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Chromium(VI) Separation from Tannery Wastes Utilizing Hydrotalcites

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ABSTRACT

Laboratory scale tests were developed to study the high consumption of water in a tannery in Toluca City, Mexico, needed to decrease the biological oxygen demand (BOD) in the wastewater. Biological oxygen demand is generally reduced by reducing the organic material with sodium hypochlorite. However, Cr(VI) is formed in this last process. Hydrotalcite (HT) and its calcination product were applied to separate the

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Cr(VI) formed. The best material found was calcined hydrotalcite, which removed not only Cr(VI) from the wastewater but also other anions, such as Cl^-, SO_4^{2-}, and HCO_3^-.

Key Words: Hydrotalcites; Thermal treatment; Cr(VI) sorption; Tannery wastewater.

INTRODUCTION

Chromium(III) salts are efficiently used as tanning agents in the leather industry, and it would be very difficult to replace them by other tanning agents for the time being. Tannery wastes usually contain high chromium concentrations. Recovery of the chromium content of this waste liquor is necessary for environmental and economic reasons. As such, tannery waste containing Cr(III) is subject to less stringent regulation by Environmental Protection Agency (EPA) than wastes containing Cr(VI), toxic to plants and animals. Bartlett[1] concluded that landfill disposal of Cr(III) would not present a pollution problem, but that disposal of Cr(VI) could cause serious difficulties because of its mobility, even at low pH, where its sorption by clay minerals is the highest.[2–5] There is concern about the oxidative behavior of trivalent chromium because hexavalent chromium is mobile in soils.

Tanning waste liquor contains a range of ionic material as well as small amounts of organic species, which include fats, proteins, and enzymes.[3] In a tannery in Toluca City, Mexico, a large amount of water is utilized to decrease the biological oxygen demand (BOD) in the waste, which is usually very high because of the organic material present. Usually, BOD was reduced by the addition of sodium hypochlorite (4% weight). However, oxidation of Cr was observed after addition of this compound. Therefore, to avoid Cr(VI) formation, sodium hypochlorite (NaClO) is no longer added to the wastes in the referred tannery. Instead, to reduce BOD present to the maximum permissible value ($150\,\mathrm{mg\,L^{-1}}$),[6] wastewaters are diluted with considerable amounts of water, leading to high costs in water consumption.

However, Cr(VI) can be separated by sorption on solids. Organic resins have been applied to the removal of anionic species from tannery wastewater.[3] Pérez-Candela et al.,[7] studied Cr(VI) removal with activated carbons. They found that sorption depends on the porous texture, nature, and preparation procedure of the carbons, with the best performance being obtained by those prepared by physical activation, a contact time near 200 hr and pH = 2. However, other inorganic material, as hydrotalcite (HT)-like compounds, also referred to in this article as HT, can be used to separate Cr(VI) from the tannery waste. The general formula for HT is

$$[M_{1-x}{}^{2+}M_x{}^{3+}(OH)_2](A^{n-})_{x/n}\cdot mH_2O \qquad (1)$$

where $M^{2+} = Mg^{2+}$, $M^{3+} = Al^{3+}$, $A^{n-} = CO_3{}^{2-}$, and $0.17 < x < 0.33$.[8]

Châtelet et al.,[8] studied the competition between monovalent anions, such as Cl^{1-}, and divalent anions, such as $CrO_4{}^{2-}$, for calcined and uncalcined HTs. They studied $CrO_4{}^{2-}$ sorption from pure solutions and from solutions where $CrO_4{}^{2-}$ was mixed with other anions as Cl^- and $SO_4{}^{2-}$ ions. They found that in the presence of mono and divalent anions, such as Cl^-, $SO_4{}^{2-}$, and $CrO_4{}^{2-}$, calcined HT behaves not only as an anion exchanger but also as an oxide adsorbent. In the case of divalent anions, the adsorbed quantity is larger than the anionic exchange capacity. The affinity of each anion in mixed solutions (Cl^- and $SO_4{}^{2-}$) also depends on their solubility. In this case, the greater affinity of $CrO_4{}^{2-}$ than $SO_4{}^{2-}$ in NaCl electrolyte for calcined HT is governed by its lower solubility. Goswamee and coworkers,[9] studied the effect of HT calcination and other parameters on removing $Cr_2O_7{}^{-2}$ ions from aqueous solutions.

When HT compounds are heated to 773 or 873 K, they undergo dehydroxylation and decarbonation, the layer structure is destroyed, and a magnesium–aluminum oxide is formed.[10] The calcined HT samples, also referred to in this work as CHT, can be rehydrated again by contacting the solid with an aqueous solution containing the desired interlayer anions, thus recovering the original structure.[11] Therefore, HT and calcined HT could be alternative solids to separate Cr(VI) from tannery wastewater. The purpose of the present work was to develop a laboratory procedure using the wastewater from the tannery, firstly by reducing the presence of organic material in the water by the well-known effect of the addition of sodium hypochlorite, and secondly by separating Cr(VI) formed with calcined and uncalcined HT.

EXPERIMENTAL

In the present works, three kinds of tannery waters were studied: the tannery liquor where considerable amounts of $[Cr(OH)SO_4]$ was added during the process, the waste from the equalization tank where all wastes were collected, and finally, the effluent waste from the plant. One milliliter of these samples was diluted to 10 mL with distilled water. Standard Cr(VI) prepared with ammonium dichromate $[(NH_4)_2Cr_2O_7]$ solution was also utilized (Fisher Scientific Reagents, Fair Lawn, New Jersey).

To decrease organic wastes and BOD in tannery wastewater samples, 1 mL of 4% sodium hypochlorite solution was added to 100 mL of water samples. The BOD was detected by the method APHA-AWWA WPCF.[12] Cr(VI) formation in the tannery liquor was studied by the addition of sodium hypochlorite utilizing 100 mL of the samples and 0.5 to 1 mL (4%) sodium hypochlorite solution.

Cr(VI) formed by NaClO addition was partially separated from the solution with HT. The prepared sorbent materials for Cr(VI) from the tannery wastewater were the HT and the calcined HT. Hydrotalcite was prepared as described previously by Sato:[13] 1000 mL of 0.25 mol L^{-1} $AlCl_3$ and 0.75 mol L^{-1} $MgCl_2 \cdot 6H_2O$ aqueous solution were added dropwise to 1000 mL of 0.5 mol L^{-1} Na_2CO_3 and 2.5 mol L^{-1} NaOH under vigorous stirring. Both solutions were previously heated and maintained at 333 K during the stirring. Once produced, HT was separated by filtration and then washed by dialysis with deionized water until chloride free. The HT was dried at room temperature in an open dish for 5 days and then again at 353 K in air for 3 hr, and finally crushed in an agate mortar. The x value in the general formula of the HT sample compound prepared was 0.25. The calcined product CHT was obtained by heating HT at 773 K in air for 18 hr. The HT and CHT were utilized as sorbents of Cr(VI) ions.

Two methods were utilized to determine Cr(VI): (1) neutron activation analyses (NAA) for the Cr(VI) sorption isotherms utilizing standard ammonium dichromate solutions and (2) UV spectroscopy to determine Cr(VI) in the tannery wastewater samples. For NAA, aliquots of 1 mL of the solution containing Cr(VI) were neutron irradiated for 30 min in the pneumatic tube facility of the TRIGA Mark III nuclear reactor with an approximate neutron flux of 10^{12} n cm^{-2} s^{-1}. The photopeak of 0.321 MeV from ^{51}Cr produced by the nuclear reaction $^{50}Cr(n, \gamma)^{51}Cr$ was utilized to determine the chromium radioactivity by using a gamma spectrometer that was set up with a Ge/hyperpure solid state detector and a computerized multichannel analyzer. For UV spectroscopy, a 0.4 mL sample of 1, 5-diphenylcarbazide (DPC) solution (0.1 mL dissolved in 10 mL acetone) was added to 10 mL Cr(VI) sample in test tubes and the solutions were mixed. This was followed by the addition of 0.2 mL of H_2SO_4 (20 g of 95% H_2SO_4 dissolved in 80 mL of water) with further mixing. After 20 min, the magenta color was compared with standard Cr(VI) solutions at 540 nm.[14]

Cr(VI) sorption isotherms on HTs using standard solutions of ammonium dichromate were determined as follows: 20 mL of different concentration ammonium dichromate solutions were mixed with 100 mg of HTs. The mixtures were agitated for 24 hr at 293 K. Each suspension was then centrifuged and the Cr(VI) in the residual supernatant solutions was measured by NAA.

Cr(VI) sorption for increasing contact times between the solids and solutions was determined in batch experiments in the tannery waste water samples as follows: 20 mL of the following solutions: (1) tannery liquor, (2) equalization tank water sample, or (3) effluent water from the plant, were mixed with 100 mg of calcined HT and uncalcined HT. The mixtures were shaken during 0.5, 1, 2, 3, 10, 24, 35, 48, and 72 hr at room temperature. Each suspension was then centrifuged and the Cr(VI) in the residual supernatant solutions was measured by UV spectroscopy. Cr(VI) sorbed in the solid was calculated by the difference between the Cr(VI) present in the standard solution and the residual supernatant solutions.

Solid samples were characterized by thermal analysis with a TGA51 TA Instruments thermogravimetric analyzer, x-ray diffraction with a Siemens D-500 diffractometer, and IR spectroscopy with a Nicolet Fourier transform infrared spectrometer model 550. pH values were measured in an Orion Research Microprocessor Ionaly Zer/901 potentiometer with a combined glass electrode.

RESULTS AND DISCUSSION

To confirm whether or not BOD was reduced in the tannery liquor, sodium hypochlorite solution (1 mL, 4% NaClO) was added to 100 mL of the water samples. It was found, as expected, that BOD was reduced from 554 to 250 mg L^{-1}. It is clear then that the water consumption needed to dilute BOD in wastewater can be considerably reduced when sodium hypochlorite is utilized. The effect of NaClO on chromium oxidation to Cr(VI) in the tannery liquor was also studied. It was found that although no Cr(VI) was observed in the untreated water sample, as soon as sodium hypochlorite was added, Cr(VI) was formed in considerable amounts, 12.9 and 26.7 mg L^{-1} for 0.5 and 1 mL of the NaClO solution added. The Cr(VI) formed was separated with HTs, described as follows.

The effect of pH on Cr(VI) sorption on HT and CHT was studied utilizing the Cr(VI) standard solution (Fig. 1) with a concentration of 139 mg L^{-1} of Cr(VI), which was adjusted to pH values from 3 to 11. Cr(VI) sorbed by HT, also refereed to in this article as Cr-HT, was high when the initial pH value of the Cr(VI) solution was between 3 and 10 and very low when the pH value was 11, because of the competition between anions of Cr(VI) and OH^- in the solution. This effect is similar to that obtained by Serrano et al.[15] when they studied the sorption of molybdate ions in calcined HTs. At low pH, the competition does not exist and, therefore, Cr(VI) retention on HT or CHT is higher.

Isotherms for Cr(VI) sorption in a standard aqueous solution ($pH = 4.1$) on (a) HT, and (b) CHT, respectively, were studied by Goswamee et al.[9]

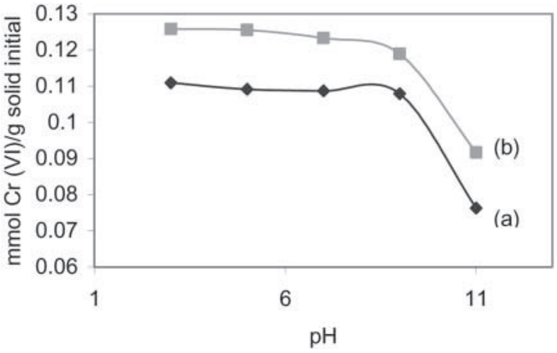

Figure 1. pH effect in Cr(VI) sorption on (a) HT, and (b) CHT.

They found that adsorption of $Cr_2O_7^{2-}$ through rehydration of CHT is considerably higher than the sorption through anion exchange of HT. The former reaches a plateau at a $Cr_2O_7^{2-}$ concentration of 30.19 meq L^{-1}, while the latter shows a slowly increasing adsorption with increase in concentration of $Cr_2O_7^{2-}$. However, in this work, Cr(VI) concentrations in the tannery wastewater is, in general, very low. The initial concentrations of total chromium in the water samples were the following: 1.39 mmol L^{-1} for the tannery liquor, 1.58×10^{-2} mmol L^{-1} for the equalization tank, and 1.23×10^{-2} mmol L^{-1} for the effluent waste of the plant. Sorption of Cr(VI) from solutions of very low concentrations on HT and CHT are similar (Fig. 2). On the other hand, for higher concentrations, sorption on CHT is much higher than in HT, as expected.

Figures 3–5 show the sorption behavior on HT and CHT of Cr(VI) present in tannery liquor, the equalization tank, and the effluent wastewater from the plant, respectively. Samples (100 mL) were treated with NaClO solution (1 mL, 4%), in (a) HT and (b) CHT, for increasing contact time. In all cases, Cr(VI) sorption for short contact periods (1–3 hr) is fast and then it increases slowly. The maximum sorption of Cr(VI) from tannery liquor was 0.0013 mmol in HT and 0.024 mmol in CHT; for the equalization tank, was 0.00051 mmol in HT and 0.00137 mmol in CHT; and finally, for the effluent waste from the plant, was 0.00032 mmol in HT and 0.00063 mmol in CHT. In all three cases, Cr(VI) sorption on CHT was higher than in HT samples, showing that CrO_4^{2-} is unable to replace CO_3^{2-} in HT. As CHT rehydrates, it takes up CrO_4^{2-} as the structure reforms. In all cases for higher Cr(VI) concentration in the wastewater samples, the higher was Cr(VI) sorption.

The results shown in Figs. 3–5 illustrate that when HT is utilized, the retention of Cr(VI) ions is probably performed by a slow anion exchange

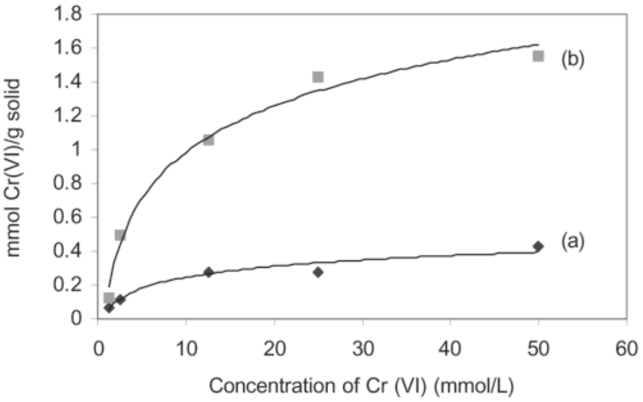

Figure 2. Cr(VI) sorption isotherm on (a) HT; and (b) CHT.

mechanism through the tightly held layer with carbonate ions in the HT interlayer positions[16] as follows:

$$[Mg_3Al(OH)_8]_2CO_3 \cdot 4H_2O + 2Cr_2O_7^{2-} \longrightarrow 2[Mg_3Al(OH)_8]CrO_4 \cdot 4H_2O + CO_3^{2-} \quad (2)$$

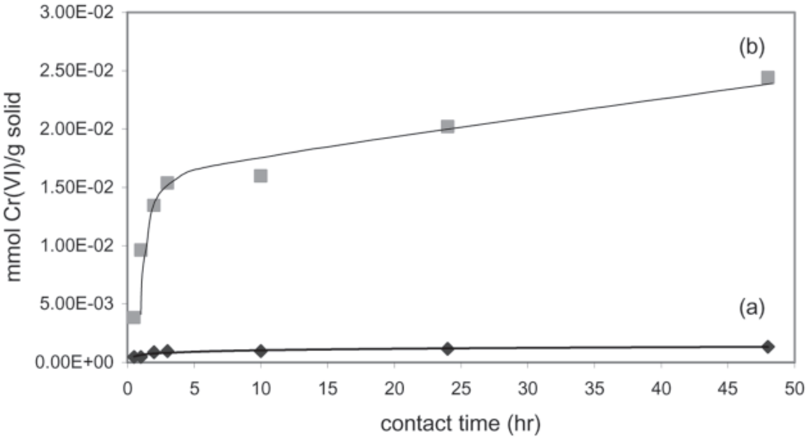

Figure 3. Cr(VI) sorption curve from the tannery liquor with (a) HT; and (b) CHT.

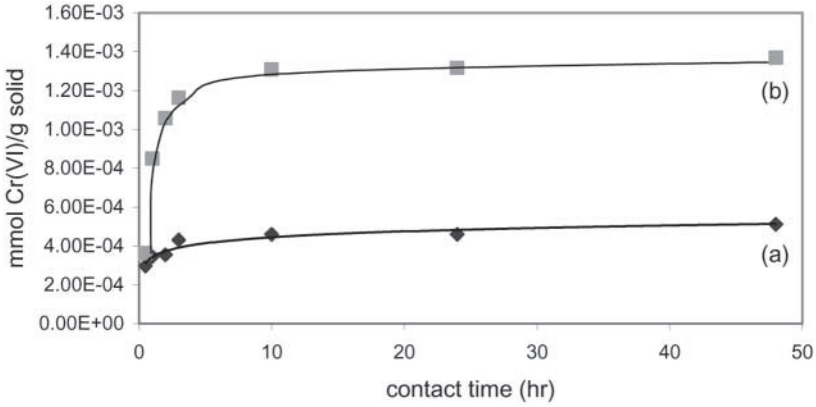

Figure 4. Cr(VI) sorption curve from the equalization tank with (a) HT; and (b) CHT.

On the other hand when CHT is utilized, the reaction was performed by the reconstruction of the HT mechanism as follows:

$$2Mg_3AlO_4(OH) + 8H_2O + Cr_2O_7{}^{2-} \longrightarrow [Mg_6Al_2(OH)_{16}](CrO_4) + 2OH^- \qquad (3)$$

Table 1 shows the variation of the pH values of the tannery wastewater samples after contact with HT. In the case of HT sample, pH increased due probably to a partial dissolution of HT.[17] In CHT, pH increase was still

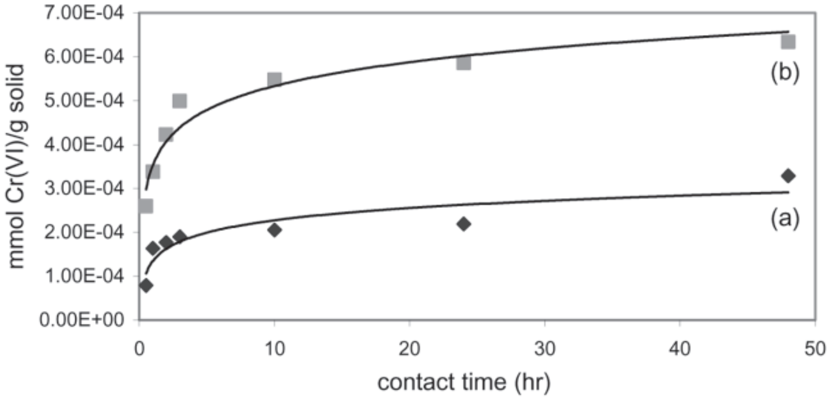

Figure 5. Cr(VI) sorption curve from the effluent from the plant with (a) HT; and (b) CHT.

Table 1. pH behavior in tannery wastewater samples before and after sorption on the solid.

	pH		
Water sample	Tannery liquor	Equalization tank	Effluent from the plant
Original sample	3.51	7.87	9.23
Sample diluted 1 to 10	3.78	7.96	8.67
After contact with HT	8.47	9.17	10.31
After contact with HTC	10.43	10.79	11.06

higher, probably because of the consumption of protons in the reconstruction of the layered HT structure,[18] as shown in Eq. (3).

The big difference between Cr(VI) sorption on HT and CHT observed in Figs. 3–5, is firstly due to the lack of ability to displace CO_3^{2-} in HT, as mentioned before, and also to the competition between other anions present in the wastewater and Cr(VI).

Chemical analyses of anions in tannery wastewater before and after Cr(VI) sorption on HTs are shown on Table 2. The presence of many other anions can be observed, which are also strongly retained on the solids, as their concentrations decrease considerably in the wastewater samples. The retained anions are mainly SO_4^{2-}, Cl^-, and HCO_3^-. On the other hand, the amount of OH^- in the solution increases because the final pH value increases considerably during reconstruction of HT.

Table 2. Chemical analyses of anions present in the tannery liquor before and after contact with CHT.

Anion	Initial anion concentration (mg/L)	Anions concentration after contact with CHT (mg/L)
Cl^-	360	190
SO_4^{2-}	142	0
OH^-	0	106
HCO_3^-	22	0
$Cr_2O_7^{2-}$	408	74

Infrared Spectroscopy

The infrared spectra (IR) of Cr-HT was found to have most of the HT and CHT vibration bands reported in the literature[18–21] (Fig. 6). However, two additional vibration bands were also found at 1126 and 867 cm^{-1}. Figure 6(a) shows the IR spectra of HT sample, the broad band at 3467 cm^{-1} resulting from an overlapping of hydrogen vibrations: stretching vibrations of structural OH^-, physically adsorbed water, and vibrations of OH^- bonded with carbonate ions in Mg and Al environments. The shoulder at 3125 cm^{-1} can be assigned to solvation water molecules highly condensed into the microporosity.[21] The low intensity band at 1636 cm^{-1} is assigned to bending vibrations of strongly adsorbed water (solvation water of compensating anions). In relation to carbonate ions, their three bands observed in a symmetric environment (ν_3: 1415 cm^{-1}, ν_2: 880 cm^{-1}, and ν_4: 680 cm^{-1}) shift to lower frequencies: ν_3: 1390 cm^{-1}, ν_2: 750 cm^{-1}, and ν_4: 639 cm^{-1}. Figure 6(b) shows the infrared spectra of calcined HT, with considerable decrease of the carbonate ions bands. The infrared spectrum of the Cr-HT see [Fig. 6(c)] shows the broad band found at 3449 cm^{-1}, corresponding to the overlap of hydrogen vibrations, but the shoulder at 3050 cm^{-1} was no longer observed, meaning that the solvation water molecules condensed in the microporosity were probably eliminated during the calcination of HT samples. The ν_3 and ν_4 frequencies of carbonate ions were observed at 1401 and

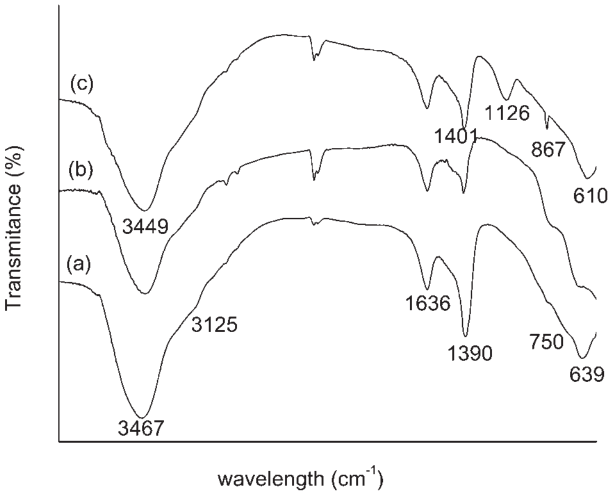

Figure 6. The IR spectra of (a) HT; (b) CHT; and (c) Cr-HT.

610 cm^{-1} but the ν_2 vibration was not clearly observed. The two new vibration bands were found, as mentioned, at 1126 cm^{-1}, assigned to sulphate ion, and a weak band at 867 cm^{-1}, assigned to CrO_4^{2-}, according to Nakamoto.[22] In the present work, a standard $K_2Cr_2O_7$ solution was utilized to prepare Cr-HT, however, IR analyses showed that Cr(VI) was retained as CrO_4^{2-} in HT and CHT. The information obtained from the infrared spectra of HT and Cr-HT was used to determine the relative concentration loss of CO_3^{2-} ions in Cr-HT, since the intensity of the band of frequency ν_3 corresponding to interlayer carbonate anions decreased,[8] thus revealing some exchange of interlayer anions. The CO_3^{2-} ions relative percent loss was found to be 36.5%.

Thermal Analysis

Figure 7 shows the TGA analyses of reconstructed Cr-HT with a tannery liquor. The curve presents two clear weight losses. The first one at 438 K, which was due mainly to loss of interlamellar water, and the second one at 715 K, due to dehydroxylation and loss of CO_3^{2-} from the residual CO_3^{2-} ions present in Cr-HT sample.[23] The third weight loss at 1191 K probably is due to chromate fusion and partial decomposition.

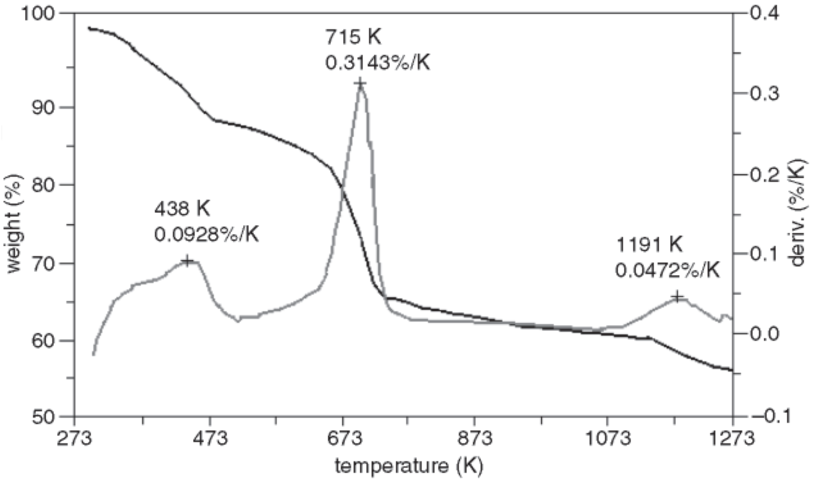

Figure 7. The TGA analyses of Cr-HT.

X-Ray Diffraction

The XRD patterns of the synthetic HT samples are shown in Fig. 8. The uncalcined HT sample showed an HT-type pattern (JCPDS card-22-700). Sample calcined at 773 K, CHT, showed the diffractograms, which are characteristic of an Al and Mg mixed oxide (JCPDS card-4-829). CHT samples, in contact with Cr(VI) present in a tannery liquor, reproduced the lamellar HT structure, as shown by the XRD patterns see [Fig. 8(c)]. The *d*(006) interplanar distances are compared in Table 3. The *d*(006) value increased in the HT samples treated with the tannery wastewater probably due

Table 3. Interplanar distances *d*(006) for calcined HT left in contact with Cr(VI) standard solution and the tannery wastewater samples.

CHT after contact with	Concentration of Cr(VI) in sample (mg/L)	*d*(006) Å 1 hr contact	*d*(006) Å 24 hr contact
Standard solution	1,970	7.705	8.251
Tannery liquor	49.6	7.801	7.801
Equalization tank	0.65	7.744	7.801
Effluent of plant	0.13	7.772	7.772

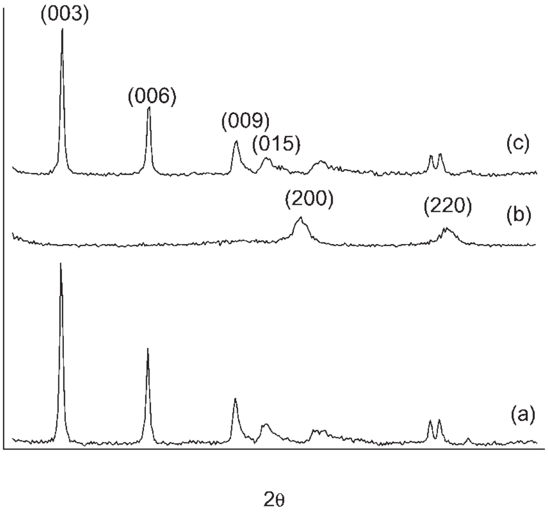

Figure 8. The XRD patterns of (a) HT; (b) CHT; and (c) Cr-HT.

to the several anions SO_4^{2-}, Cl^-, and HCO_3^- retained in CHT during the reconstruction of the lamellar HT.

CONCLUSION

It was found that water consumption in the studied tannery can be minimized decreasing the BOD by the addition of sodium hypochlorite solution. Cr(VI) formed by the addition of the sodium hypochlorite, can be reduced from 0.92×10^{-3} to 0.22×10^{-3} mmol by HT through an ion exchange mechanism, and from 0.92×10^{-3} to 0.15×10^{-3} mmol by calcined HT through the HT reconstruction mechanism.

Sorption of Cr(VI) on calcined HT was higher than on HT and it depended strongly on the pH and the concentrations of the Cr(VI) in the wastewater samples.

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