



Removal of Cr³⁺ From Tannery Wastewater Using Unmodified And Acid-Modified Arabica Coffee Husk Adsorbent

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Abstract— Tannery effluent containing chromium is a major problem in leather industry. In Kenya, tannery effluent is discharged directly into the main domestic sewage system, making wastewater treatment plants more difficult to operate. Chromium has long been employed in tanning because of the outstanding characteristics it provides to the leather as well as its ease of use. Only 60% of the entire chromium salt reacts with the hides, though. In other words, around 40% of the chromium remains in the solid and liquid wastes. Subsequently, the removal and reuse of the chromium content of these wastewaters is vital for environmental protection and economic reasons. This study explored the potential for removal and recovery of chromium from tannery effluent using unmodified (UCH) and modified (MCH) coffee husk biomass adsorbents. The raw coffee husk was subjected to sulphuric acid treatment, followed by characterization using FTIR and SEM analysis. The effects of initial metal ion concentration, agitation time, dosage, and pH were investigated in batch experiments. Effluent was obtained from Dogbones tannery in Dandora, Nairobi and was subjected to adsorption process at optimum conditions. Batch adsorption tests on these coffee husks revealed that as the initial metal ion concentration increased, the adsorption of metal ions increased as well. At pH = 4.5, the highest metal uptake was recorded. Maximum percentage removal was 47.52 % and 69.3 % for the UCH and MCH, respectively. For the UCH and MCH, the adsorption equilibrium was attained after 25 minutes and 15 minutes, respectively. Optimum dose of 3 g was realized for the two adsorbents. The presence of hydroxyl, carboxylic, and carbonyl functional groups was detected using FTIR. The surface texture and morphology of the biosorbent were revealed by scanning electron microscopy. The findings imply that coffee husk, in both modified and unmodified forms, is a low-cost, ecologically acceptable biosorbent that can be used to remove chromium ions from tannery effluent and other industrial effluents.

Keywords— Arabica coffee husk, Biosorption, chromium (III), adsorption efficiency, Tannery

I. INTRODUCTION

Water pollution by heavy metals is among the major ecological problems in the world and possess a great danger to the health of human beings, plants and animals [1, 2]. The greatest causes of water pollution, are discharge of raw effluents from industries, sewers, agricultural field chemicals and runoff waters [3]. Leather tanneries convert raw hides into finished leather. In the process they use chromium salts. Effluents contain large levels of chromium ions, which need to be removed before the disposal of the effluent. Other industrial activities such as textile dyeing, petroleum refining, painting, printing, glass and ceramic production, mining operations use chromium. Chromium (III) is taken up directly in the water by food chain and accumulates in the aquatic animals [4]. Drinking water polluted by Cr³⁺ ions for a long term, even if in trace levels, contributes to development of several health diseases, such as malignancy, queasiness, loss of consciousness, renal failure and elusive effects on metabolism and intelligence [5]. To remove Cr³⁺ from

wastewater, numerous researchers have looked into chemical precipitation [6], coagulation, flotation, adsorption, ion exchange, reverse osmosis [7], and electro-dialysis [5]. The high cost of adsorbents (e.g. activated carbon) is one of the most prominent drawbacks of adsorption techniques [8, 9]. Precipitation and ion exchange produce large amounts of solid waste, but osmosis and electro dialysis are hampered by membrane instability [10, 11]. The utilization of biomass as a low-cost natural adsorbent for the heavy metals removal from polluted water is a key area of attention within the field. Many studies have been reported using various biomasses such as; orange peel [12], *Sargassum sp* biomass [13], rice husk, sesame husk, avocado seed [14] the sunflower husk and tea waste. Additionally, Angel *et al* studied the corn residual biomass to eliminate Cr (VI) from aqueous solution under optimal adsorption [15]. Raw and treated biomass of maize stocks were considered for Cr³⁺ removal from aqueous solution for water treatment. As a result, this research focused on modifying coffee husk with H₂SO₄, characterization of the coffee husk biomass with SEM and

FTIR, and testing the applicability and effectiveness of coffee husk adsorbent as a waste management method for chromium removal from aqueous solutions by examining the withholding profile of chromium ions at various adsorption parameters.

II. MATERIALS AND METHODS

Materials

Analytical grade chemicals and reagents were used in this study (Purity > 98.9%); chromium chloride hexahydrate ($\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$), hydrochloric acid (HCl), sodium hydroxide (NaOH), sodium acetate, sulphuric acid (H_2SO_4) were commercially purchased from Sigma Aldrich (Kobian, Nairobi). Coffee husk biomass were collected from Dedan Kimathi University of Technology Coffee farm. Tannery wastewater was collected from Dogbone tannery, Dandora, Nairobi.

Adsorbate

Chromium stock solution (1000 mg/L) was made by dissolving 5.124 g of $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ in 500 mL distilled water then topped to a liter. The sodium acetate buffer was used to maintain the pH of the stock solution. Working solutions were obtained through dilution of stock solution with distilled water. For each batch experiment, fresh dilutions were prepared. The pH of working Cr^{3+} solutions was controlled with HCl and NaOH solutions (0.1 M).

Adsorbent

Distilled water was used to thoroughly wash the coffee husk biomass. The samples were then sieved through fine-mesh sieve, washed and then oven-dried at 105°C until there was no further change in weight. The samples were divided into two portions, one portion was subjected to acid modification while another one was labelled "unmodified coffee husk". Chemical modification of the coffee husk was performed as described by [16] using sulphuric acid with slight modification. For six hours, 5 g of UCH was shaken at 120 rpm with 100 mL of 0.1 M H_2SO_4 . Filtration of the mixture was done and the residue dried at room temperature and stored in an airtight bottle labelled "modified coffee husk" (MCH).

Instrumentation

The structure and appearance of the surface of the biomass was investigated using a SEM (JEOL JSM-6510 LA). The functional groups contained in UCH and MCH were determined by FTIR (IRT Tracer- 100, SHIMADZU, Japan).

A mechanical shaker was used in the experiments. The solution pH was carefully monitored using a pH meter (HANNA model). Using AAS, (AA-6200, SHIMADZU), the concentration of chromium ions in the filtrate solution was determined.

SEM Analysis

The surface morphology of the coffee biomass (unmodified, acid-modified and modified Cr-loaded form) was determined using the Scanning Electron Microscope.

The sputter coater (SCD 0050 – Baltec, Liechtenstein) was used to cover the dry-powdered samples with a thin layer of carbon and observed using JEOL JSM-6510 LA scanning electron microscope (10 kV) under vacuum of 1.33×10^{-6} mBar.

FTIR Analysis

The FTIR spectra were acquired at a $4500\text{-}500\text{ cm}^{-1}$ mid-range at room temperature with a resolution of 10 scans per single spectrum after being changed to % transmission mode.

Adsorption experiments

Batch adsorption experiments were done at room temperature in a 250 mL glass conical flasks at 120 rpm on a mechanical shaker. Various influencing adsorption parameters (pH, initial chromium ions concentration, and biomass dose and contact time) were varied.

Effect of pH

The effect of pH on the Cr^{3+} ions uptake by the UCH and MCH was studied by putting 1 g of each of the adsorbent in a 250 mL capacity conical flask containing 100 mL of 50 mgL^{-1} of Cr^{3+} ions adjusted to pH range (2-7) by 0.1M of HCl or NaOH. The mixture was then agitated for 85 minutes, filtered, and AAS was used to determine the concentration of Cr^{3+} ions in the filtrate.

Effect of contact time

The effect of contact time on Cr^{3+} ion adsorption was probed by mixing 100 mL of 50 mgL^{-1} of Cr^{3+} ions at pH 4.5 with 1 g of each UCH and MCH adsorbent. The mixtures were agitated to equilibrium at different time intervals (0 – 85 minutes). After filtering the solid materials with Whatman filter paper No. 42, the concentration of Cr^{3+} in the filtrate was determined.

Effect of adsorbent dose

At varied masses of biomass ranging from 0.5 g to 4.5 g and agitation speeds of 120 rpm, the effect of the dose of the adsorbent on the UCH and MCH was investigated. The optimum conditions of pH = 4.5 and contact time = 25 minutes (UCH); 15 minutes (MCH) were used. The mixture were equilibrated, filtered and amount of residual Cr^{3+} ions analyzed.

Effect of initial Cr^{3+} ions concentration

The effect of initial Cr^{3+} ions concentration was carried out using 100 ml of various concentration of metal ions ($10 - 100\text{ mgL}^{-1}$) mixed with optimal dose of 3 g of each of UCH and MCH biomass at pH 4.5. The mixtures were then agitated for 25 minutes (UCH) and 15 minutes (MCH). Filtration was carried out, and the concentration of Cr^{3+} was determined.

Tannery wastewater

The samples of tannery wastewater were collected from Dogbones tannery in Dandora, Nairobi for three consecutive days. The water samples were collected along the chrome liquor drainage channel to the retention tank.

To prevent contamination of the sample with contaminants from other areas, the sample collection bottles were first rinsed in laboratory with distilled water and then rinsed again with wastewater to be analyzed soon before collection of the samples. The samples were subsequently delivered in a cooling box at 4°C to the laboratory within 2 hrs for chromium analysis. The samples were filtered using Whatman No. 42 filter paper to remove suspended matters.

III. RESULTS AND DISCUSSIONS

The adsorbents were characterized using SEM for surface morphology and texture and FTIR was used to check the functional groups responsible for adsorption. The impact of dose of adsorbent, contact time, pH of chromium solution and initial metal ion concentration were investigated. The results were expressed as the adsorbent's removal efficiency (% Removal).

Surface characterization of coffee husk

The SEM images reveal the surface texture and morphology of the coffee husk adsorbent as shown in fig 1. Fig 1 (a) shows that prior to H₂SO₄ treatment, the coffee husk has a rigid, coarse, and restrained surface.

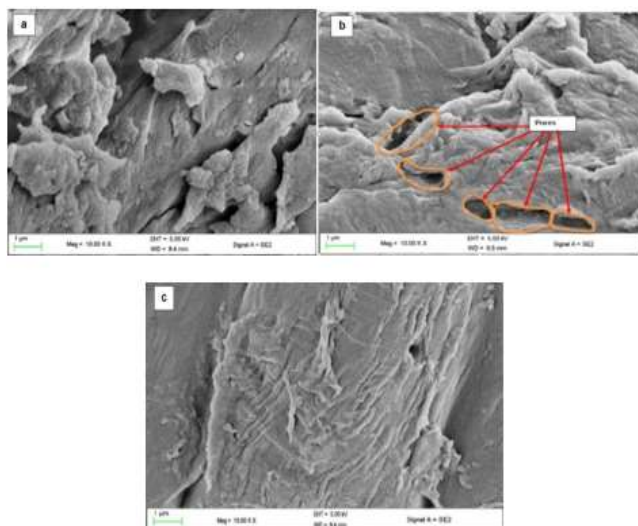


Fig 1: SEM images for (a) UCH (b) MCH (c) MCH loaded with Cr³⁺ ions

However, after modification with H₂SO₄, as seen in fig 1 (b), well-defined pores occurred on the coffee husk surface. This implies that the biomass undergoes morphological variations in its structure, this could be as a result of the partial degradation of hemicellulose, cellulose, and lignin caused by sulfuric acid treatment [14]. These changes in the morphology of acid-modified coffee husk contributed to an improvement in its adsorption properties. Fig 1 (c) shows that after adsorption of Cr³⁺ ions on the coffee husk biomass surface; the caves, pores and surfaces of the adsorbent were covered by the metal ions. The modified Cr³⁺ ions-loaded coffee husk has more rigid and restrained surface as compared to H₂SO₄ treated, this can be attributed to Cr³⁺ ions occupying the pores that were seen in acid treated biomass.

Similar results were reported by [13] on *Sargassum sp* biomass and sea weed, acid-treated avocado seed and orange peel [12].

FTIR Analysis

FTIR spectra of unmodified and modified coffee husk adsorbent is presented by fig 2.

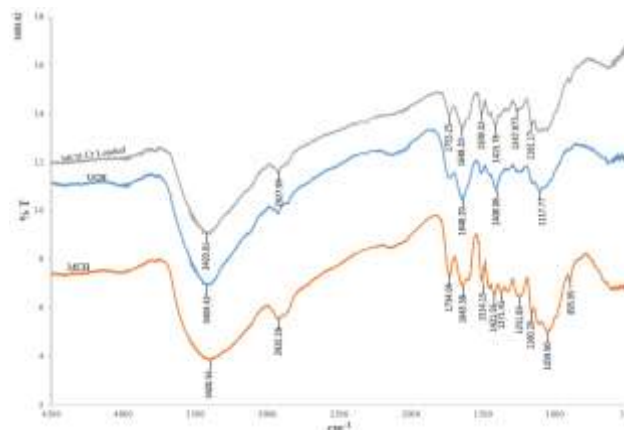


Fig 2: FTIR spectrum UMC, MCH and MCH-Cr Loaded

The FTIR spectra of unmodified (UCH), modified (MCH) and modified coffee husk chrome-loaded (MCH-Cr loaded) are shown in figure 2. The characteristics adsorption bands assigned to hydroxyl, carbonyl, phenolic, and amine are shown by the spectra. Broad bands at region 3404.42 cm⁻¹, 3400.56 cm⁻¹ and 3420.81 cm⁻¹ in the unmodified, modified and modified chrome loaded coffee husk are attributed to the stretching vibrations of O-H because of the intra and inter molecular hydrogen bonding of polymeric complexes like carboxylic acids, alcohols, and phenols [19]. The peaks at 2900 cm⁻¹, 2920.28 cm⁻¹ and 2927.99 cm⁻¹ are assigned to symmetric and asymmetric vibrations of C-H [20]. The peaks at 1734.04 cm⁻¹, 1755.25 cm⁻¹ are assigned to -C=O, C=C groups and the ones observed at 1408.06 cm⁻¹, 1521.56 cm⁻¹ and 1415.78 cm⁻¹ are assigned to -COO⁻¹ groups [17,21]. The peaks in between 1200-1000cm⁻¹ are as a result of stretching vibrations of carboxylic acids and alcohol assigned to C-O. The comparison of the FTIR spectra of the three biomass shows the shifting of O-H group stretching vibration from 3404.42 cm⁻¹ to 3400.56 cm⁻¹ to 3420.81 cm⁻¹ in the UCH, MCH and MCH-Cr loaded, respectively. The appearance of the new peaks at 1734.04 cm⁻¹, 1514.15 cm⁻¹, 1371.41 cm⁻¹, 1251.82 cm⁻¹, 1059.90 cm⁻¹, 895.95 cm⁻¹ could be attributed to the chemical modification [22]. The shifting of the peaks reveals the chemical interactions between the hydroxyl group and the metal ions on the surface of the biomass [14].

Optimization experiments

Effect of pH

The pH remains significant factor in metal ion removal, since it has an effect on the solution chemistry and the adsorbent's surface characteristics. Fig 3 depicts the pH influence on ion removal percentage of chromium. The adsorption of Cr³⁺ on both the UCH and MCH is

significantly affected by increasing the solution pH from 2 to 7. From pH 2 to pH 4.5, the percentage of metal ion elimination increased, then began to decline. Both the UCH and the MCH had an optimum pH of 4.5, which was employed in the remaining studies. The adsorbent's surface is strongly associated with protonation of the functional groups at low pH levels, resulting in a reduction in the percentage removal of chromium ions. Chromium hydroxide begins to form when the pH exceeds 4.5. Precipitation has an impact on the process of adsorption since it stops the metal ions from moving around, making them inaccessible for adsorption. These findings are also consistent with previous studies on adsorption that have reported a range of 4-5 in pH as their optimum pH for Cr^{3+} removal by a variety of adsorbents [23].

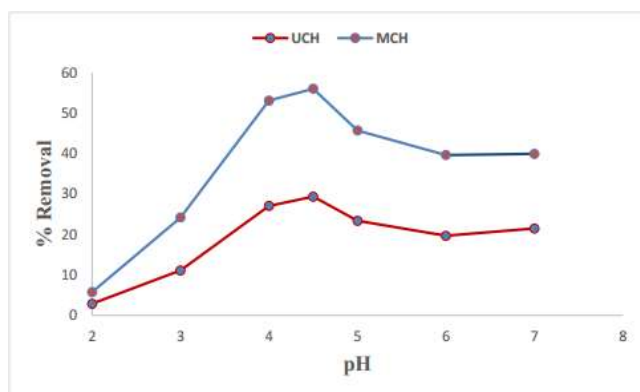


Fig 3: Effect of pH on adsorption of Cr^{3+} on UCH and MCH

Effect of contact time

In batch adsorption investigation, adsorption rate is crucial. The effects of contact time were investigated by measuring the metal ions' uptake in the model solution over an 85-minute period at room temperature. For the UCH and MCH, the maximum % metal uptake was obtained after 15 and 25 minutes, respectively (Fig 4). The capacity of coffee husk biomass being an excellent adsorbent for faster removal of chromium metal ions from polluted waterways is demonstrated by the short contact time.

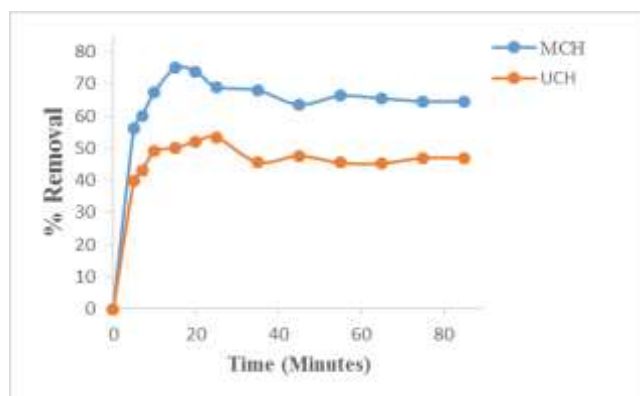


Fig 4: Effect of contact time on adsorption of Cr^{3+} ions onto UCH and MCH

Effect of adsorbent mass

By altering the mass of the adsorbent from 0.5 to 4.5 g, the effect of adsorbent mass on metal ion removal was

investigated and equilibrating with a 100 mL modelled solution of 50 mgL^{-1} . The percentage of metal removed versus dosage is shown in fig 5. The adsorption efficiency of the biomass improves rapidly as the biomass dose rises from 0.5 to 3 g, then plateaus at 65.81% and 75.7% for UCH and MCH, respectively.

The findings can be explained by the fact that gradually increasing the biomass mass increases the number of metal ion adsorption sites. A considerable increase in adsorption does not occur when the mass of the adsorbent is increased further, a phenomenon that could be explained by adsorption site overlapping as a result of biomass overcrowding [24, 25]. Based on the findings, a minimum adsorbent dose of 3 g per 100 mL of adsorbate solution was used in all subsequent studies for UCH and MCH.

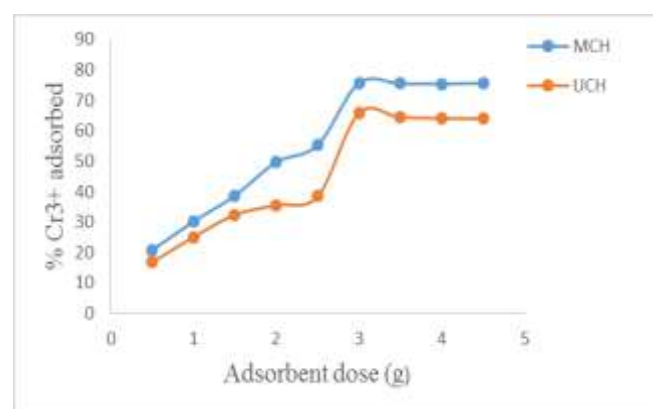


Fig 5: Effect of mass of the biomass on adsorption of Cr^{3+} onto UCH and MCH

Effect of initial metal ion concentration

The effect initial ion concentration was investigated by increasing the concentration from 10 mgL^{-1} to 100 mgL^{-1} while maintaining other parameters at their optimum conditions ($\text{pH} = 4.5$ g, 25 mins for UCH, 15 mins for MCH, mass = 3 g) As demonstrated in fig 6, the percentage of Cr^{3+} uptake increased when the metal concentration was increased from 10 mgL^{-1} to 100 mgL^{-1} , from 20.6% and 31.29% to 55.81% and 75.7%, respectively for UCH and MCH. The graph remains constant above 50 mgL^{-1} concentration. Because a fixed amount of adsorbent has a constant number of binding sites, this could be due to the oversaturation of adsorption sites [26, 27].

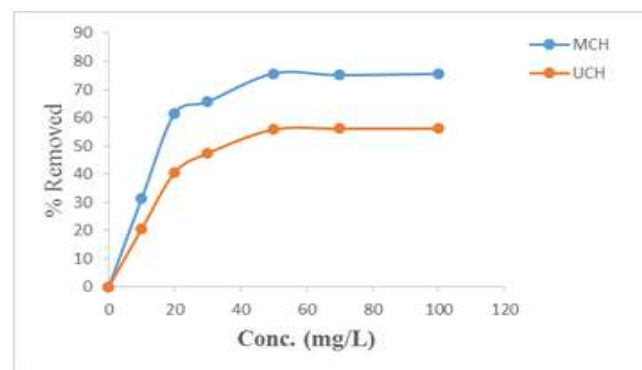


Fig 6: Effect of initial Cr^{3+} ions on adsorption by UCH and MCH

Removal of chromium from tannery effluent

The samples of tannery effluents described were subjected to adsorption by both unmodified coffee husk and modified coffee husk at optimized conditions, pH 4.5, dosage= 3 g and contact time 25 and 15 minutes for the unmodified and modified coffee husk, respectively and removal efficiency was found to be 47.52% and 69.3% for the two samples. Tannery effluent had a lower efficiency than standard chromium solution 55.81% and 75.7% for the unmodified and modified coffee husk, respectively. This could be because other metals in the tannery effluent compete with Cr for adsorption on the coffee husk biomass [28, 29].

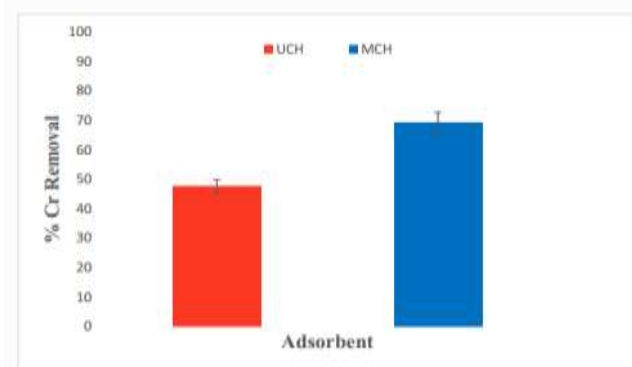


Fig 7: Removal of Cr³⁺ from tannery effluent

IV. CONCLUSION

In this study, unmodified coffee husk (UCH) biomass was chemically modified with sulphuric acid. The FTIR spectra showed more intense peaks that indicated an increase in the number of functional groups in the modified coffee husk (MCH) for Cr³⁺ ions adsorption. The adsorption process was greatly influenced by pH, initial metal ion concentration, and biomass dosage and contact time. Removal of Cr³⁺ from tannery wastewater at optimized parameters showed an efficiency of 47.52% and 69.3% for the UCH and MCH, respectively.

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