Biosorption of Chromium (VI) Ion from Tannery Waste Water Using Two Novel Agricultural By-Products

S. Abdulsalam, J. Mohammed, Deji E. Olubiyo, and Ibrahim I. Tenimu

Abstract—**This study was carried out to investigate the use of two novel agricultural by-products namely: oil-free *Moringa oleifera* cake and sweet potatoes peels for the removal of Cr (VI) ion from a typical tannery wastewater. Biosorbents were produced from these two agricultural by-products, surfaces of these biosorbents were modified using 1N NaOH and 6N HCl, subsequently the modified and unmodified biosorbents were used for the removal of Cr (VI) ion from tannery wastewater using the batch process technique. Results obtained showed that the acid modified biosorbents gave the best results with removal efficiencies of 59 and 74% for oil-free *Moringa oleifera* cake and sweet potatoes peels respectively at pH of 7.0. Kinetics investigation revealed that both agricultural by-products followed the pseudo-first order kinetics. From the results obtained, oil-free *Moringa oleifera* cake and sweet potatoes peels were effective biosorbent for the removal of Cr (VI) ion from tannery waste water.**

Index Terms—**Tannery wastewater, chromium (VI) ions, batch process, surface modification.**

I. Introduction

In recent times, heavy metal pollution has become one of the most serious environmental problems. The presence of heavy metals in the environment is of major concern because of their toxicity to many life forms. Various industries produce and discharge wastes containing different heavy metals into the environment, such noxious environment is as a result of intense mining, smelting, metal finishing, leather tanning, dyeing, radioactive material processing, pesticide and fertilizer application, etc. [1].

Tannery effluent containing chromium is one of the most obvious problems in leather industry. Although chromium is toxic to human and the environment, its processing speed, low costs, light color of leather and greater stability of the resulting leather are the obvious reasons for its preference over other tanning compounds for the processing of hides [2], [3]. Researches and experience had shown that 30-40% of the amount of chromium used in tanning are unreacted. This amount remains in the solid or liquid wastes (spent tanning solution or wastewater), which is usually discharged untreated into running water or the like.

Generally, chromium exists in natural water in two main oxidation states: hexavalent chromium [Cr (VI)] and trivalent chromium [Cr (III)]. Cr (VI) is more hazardous, carcinogenic and mutagenic to living organisms, and as a result of this, couple with its threat to the environment, it is included in the priority list of hazardous substances. The tolerance limits for chromium (VI) are 0.05 and 0.1 mg/L in drinking and inland surface waters respectively [4].

Many methods have been employed for the removal of Cr (VI) ion from aqueous solution and/or wastewater with high degrees of effectiveness. These methods include: reduction followed by electrochemical precipitation, chemical precipitation, chemical oxidation-reduction, ultra-filtration, ion exchange, reverse osmosis, solvent extraction, electro-dialysis, electrochemical, coagulation, evaporation and adsorption [5]. Most of these methods suffer from the following setbacks; high capital and operational costs and disposal of residual metal sludge. In addition, high cost of activated carbon sometimes limits its applicability for heavy metal removal. In view of the aforementioned setbacks, interests of researches have increased in the use of alternative materials, which have low cost, readily available and extremely effective adsorbents [6]. Biosorption technique combines the aforementioned merits in addition to its environmental friendliness.

Biosorption (a biological process) tends to provide solution to the aforementioned problems of the conventional treatment techniques [7]. Biosorption is a technically efficient, commercially viable and eco-friendly technologies and it can be defined as the ability of biological materials to accumulate heavy metals from wastewater through metabolically mediated or physico-chemical pathways of uptake [8]. It employs the use of various divergent biomass, such as agro wastes including husk, bagasse, sawdust, seed cake, etc. Biological cell walls contain large quantity of polysaccharides and proteins offered many functional groups, such as carboxyl, hydroxyl, sulphate, phosphate and amino groups, which can bind metal ions. The performance of a biosorbent depends on its surface characteristics, which can be changed by modification process. The use of raw and modified biosorbents for metal ions removal has gained importance in recent years (Greene and Darnall, 1990; Crist *et al*., 1992).

From the above, it is evident that bio-waste materials and/or agricultural by-products are efficient biosorbents for oil, dye and heavy metal removals. However, types and availability of bio-waste materials varies from location to location. In view of this, there arouse the need to carry out research into the use of some locally available agricultural by-products in our environment as potential biosorbents for the removal of heavy metals from wastewater. This study investigated uses of two novel agricultural by-products namely; sweet potatoes peel and oil-free *Moringa oleifera* cake for the removal of chromium (VI) ion from tannery

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wastewater.

The *Moringa oleifera* seed cakes are the secondary by-product generated after oil extraction from oleaginous seeds, which are poorly utilized, *Moringa oleifera* seed cake is one of those seeds poorly utilized. Recently, *Moringa* oil is being utilized as bio-diesel; however, the residual *Moringa* seed cake imposes a disposal problem. Although, this cake has been found suitable for water treatment [9] and used as animal feeds. However, it use as biosorbent could result in diversification of its areas of application and more so, a supplement to conventional means of removal of heavy metals from wastewater.

Traditionally, sweet potatoes are peeled before eating just like yams and other similar food items. Researchers have shown that sweet potatoes skin is not harmful to human and thus can be eating. Despite of the outcomes of these researches and worldwide sensitization on this, millions of people still prefer eating peeled sweet potatoes and thus the peel or skin constitutes nuisance to the environment. While sensitization is still going on, there arouse the need to effectively manage or utilize this so called bio-waste "sweet potatoes skin or peel".

II. Materials and Methods

A. Materials

Tannery wastewater was obtained from a typical leather and tanning industry in Kano State-Nigeria and stored in a deep freezer in the Chemical Engineering Programme's Laboratory, Abubakar Tafawa Balewa University, Bauchi-Nigeria until the time of this study. Mature seeds of *Moringa oleifera* were obtained at Ganjuwa local Government Area of Bauchi State, Nigeria and the 'sweet potatoes peels' were collected from a dump site close to Yelwa Market, Bauchi State, Nigeria.

B. Methods

1) Characterization of tannery effluent

The physicochemical characteristics of the tannery wastewater were carried out using standard methods. Parameters determined include; pH (digital pH meter), Conductivity, Chemical Oxygen Demand, COD, Biochemical Oxygen Demand, BOD, Total Dissolved Solid, TDS, Color. The above mentioned parameters were carried out by a method well detailed in Abdulsalam *et al*. [9]. In addition, the chromium ion concentrations were determined using the DR/2000 UV Spectrophotometer.

2) Preparation and pretreatment of oil-free Moringa oleifera and sweet potatoes powder

Moringa oleifera seeds obtained were sun dried for three days, then shelled and subsequently reduced to powdery form using mortar and pestle, and sieved through 212 µm mesh. Oil content in the powder was removed using solvent extraction technique [10]. The classified oil-free *Moringa oleifera* powder obtained was washed thoroughly with distilled water in order to remove dirt and other soluble impurities. It was then sun dried for seven days and thereafter washed again with distilled water until a clear solution was obtained and thereafter dried in an oven at 50^{o}C for 24 h.

The processed oil-free *Moringa oleifera* powder was pre-treated separately with 6N HCl (acid treatment) and 1N NaOH (alkaline treatment) for 1h [11] and the pretreated samples were filtered, washed thoroughly with distilled water until a clear mixtures were obtained (i.e. turbidity free). The water layer in each case was decanted and the pretreated oil-free *Moringa oleifera* powder was dried in an oven at 50^{o}C for 24 h and weighed to constant weight. Just as in the case of oil-free *Moringa oleifera cake*, the 'sweet potatoes peels' were also pretreated by employing same procedure except that the oil extracting part was skipped because there was no need for it.

3) Biosorption experiment

This study was carried out in two stages; the first was to determine the optimum dosages required to remove Cr (VI) ion from tannery wastewater using unmodified (UMOD), acid modified (AMOD) and base modified (BMOD) oil-free *Moringa oleifera* and the second was to determine variations of metal uptakes with contact times at the pre-determined optimum biosorbent dosages for the three biosorbent conditions considered in this study (i.e. UMOD, AMOD and BMOD).

Firstly, 100 ml each of tannery wastewater were measured into six 250 ml Erlenmeyer flask and UMOD oil-free *Moringa oleifera* powder were stirred into each flask in the increment of 0.5 g, starting from 0.5 g and ending at 3.0 g. The suspension in all sorption assays were stirred using the JJ-4 six couplet digital electric mixers at a speed of 150 rpm for 1h and then filtered through Whatmann filter paper to remove suspended adsorbent. Concentrations of Cr (VI) ion present in the supernatant were determined using DR/2000 UV spectrophotometer. The above procedure was repeated for AMOD and BMOD biosorbents.

Secondly, at the pre-determined optimum dosages for various biosorbent conditions considered above, effects of contact times on Cr (VI) ion removals were carried out using the same volume of wastewater sample as above (i.e. 100 ml). Residual heavy metal concentrations were determined at 10 min interval until a plateau was reached.

III. Results and Discussion

A. Physicochemical Properties of Tannery Wastewater

Table I showed the physicochemical parameters of tannery wastewater used in this study. From this table, it could be seen that all the measured parameters were above the world health organization's standard (WHO) and therefore required intervention for human and environmental safety.

The purpose of limiting the measurement of heavy metal in the tannery wastewater to that of Cr (VI) ion was because it is one of the most harmful heavy metals to human and the environment at large. More so, it was the metal of interest in this study. Therefore, emphasis was laid on the removal Cr (VI) ion and not in the treatment of the tannery waste water as a whole. From Table I, it could be seen that the concentrations of this heavy metals was above the threshold limit and therefore, the need to reduce its concentration to acceptable limits before discharging into the environment.

TABLE I: PHYSICO-CHEMICAL PARAMETERS OF THE TANNERY WASTEWATER

Parameter (WHO)	Value	Portable water
Temperature, °C	28.70	40
pH	11.10	6-9
Conductivity, µs/cm	391.00	N/A
COD, mg/L	1044.00	160
BOD, mg/L	420.00	50
Colour, pt-Co	1408.00	-
Turbidity, NTU	376.00	-
Cr (VI) ion, mg/L	0.75	0.1

Data presented are average values of triplicate determinations; COD-Chemical oxygen demand; BOD-Biological oxygen demand

B. Effect of Biosorbent Dosage on the Uptake of Cr (VI) Ion Oil-Free Moringa Oleifera Powder

Fig. 1 showed the variation in biosorbent dosage with percentage uptake of Cr (VI) ion for the three biosorbent conditions considered in this study (i.e. UMOD, BMOD and AMOD).

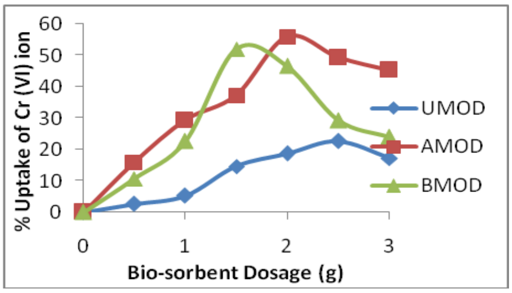

Fig. 1. Effect of biosorbent dosage on cr (VI) ion removal for oil-free *moringa oleifera* powder sweet potatoes peels.

From this figure, it could be seen that there were increased in percentage uptakes of Cr (VI) ion with increasing biosorbent dosages; 0-2.5 g (22.67% uptake of Cr (VI) ion), 0-1.5 g (52% uptake of Cr (VI) ion) and 0-2.0 g (56% uptake of Cr (VI) ion) for UMOD, BMOD and AMOD respectively. Then, the uptake efficiencies started declining with increasing biosorbent dosages. Therefore, the optimum biosorbent dosages for Cr (VI) ion removals were 2.5, 1.5 and 2.0 g for UMOD, BMOD and AMOD respectively.

Fig. 2 showed the variation in biosorbent dosage with percentage uptake of Cr (VI) ion for the three biosorbent conditions considered in this study (i.e. UMOD, AMOD and BMOD). From this figure, it could be seen that there were increased in percentage uptakes of Cr (VI) ion with increasing biosorbent dosages (i.e. between 0-1.5 g, 0-2.5 g and 0-2.5 g for UMOD, AMOD and BMOD respectively). The optimum Cr (VI) ion uptakes were 37.33, 72.00 and 69.33%. Then the percentage uptakes of Cr (VI) ion decreased with increasing biosorbent dosage.

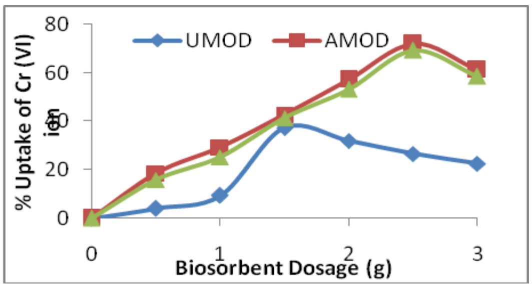

Fig. 2. Effect of bio-sorbent dosage on cr (VI) ion removal for sweet potatoes powder.

From the above, the optimum dosages of the two biosorbent materials used at the three biosorbent conditions are presented in Table II.

TABLE II: OPTIMUM DOSAGES OF BIOSORBENTS FOR CR (VI) ION UPTAKE

	Optimum dosage (g)		Maximum uptake (%)	
Biosorbent	OFMOC	SPP	OFMOC	SPP
UMOD	2.5	1.5	23	37
AMOD	1.5	2.5	52	72
BMOD	2.0	2.5	56	69

OFMOC: oil-free *Moringa oleifera* cake; SPP: sweet potatoes peels

C. Rate of Cr (VI) Ion Removal

The variation of the rate of removal of Cr (VI) ion from tannery wastewater using oil-free *Moringa oleifera* cake as biosorbent is presented in Fig. 3. From this figure, it could be seen that the removal of Cr (VI) ion decreased with increasing contact time, which is typical of any degradation process [12] and then got to constant values between the contact times of 70 and 80 min indicating the end of removal process. The removal efficiencies for Cr (VI) ion were obtained as 33.33, 54.67 and 58.67% for UMOD, BMOD and AMOD respectively and their final residual Cr (VI) ion concentrations were 0.50, 0.34 and 0.31 mg/L. These results showed that surface modifications of the biosorbent material (oil-free *Moringa oleifera* cake) had great impact on the adsorptive capacities of Cr (VI) ion as indicated by 1.64 and 1.76 times effectiveness of the BMOD and AMOD respectively over UMOD.

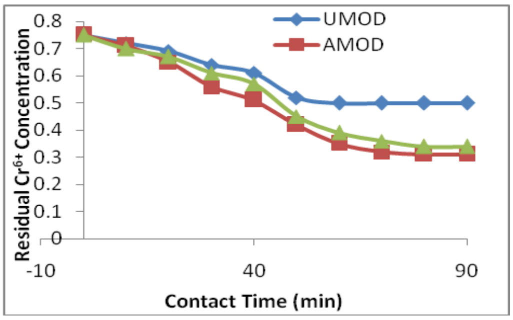

Fig. 3. Rate of removal of cr (VI) ion for oil-free *moringa oleifera* powder at optimum biosorbent dosage.

Fig. 4 showed the percentage uptake of Cr (VI) ion with contact time at the pre-determined optimum biosorbent dosages using sweet potatoes peels as biosorbent. From this figure, it could be seen that percentage uptakes of Cr (VI) ion for the three biosorbent conditions considered in this study increased with increasing contact time. The percentage uptakes of Cr (VI) ion increased from 0 and attained maximum values of 36, 73.33 and 74.67% at contact times of 70, 60 and 80 min for UMOD, AMOD and BMOD respectively. From this observation, it showed that percentage uptakes of Cr (VI) ion by AMOD and BMOD were 2.04 and 2.07 folds more effective than that of UMOD. Therefore, surface modifications (acidic and/or alkaline) of the sweet potatoes peel powder had great impact on the adsorptive capacity of Cr (VI) ion (i.e. it enhanced surface metal-binding capacity of the biosorbent). This result is in line with the literature that surface modification of biosorbent enhances the absorptive capacity of metal-binding [11].

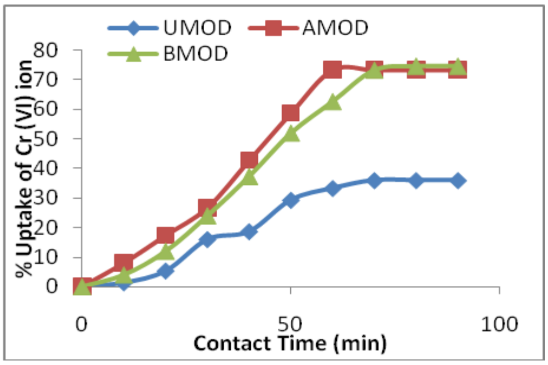

Fig. 4. Variation of percentage uptake of Cr (VI) ion with contact time at optimum biosorbent dosage for sweet potatoeses peels.

From the results obtained, there were significant reductions in the concentrations of Cr (VI) ion by the various conditions of biosorbent considered. However, the residual metal concentrations were above the limits permissible by the regulatory authority [4]. Comparing results obtained in this study with those of others conducted using synthetic wastewater in a batch system (Table III), showed that the two biosorbents considered in this study compete favorably with most results obtained from previous studies. In addition, if the pH of the tannery wastewater used was controlled, an improvement in percentage uptake of Cr (VI) ion could result. Therefore, oil-free *Moringa oleifera* cake and sweet potatoes peels are effective biosorbents for the removal of Cr (VI) ion from tannery wastewater.

TABLE III: AGRICULTURAL BY-PRODUCTS ABSORBENTS USED IN CR (VI) ION UPTAKE

Biosorbent	Cr (VI) ion removal (%)	pH
Hazlenut shell*	99.4 98.9 97.8	1.0 2.0 3.0
Wool*	69.3 5.8	2.0 5.0
Olive cake*	47.1 8.4	2.0 5.0
Sawdust*	53.5 13.8	2.0 5.0
Almond shell*	90.2 55.2	2.0 5.0
Coal*	19.8 8.2	2.0 5.0
Walnut*	85.3	3.5
OFMOC**	**59.0**	**7.0**
SPP**	**74.0**	**7.0**

*Manfe *et al*. [6]; **This Study

D. Kinetic Studies

Of the three biosorbent conditions considered above (i.e. UMOD, AMOD and BMOD) and from the results obtained, the acid modified (AMOD) sample can be chosen as representative sample based on the percentage uptakes of Cr (VI) ion. Therefore, kinetic studies in this research work were based on data collected for the AMOD for both the sweet potatoes powder and oil-free *Moringa oleifera* cake.

Fig. 5 and Fig. 6 present the pseudo-first order and pseudo-second order plots for the adsorption of Cr (VI) ion for OFMOC and SPP respectively. From these plots, it could be seen that the data obtained fitted best the pseudo-first order kinetics judging from the regression coefficients obtained (Table IV). In addition, the kinetic parameters for the two biosorbents for pseudo-first and pseudo-second order kinetics were presented in Table IV.

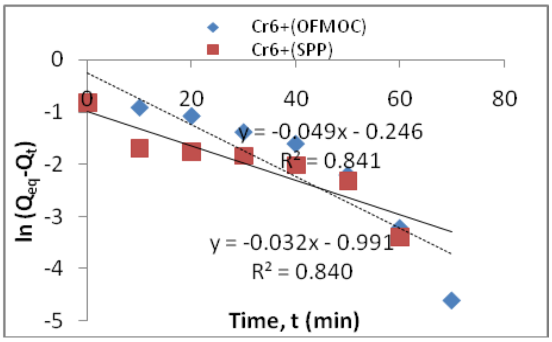

Fig. 5. Pseudo-first order plot for the adsorption of Cr (VI) ion for oil-free *Moringa oleifera* cake and sweet potatoes powder.

From this observation, it could be inferred that the mechanism of adsorption of Cr (VI) ions for the two agricultural by-products considered was pseudo-first order. Although most biosorbent materials follow pseudo-second other kinetics [13]-[15], few literatures have reported some biosorbent materials following the pseudo-first order [16], [17]. The two biosorbents considered in this study followed the pseudo-first order kinetics.

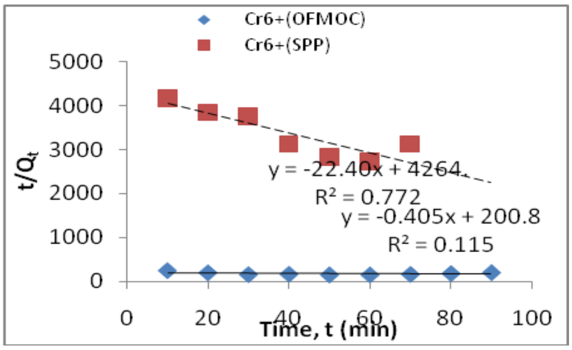

Fig. 6. Pseudo-second order plot for the adsorption of Cr (VI) ion for oil-free *Moringa oleifera* cake and sweet potatoes powder.

TABLE IV: KINETIC PARAMETERS FOR PSEUDO-FIRST AND PSEUDO-SECOND ORDER FOR THE ADSORPTION OF CR (VI) ION USING OIL-FREE *MORINGA OLEIFERA* CAKE SWEET POTATOES PEEL POWDER

Kinetic Parameters				
Biosorbent	Pseudo-first order		Pseudo-second order	
	k_1 (min^{-1})	R^2	k_1 (g/mg. min)	R^2
OFMOC	0.049 0	0.8410	0.000 82	0.1150
SPP	0.032 0	0.8400	0.117 70	0.772 2

IV. CONCLUSION

The potentials of two novel agricultural by-products (oil-free *moringa oleifera* cake and sweet potatoes peel powder) were sought in this study for the removal of Cr (VI) ion from tannery wastewater. These two agricultural by-products were confirmed to be effective biosorbents for the removal of Cr (VI) ion with removal efficiencies of up to 59% and 74% for OFMOC and SPP respectively. It was discovered that surface modification of the biosorbent with HCl and NaOH enhanced significantly the efficiencies of Cr (VI) ion uptake. The mechanism for adsorption followed the pseudo-first order kinetic model.

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