cr (III) species exhibit limited solubility, predominantly occurring as soluble complexes or insoluble phases. Among other removal methods, electrochemical precipitation, ion exchange, adsorption, solvent extraction, membrane separation by method, foam separation, reverse osmosis, adsorption, and evaporation of microorganisms can be counted [3].

Table 1. *Characterization of Leather Wastewater*

Parameter	pH	COD (mg/L)	TSS (mg/L)	Cr (VI) (mg/L)	T.Cr (mg/L)
Value	6.95	15016	10080	7.504	7.784

Tannins can be used both in leather tanning and in the removal of chromium ions that occur during tanning. Tannins, non-water-soluble complexes, and phenolic hydroxyl groups found in large quantity in water-soluble forms of phenols by binding to proteins. The most probable source for tannin as in trade is the oak gall. Tannin with different heterogenic groups forms compounds with other molecules under the effect of its grain structure (degree of polymerization, molecular weight) [3] (Figure 1). Tending to form compounds with the protein structure for tannins depends on the protein size and the load [4,5]. Vegetable tannins are used as tanning agents in the leather industry for tanning leather [6,7]. Traditional chrome tanning, which uses chromium salts (a type of mineral tannin), generates wastewater contaminated with highly toxic hexavalent chromium, posing severe risks to aquatic ecosystems and drinking water if not treated rigorously.

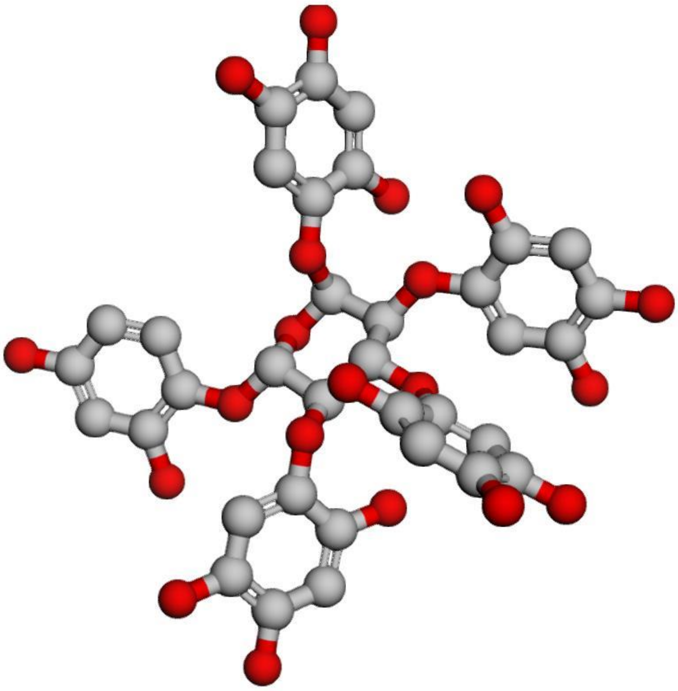

Figure 1. *Structure of a tannin*

Cr (VI) exposure mediates genotoxic effects, impairing DNA integrity through damage to the sugar-phosphate backbone, histones, nucleotide base pairing, and chromosomal structure. Due to its elevated toxicity relative to other metals, the effective removal of Cr (VI) from aqueous environments, including water and wastewater, is a critical environmental priority. While physicochemical methods for Cr (VI) removal exist, they are often cost-prohibitive and generate secondary hazardous sludge. Consequently, the development of economically viable and environmentally sustainable removal technologies is imperative. Biological strategies—encompassing biosorption, bioreduction, and bioaccumulation—represent promising, cost-effective, and ecologically benign alternatives for Cr (VI) remediation [8].

Biological adsorption (biosorption) is one of the alternative technologies in which less adsorption materials are used for removal of heavy metals, such as Cu, Cr, Ni, Pb, Cd, Mn, Fe, Co, Se, from aqueous solutions. Most of the biological materials are to bind heavy metals, but those who are high enough for the metal binding capacity and heavy metals selection find wide application area.

The main reason for choosing adsorption is being abundant and inexpensive in the field of biomaterials [8-11]. There are several research projects completed in this area such as beech tree talai, fly ash, dried active mud, bagasse, sweet almond shells obtained from the activated carbon, natural zeolite and animal bones are recorded as the substances already used in removal of heavy metals [12-16]. As an Eastern Mediterranean plant, oak acorn (Quercus ithaburensis), contains 45% of tannin, shows a wide distribution in Turkiye and ideal for use in adsorption. In Turkiye, Thrace, Western, Central, South and Southeastern parts of Anatolia are perfect places for the growth of the plant. Q. ithaburensis has been used as a dye plant since ancient times [7]. Because of its quite advanced calycate feature, oak acorn (valonia oak) attracts more attention compared to other oaks.

In this study, using shells of oak acorn as factory waste to remove heavy metal ions not only mitigates its environmental pollution but also provides a cost-effective adsorbent alternative to activated carbon. Besides, the second part of the study focused on the simultaneous removal of COD, SS, BOD, and Cr (VI) from composite tannery wastewater samples.

2. MATERIAL AND METHOD

Tannin acorn wastes have been provided by Salihli Bonito and Valeks factory of Artur Chemistry Company. Acorn waste was dried at 50 ˚C for 2 hours for removal of moisture.

Adsorption operations were performed in 250 ml beaker using magnetic stirrer by batch experiment conditions. All experiments were repeated twice, and the results were averaged. The experiments other than determining the effect of temperature on adsorption were conducted at 20°C. In experiments, the stock solution of $K_2Cr_2O_7$ at 1000 mg Cr^{6+} / L was prepared, and the desired concentration of solutions from the stock solution was prepared by dilution, in amounts respectively 500, 250, 100, 50, 20, 10, 5 mg Cr^{6+} / L. Except the experiments on determining the effect of adsorbents amount of adsorption efficiency, official tests were made on 1.0 g of adsorbents and solution of 20 mg Cr^{6+}/ L and 100 ml prepared from $K_2Cr_2O_7$. The reason for using potassium dichromate as chromium source is that it provides a well-characterized, strongly oxidizing source of hexavalent chromium Cr (VI) in the form of the dichromate ion ($Cr_2O_7^{2-}$), making it the benchmark compound for studying Cr (VI) toxicity, environmental behavior, and reduction processes to Cr (III).

After centrifuging the samples taken from the liquid phase at the end of the experiment, by dilution at specific rate chromium concentration in the solution was determined through testing on atomic flame absorption spectrophotometer. For pH adjustment, concentrated HCl and 1.0 M (mol / L) NaOH were used.

The parameters used in the adsorption process and the selected values are given in Table 2. These parameters except for determining the optimum adsorption time experiments were used through 10 min contact time for the adsorbent.

Absorption atomic spectrometry as a technique was used in determination of chromium concentration in a solution [17-19]. For the measurement of chromium (VI), Flame Atomic Absorption Spectrophotometer with the brand of Varian (Varian 220FS Fast Sequential SpectrAA) was used. The solution was centrifuged at the end of the adsorption process, and certain volume of the sample taken for determination of Cr^{6+} were performed in flame AAS. Taking into consideration the amount of Cr^{6+} remaining in solution, yield for adsorption data was calculated and the necessary graphics were drawn.

Table 2. *Parameters used and values chosen in Adsorption*

Parameter	**Chosen Value**
Particle size(µm)	35, 50, 200, 355, 500
Temperature (°C)	12, 20, 30, 45, 55, 70
Stirring time (min)	1, 3, 5, 10, 15, 20, 30
Mixing rate (rpm)	300, 400, 550, 700, 900
pH	1, 3, 5, 7, 9, 11
Solid to liquid ratio (g/100ml)	0.25, 0.50, 1, 2, 5, 10
Cr^{6+} concentration (mg/L)	5, 10, 20, 50, 100, 250, 500

3. RESULTS

Changing the contact time to find out best adsorption values was chosen as first parameter by keeping constant pH at 7, adsorbent amount as 1.0 g, beginning concentration of Cr^{+6} solution as 20 mg/L (ppm), solution volume as 100 ml, stirring rate as 550 rpm, particle size of oak acorn waste as 200 µm, and at room temperature (20 °C). Adsorbed amount values were distributed between 3.671 and 4.287 ppm. The highest value for adsorbed amount of Cr^{6+} ion was 4.287 ppm at 20 min. The lowest value was at 1 min (3.671ppm). 10 min (4.234 ppm) was chosen considering the best performance conditions. The reason for the rapid retention (in this case 10 min) of Cr(VI) on protonated surfaces is the strong and selective interactions between the functional groups (–OH, –COOH, etc.) on the adsorbent surface and the pollutant, allowing the equilibrium to be reached quickly (Figure 2).

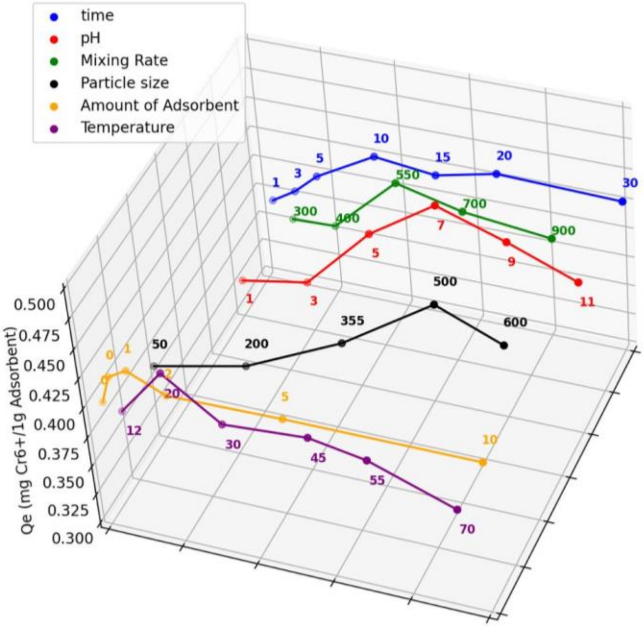

Figure 2. *Q_e change of Cr^{6+} ions versus each parameter optimized in this study*

The following parameter was pH with keeping constants other parameters, and the results are shown in the tables. The experiments were carried out at 10 min of contact time, 1.0 g of adsorbent amount, 20 ppm of beginning concentration of Cr^{+6} solution, 100 ml of solution volume, 550rpm of stirring speed, and room temperature at 20 °C. The highest value for pH was found at 7 as 4.674 ppm. Cr (VI) exists as oxyanions in aqueous solutions either as $HCrO_4^-$ (pH 1–6.5) and or as CrO_4^{2-} (pH > 6.5). Owing to $HCrO_4^-$ remains the dominant type near pH 7. Its monovalent charge facilitates stronger electrostatic attraction to positively charged adsorbents compared to divalent CrO_4^{2-}. For higher values than 7 of pH, the adsorbed amount of Cr^{+6} gets lower values. At pH=9, the value was 4.484 ppm while it was 4.265 ppm at pH=11.

Stirring rate was the next parameter to find out optimum value. The experiments were carried out at pH 7, temperature at 20 °C, adsorbent amount at 1.0 g, beginning concentration of Cr^{+6} solution at 20 ppm, solution volume at 100 mL, and contact time at 10 min. The highest concentration of adsorbed amount was detected at 550 rpm as 5.176 ppm. The second highest value was found at 700 rpm as 5.049 ppm. The values increased from 300 rpm to 550 rpm and then started dropping to 700 rpm and continued to 900 rpm. The particles of adsorbent and Cr ions contact each other at most when the stirring rate reached to 550 rpm. Over and below this speed the duration and meeting chance of particles with each other are lower than 550 rpm.

Four different sizes were chosen for determination of the best particle size for adsorption. The following parameters in the experiments are kept constant for finding the best particle size. The experiments were carried out at pH 7, at 20 °C, adsorbent amount as 1.0 g, beginning concentration of Cr^{+6} solution as 20 ppm, solution volume as 100 mL, contact time as 10 min, and stirring rate as 550 rpm. Particle size under 50 µm of oak acorn waste had the lowest adsorbed amount as 4.021 ppm. It increased with increasing particle size until the particle size of 355-500 µm from 4.021 to 5.038 ppm respectively. Then, it decreased for over 500 µm as 4.824 ppm.

The amount of adsorbent was changed from 0.25 to 10.0 g as 6 values (0.25, 0.50, 1.00, 2.00, 5.00, and 10.0 g) to decide about the best value. The experiments were carried out at pH 7, 20 °C, 20 ppm, volume as 100 mL, and 10 min of contact time. The adsorbed amount increased from 0.25 g (4.4045 ppm) to 1.00 g (4.362 ppm). After 1.00 g, it started decreasing from 4.362 ppm (1.00 g) to 4.223 ppm (2.00 g). Then, it moved nearly stable until 10.0 g (4.246 ppm).

Optimum temperature was chosen using 12, 20, 30, 45, 55, and 70 °C for the best adsorption conditions. By changing temperature from 12 to 20 °C, the adsorbed amount shifted from 4.451 ppm to 4.842 ppm. Then, it started dropping constantly from 4.842 ppm (20 °C) to 4.248 ppm (70 °C).

The experiments of beginning concentration on Cr (VI) were carried out at pH 7, adsorbent amount as 1.0 g, solution volume as 100 mL, contact time as 10 min, stirring speed as 550 rpm, and room temperature at 20°C. The adsorbed amount increased by rising the beginning concentration values of Cr (VI). It starts at 0.953 (at 5 ppm) and ends at 82.048 ppm (at 500 ppm). Adsorption percentages change between 16.41 and 31.34. Until the percentage reaches to 50 ppm of concentration, it showed a drop at 10 ppm (from 19.06 % to 17.53 %) and then a straight increase (from 17.53 % to 31.34 %). Over 50 ppm, the adsorption percentages become dropping from 31.34 % to 16.41 %.

Wastewater sample taken from enter of effluent water equalization pool in leather industry waste treatment plant was used with dolomite and oak acorn waste in adsorption experiments. Adsorption experiment was carried under the conditions of the best results obtained in experimental tests. In the conditions of best chromium retention, leather wastewater has been studied in adsorption experiments and has been analyzed for COD, TSS, BOD, Cr (VI) values.

While finding the best values for COD, TSS, BOD, and Cr (VI), in experiments with oak acorn waste, contact time, pH, adsorbent amount, stirring speed and adsorbent size were 10 min, 9.1g, 550 rpm, and 200 µm, the temperature was kept constant at 20 ˚C.

At the end of the adsorption process, in wastewater COD, TSS, BOD, Cr (VI) were analyzed, removal in leather wastewater is calculated for each adsorbent. The changes at the parameters of COD, TSS, BOD, and Cr (VI) in wastewater were shown in Table 3. The data received in the determination of dolomite and oak acorns for waste pre-adsorption (input) and after adsorption (output) to A is as follows. The beginning pH of tannery wastewater was measured as 6.78. Dolomite and bonito for waste oak were administered at pH 9 and 20°C.

Table 3. *Changes in parameters of leather wastewater*

Parameter		**Oak acorn waste (mg/L)**
COD	Input	3720
	Output	3680
TSS	Input	2088
	Output	1743
BOD	Input	800
	Output	800
Cr (VI)	Input	7.433
	Output	6.542

4. CONCLUSIONS

The results can be summarized as follows:

- With increasing mixing time, the adsorption of Cr^{+6} ions is increased, and after 10 minutes the adsorption is fixed. Optimal mixing time is determined as 10 min.

- With increasing pH adsorption capacity for Cr^{+6} ions showed an increase in the aqueous solution. Waste oak acorn had the maximum adsorption at pH 7. Adsorbent surface becomes more negative with increasing pH and adsorbents can be more easily interact with chromium.

- Temperature dependence of the adsorption reactions is determined by changing temperature in adsorption process. The results show that with the increase in temperature Cr^{+6} adsorption capacity of oak acorns there is not much of a change observed for the waste removal by temperature. Thermally stable functional groups (e.g., quaternary amines) might not alter affinity for Cr (VI) oxyanions ($HCrO_4^-$/CrO_4^{2-}) with temperature. In addition, physical adsorption is typically exothermic and decreases with rising temperature.

- After a slight increase by the amount of adsorbent for waste oak acorns, adsorption capacity stayed constant. As a result, the increase of the amount of adsorbent is not affected on the adsorption of Cr^{+6} ions.

- Adsorption efficiency is increased when the particle size of the oak acorn waste gets smaller, there is no change occurred by particle size.

- Adsorption yield shows an increase with the increasing stirring speed once then a slight decrease in the yield for grain waste. As a result, approximately at 550 rpm of stirring speed, adsorption has occurred at maximum for Cr^{+6}.

- Keeping constant the weight of adsorbents at 1.00 g by increasing Cr^{+6} concentrations in solution for 100 ml sample volume adsorption efficiency has increased. This is because at low concentrations, all the hot spots on the surface of adsorbent have not been occupied by molecules, and by increasing concentration of adsorbent in the adsorbent surface, adsorption point of the blank is maintained active.

Besides adsorption experiments, with acorn waste leather wastewater was tested for removal parameters COD, TSS, BOD, and chromium (VI) as following were obtained. As one of the wastewater pollution parameters, COD adsorption occurred 20% with the use of oak acorn waste. This confirms the material's activity against organic pollutants but suggests its efficiency for this specific purpose is limited. TSS removal in wastewater parameters was found to be 17% for acorn oak waste. Acorn oak waste reduced Total Suspended Solids (TSS) in tannery wastewater by 17%, demonstrating limited but measurable efficacy in filtering particulate pollutants due to its physical adsorption properties. The use of oak acorn waste has caused some change in the BOD.

Acorn oak for waste adsorption efficiency was found to be 12%. As a result, according to laboratory conditions created in artificial chromium pollution, acorn oak waste has a good adsorption capacity for Cr (VI) [19-22]. Acorn oak waste can be used as adsorbents for leather wastewater treatment in terms of metal holding capacity.

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CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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