

Continuous Sorption of Chromium Ions from Simulated Effluents using Citric Acid Modified Sweet Potato Peels

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ABSTRACT: Adsorptive removal of chromium ion in aqueous medium using activated sweet potato peel (SPP) was studied in a laboratory-scale fixed bed column. Specifically, the effect of process parameters such as bed depth, flowrate and chromium ion concentration in aqueous solution, on the adsorption efficiency of the acid modified sweet potato peel was examined. Column adsorption analysis showed that at the flow rate of 0.5 cm³/min, bed height of 6 cm and column influent concentration of 30 mg/dm³, the optimum chromium (VI) ion removal of 87.5% was attained with the equilibrium adsorption capacity of 2.4548 mg/g. Continuous adsorption models such as Yoon-Nelson, Adam-Bohart and the Bed-Depth Service Time (BDST) model, were used to analyse the experimental data and based on correlation coefficient, BDST model best aligned with the obtained experimental data with correlation coefficient, R², value of 96.43%. The bed capacity, N_o, and the rate constant, K_a, were calculated as 4.259 mg/dm³ and 0.01045 L/mgmin respectively at optimum column conditions. Results confirmed that acid modified SPP can be used to remove or reduce concentrations of Cr (VI) ions to allowable limits before disposal into water bodies.

KEYWORDS: Continuous sorption, chromium ions, sweet potato peel, kinetic isotherms, breakthrough curves

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I. INTRODUCTION

In recent times, heavy metal pollution mostly due to industrialisation has become one of the most serious environmental problems in developing countries such as Nigeria. The discharge wastewater resulting from industrial processes has indicated high levels of heavy metals which, even in trace quantities, are toxic, have high potential threats to air, water and soil as well as bioaccumulation throughout the food chain and finally affect human beings (Alok et al., 2015). The recommended threshold limit value of Cr (VI) ions before it is discharged into receiving water bodies is 0.1 mg/dm³ (Blanes et al., 2016). Cr (VI) ions enters the environment as a result of various industrial activities such as tannery, metal fishing, inorganic chemicals production, steel manufacturing and electroplating which make use of chromium or its compounds in its production. It is rapidly absorbed into the bloodstream and have adverse effects to major organs, causing ill health such as hypersensitive reactions, rashes on skin, irritations, bleeding through nostrils, weakened immune system, alteration of genetic material, kidney and liver impairment and most likely results to death

of the individual (Podder et al. 2016; Ahmad and Haydar, 2016).

Several methods have been employed to remove ions of heavy metal from the effluent of industries before it is discharged into the environment. Farooq et al., (2010) lists out methods such as ion exchange, chemical precipitation, coagulation, phytoremediation, electrolysis and reverse osmosis as some of the effective means of removing or reducing the concentration of heavy metal ions from aqueous solution. Although these methods can achieve tremendous positive result when employed on a lab scale analysis, they have proven to be inefficient, ineffective or uneconomical for use in the treatment of large quantity of contaminated effluents Farooq et al., (2010).

Due to the recognised bioaccumulation nature of heavy metals and its nocuous tendency on living organisms, even in relatively small amount, it has become a necessity to explore highly efficient and economical way of treating solutions containing these ions (Fernanda et al., 2016). Adsorption is one of the conventional and prolific methods employed for the treatment of large volume of solutions contaminated with heavy metals ion (Shalaby et al., 2016). The major advantages

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of adsorption over other conventional methods of treatment are low cost of adsorbent/adsorbent materials, flexibility of operation and design, high efficiency, regeneration of adsorbent, simplicity and possibility of adsorbate ion/molecule recovery (Amosa et al., 2015).

The adsorption potential and capacity of several agriculturally based biosorbents such as orange peels, rice husk, neem seed, ficus carcia leaves, tamarack bark, cotton seed hulls, have reportedly been investigated for the removal of heavy metals. The results confirmed that biosorbents are highly effective for removal of ions from heavy metals-laden wastewaters due to their porosity and presence of multifunctional groups such as -OH-, -COOH-, -NH₂- on their surfaces (Ahmad et al., 2015).

Sweet potato (*Ipomoea Batatas*) is wildy grown in Nigeria and has been on an increase from 28000 hectares in 1990 to 933500 hectares in 2010 (Kathryn et al., 2012). Making Nigeria the third world producer with an annual production of 3.46 million metric tons. Its peels which are readily available, cheap and environmentally friendly are considered as wastes. The use of sweet potato peels as an adsorbent in the adsorption of heavy metals has been advocated in recent years. Batch adsorption studies for the effective removal of heavy metals using sweet potato peel which includes the adsorption of Fe (III) ions (Prasad and Abdullah, 2009), removal of Ni (II) ions (Kakanlana et al., 2012), removal of Cd (II) ions, (Okoro and Abii, 2011) have also been conducted by researchers. However, limited or no studies are available on the use of sweet potato peels for the removal of Cr (VI) ions. Therefore, this research work seeks to study the adsorption dynamics of Cr (VI) ions ion using acid modified sweet potato peel (SPP) in a continuous adsorption system.

II. MATERIALS AND METHODS

A. Materials

Sweet potato peels were obtained from local potato chips making unit in Gidan Kwano, Minna, Niger State, and Nigeria. Chemicals used for the research namely potassium dichromate (K₂Cr₂O₇) and citric acid were of analytical grade.

B. Methods

1.) Sample preparation

The sweet potato peel (SPP) collected was thoroughly washed with clean water to remove the earthly matter and all other dirt particles. This was then rinsed with deionized water to ensure neutrality of the precursor material and oven dried at 70-80°C for about 16 hours a temperature range and time when a constant weight of the SPP sample was attained and ensured most of the water was removed without decomposing the sample as recommended by Jones et al., (1991). The dried SPP was grounded and sieved to get a uniform adsorbent of size 350 µm. The SPP was placed in a plastic bottle container and kept in a desiccator before its use to preserve it and minimize contact with humidity.

2.) Modification of the adsorbent

The adsorbent was modified to enhance the pore sizes using the method reported by Lu and Liang (2013). 10 g of unmodified SPP powder was added into 1.0 mol/dm³ citric

acid (C₆H₈O₇) solution (70 ml) and the acid/powder slurry stirred for 30 minutes to mix completely and then dried without stirring for 12 hours at 50 °C and then further dried for 90 minutes at 120 °C. Thereafter, it was rinsed thoroughly to remove excess acid and finally dried at 75 °C for 16 hours.

3.) Preparation of reagent (solution)

A stock solution of 1000 mg/dm³ of chromium (VI) ion was made by dissolving 2.828 g of potassium dichromate (K₂Cr₂O₇) in 1000 cm³ volumetric flask with distilled water and made up to mark. Serial dilutions were prepared from the stock.

4.) Experimental methods

Column adsorption studies were conducted using a tubular column of size 2.74 cm in diameter and 30 cm in height. Varying bed height of 2 cm, 4 cm and 6 cm were used in the experiment to examine the effect of bed height on the adsorption process. The column was charged with the stock solution in a downward flow mode using a peristaltic pump model Longer, BQ50-1J manufactured by Longer precision company, Beijing, China. The concentration of the solution charged into the column was varied (5, 15, and 30 mg/cm³) and the effect of flow rate was studied with varying flow rates of 0.5, 1.5 and 2 cm³/min. The column was operated at sea level, and column internal pressure kept close to atmospheric pressure (Yahya and Odigure, 2015).

The aliquot was collected at 2-hour intervals, for 48 hours and then analysed using a PinAAcle Atomic Absorption Spectrophotometer model 900H manufactured by Perkin Elmer (USA) equipped with a combined flame/furnace system and continuum source background correction, was used to determine the instantaneous concentration of Cr (VI) ions in the downstream. The effect of bed height, flow rate and initial metal ion concentrations was studied. The optimum pH was taken after each sample was collected.

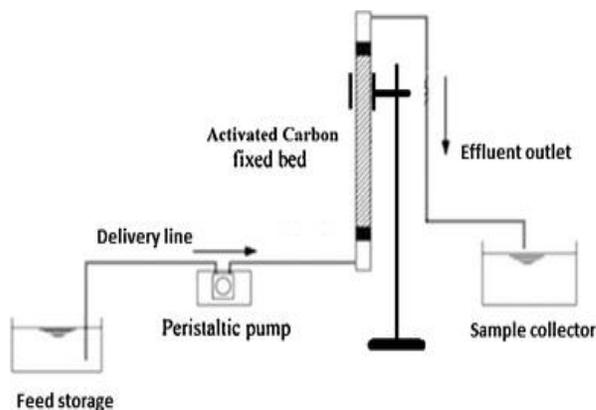


Figure1: Schematic diagram of the packed column.



Plate 1: The packed column

5.) Characterization of the raw and adsorbed SPP

Both raw and adsorbed SPP were characterised using Fourier-Transform Infrared (FT-IR) spectrophotometer for the determination of functional groups responsible for the binding of the metal ions on the material. Scanning Electron Microscopy (SEM) test indicate the morphology, pores and cavities present at high resolution while the Energy Dispersive X-ray Analysis (EDAX) was also used to determine the elemental composition of the samples.

6.) Fourier-transform infrared spectroscopy (FT-IR)

FT-IR Spectrophotometer was used before and after adsorption process for the determination of the functional groups. The percentage transmittance was plotted against wave number at a range of 400-4000 cm^{-1} . Raw sample FT-IR spectrum shown in Figure 2a indicated the major bands with peaks at range of 3420.12-3566.09 cm^{-1} indicating O-H stretches and thus representing hydroxyl groups (Cozzolino et al., 2014). During adsorption of the Cr (VI) ions, peaks at 1376.74-1507.01 cm^{-1} as shown in Figure 2b indicated spread out to several peaks, in the range of 1614.90-1738.29 cm^{-1} .

This assignation corresponded to C=O band stretch showing the presence of carboxylic acids or their esters groups as reported by Li et al. (2007) and Iqbal et al. (2009). Range of peaks from 1557.43-1634.11 cm^{-1} indicates the N-H bend showing the primary amine groups while range from 1455.92-1538.64 cm^{-1} shows the secondary amine groups. Peak at 1014.03-1339.74 cm^{-1} shifted to 1033.07-1376.82 cm^{-1} respectively indicating the presence of nitro groups while 1033.07, 721.36 and 422.93 shows C-O stretch, C-Cl stretch and C-Br stretch respectively (Cozzolino et al., 2014; John, 2000). This could be attributed to the functional groups bonding with the metal ions. Studies have also indicated that the shifting of wave number depends on the concentration of the metal present in the given sample (Ushamary and Madhu, 2014). It can also be inferred from this study that several binding and chelating groups such as hydroxyl, carboxyl and the carbonyl groups could be responsible for the biosorption of Cr (VI) ions onto the surface of the SPP.

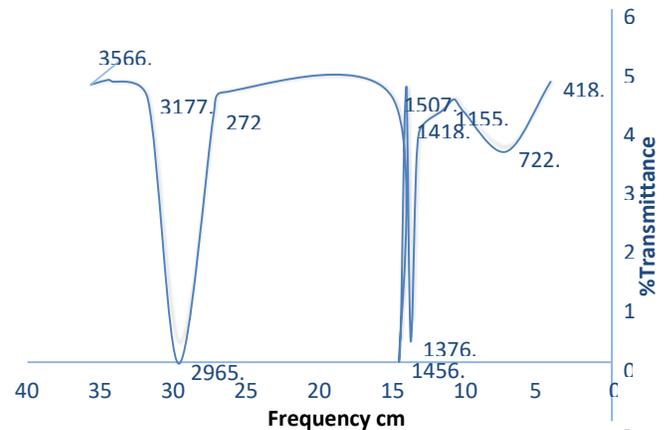


Figure 2a: FT-IR result for raw SPP.

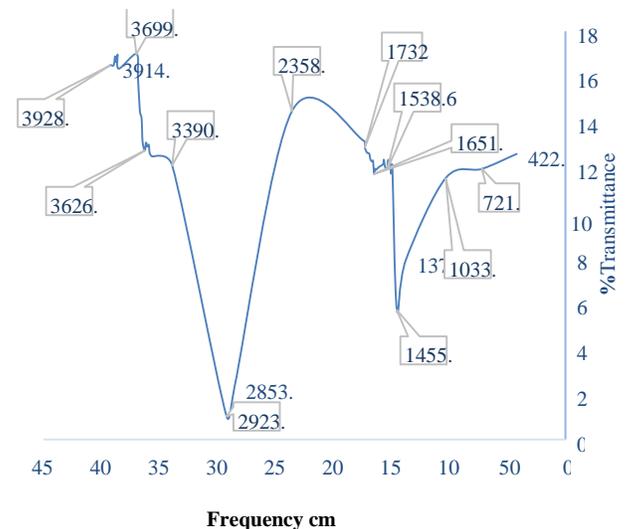


Figure 2b: FT-IR result for SPP after adsorption.

7.) Scanning electron microscope analysis

The SEM image showed the morphological characteristic of the adsorbent before and after adsorption as shown in Figures 3a and 3b. This was carried out using SEM coupled with energy dispersive X-ray spectrometry of model JEOL JMT300 machine. The SEM micrograph of the original peels showed a surface morphology that is irregular and porous which implies high surface area capable of adsorbing the chromium ions. However, after the adsorption of Cr (VI) ions, the spherical irregular surface flattened out because of the relaxation of the SPP extracellular matrix that has captured the Cr (VI) ions. The X-ray energy dispersion analysis (EDAX) results (Figures 4a and 4b) revealed the emergence of the chromium ions after the adsorption process, high concentration of K and appearance of Na and Si which may be as a results of the change in the cell wall that consist of polysaccharides especially pectin (Blanes et al. 2016).

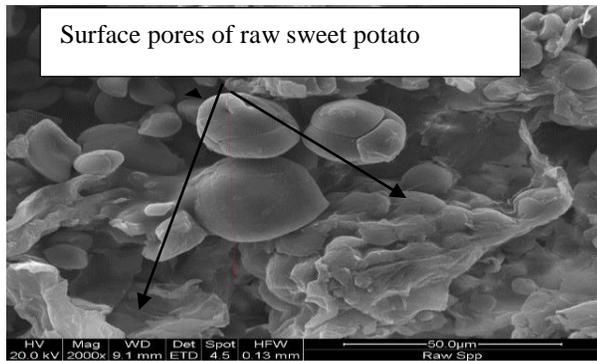


Figure 3a: Raw sweet potato



Figure 3b: SPP after adsorption.

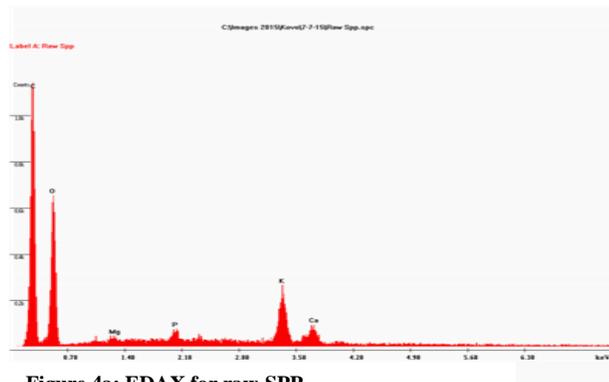


Figure 4a: EDAX for raw SPP.

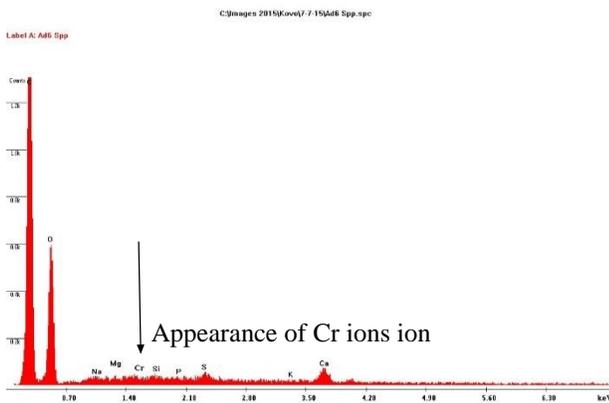


Figure 4b: EDAX for SPP after adsorption.

III. RESULTS AND DISCUSSION

A. Presentation of Results

A.1 Analysis of column data

The results obtained from the analysis of the column data are presented in Table 1.

Table 1: Column Data Calculations

Parameters	X(g)	V _{eff} (ml)	T _{total} (min)	Area(cm ²)	Q _{total} (mg)	M _{total} (mg)	Y%	q _{eq} (mg/g)
Bed height (cm) at fixed influent flow rate of 0.5 cm ³ /min and concentration of 5 mg/dm ³								
2	3.5	1440	2880	12174.96	6.0875	7.2	84.5	1.7393
4	7.3	1440	2880	12237.66	6.1188	7.2	84.9	0.8382
6	15.4	1440	2880	12340.80	6.1704	7.2	85.9	0.4007
Flowrate (cm ³ /min) at the fixed bed height of 6 cm and influent concentration of 5 mg/dm ³								
0.5	15.4	1440	2880	12340.80	6.1704	7.2	85.9	0.4007
1.5	15.4	3600	2400	9039.90	13.5599	18.0	75.3	0.8805
2.0	15.4	5280	2640	8904.90	17.8098	26.4	69.5	1.1564
Concentration (mg/dm ³) at fixed bed height of 6 cm and influent flow rate of 0.5 cm ³ /min								
5	15.4	1440	2880	12340.80	6.1704	7.2	85.9	0.4007
15	15.4	1440	2880	37132.20	18.5661	21.6	86.0	1.2056
30	15.4	1440	2880	75609.36	37.8047	43.2	87.5	2.4548

A.2 Effect of bed height

Adsorption of metals in a column of fixed dimensions depends on the amount of adsorbent in the column. Figure 5a shows the plot of normalised concentration of Cr (VI) ions against time at varying bed heights of 2, 4 and 6 cm under a fixed flow rate of 0.5 cm³/min and at constant influent concentration of 5 mg/dm³. According to Emine and Yasar (2006) the increase recorded in adsorption of Cr (VI) ions was as a result of more SPP which provide availability of binding sites and contact time. Singh et al., (2015), noted that higher bed height in column implies increase in the amount of adsorbent pore spaces available to accommodate the adsorbate in the solution and this consequently decreases the solute concentration. As the bed height increases, the gradient of the breakthrough curve decreases and thus, the extension of the mass transfer zone. The values obtained indicated that the maximum column capacity (q_{total}) and removal percentage increased from 6.088 - 6.170 mg and 84.5 - 85.7% respectively (Table 1). Similar work has been reported by Chikara et al., (2010); Malkoc and Nuhoglu, (2006).

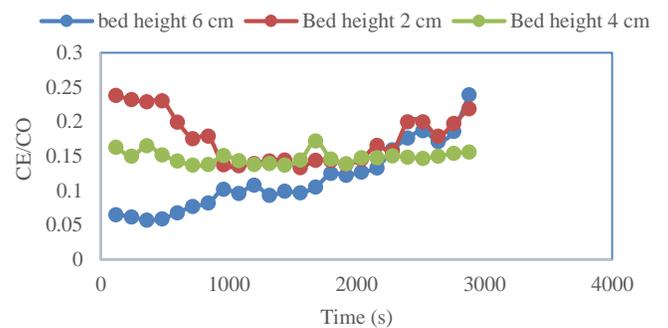


Figure 5a: Breakthrough curve for the adsorption of Cr (VI) ions on acid modified SPP at varying bed heights of flowrate at 0.5 cm³/min and initial metal concentration of 5 mg/dm³.

A.3 Effect of flow rate

Adsorption of Cr (VI) ions onto acid modified SPP was studied by varying the flow rate (0.5 cm³/min, 1.5 cm³/min and 2.0 cm³/min) at a fixed bed height of 6 cm and constant influent concentration of 5 mg/dm³. From the results obtained, it can be observed that with increasing flow rate the percentage

of Cr(VI) ions ion adsorbed decreases from 85.9-69.5% and invariably the total mass of Cr (VI) ions adsorbed increased from 6.1704 –17.8098 making the uptake to also increase. This was as a result of inadequate residence time necessary for the complete interaction of Cr (VI) ions and SPP in the column. Furthermore, there is diffusion limitations of the Cr (VI) ions into the pores of the acid modified SPP at higher flow rates (Abdul et al., 2015). From this indication, the column will perform better at a lower flow rate (Emine and Yasar, 2006).

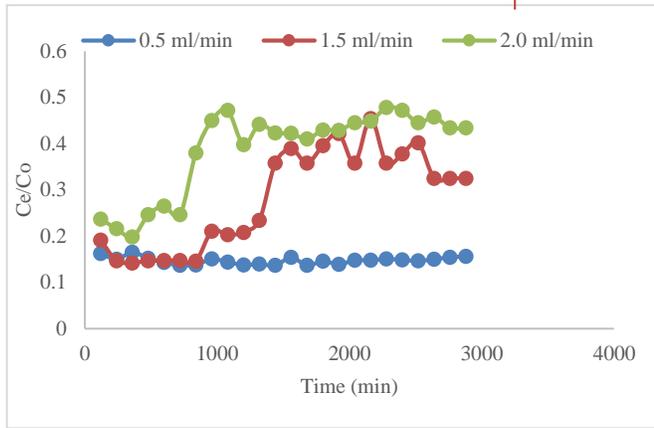


Figure 5b: Breakthrough curve for Cr (VI) ions adsorption onto SPP at varying flow rates of 6 cm bed height and metal concentration of 5 mg/dm³.

A.4 Effect of Initial Concentration

The initial concentration of Cr (VI) ions charged into the column was varied at 5, 15, and 30 mg/dm³ at a constant flow rate of 0.5 cm³/min and fixed bed height of 6 cm. The high metal ions in the solution resulted in an increase in total adsorbed metal capacity and metal uptake increased from 6.1704-37.8047 mg/g and 0.4007 - 2.4548 mg. The high concentration gradient of Cr (VI) ions ion on the SPP and the Cr (VI) ions ion in the solution served as the driving force that accelerate the adsorption process. Molecules from the region of higher concentration (solution) tends to move faster to the region of lower concentration of Cr (VI) ions (SPP surface) until equilibrium is established. Thus, the higher driving force (concentration gradient) the better the column efficiency (Chikara et al., 2010; Malkoc and Nuhoglu, 2006).

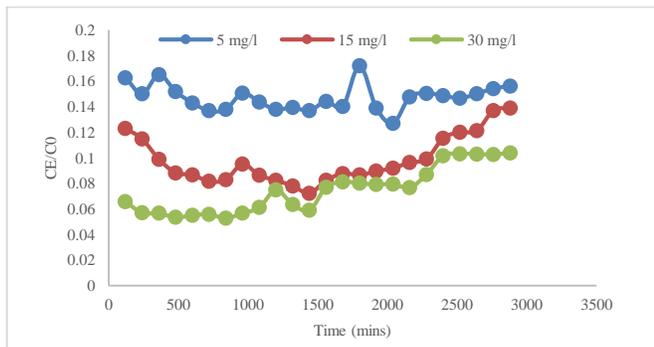


Figure 5c: Breakthrough curve for Cr (VI) ions adsorption onto SPP at varying concentrations of 0.5 cm³/min flowrate and bed height of 6 cm.

A.5 Kinetic models

In this study, Adam-Bohart, Yoon-Nelson and Bed Service Time are the empirical models used for the evaluation and correlation of the experimental data.

A.5.1) Yoon Nelson model

Yoon Nelson model was applied to the experimental data obtained to investigate the breakthrough behavior of chromium (VI) adsorption on acid modified SPP. This model use the parameter τ , which signifies time taken for the effluent Cr (VI) ions concentration (C_t) to be half of the initial concentration ($C_o/2$). The model is expressed as Equation (1).

$$\frac{C_t}{C_o - C_t} = \exp(k_{YN}\tau - t_{0.5}k_{YN}) \tag{1}$$

where k_{YN} (dm³/min) is the rate constant, $t_{0.5}$ (min) is the time required for half the total Cr (VI) ions in the influent solution to breakthrough.

The values of τ and k_{YN} were determined from the intercept and slope of the linear plot of $\ln[\frac{C_t}{C_o - C_t}]$ against sampling time (mins). where $q_o = C_o\Theta\tau$: given that Θ is the solute flow rate. The values of k_{YN} , t , and q_o were obtained and are as listed in Table 2. The Yoon Nelson’s rate velocity constant, k_{YN} , decreased with increase in bed height and flow rate, but increased with subsequent increase in initial concentration (Nwabanne and Igbokwe, 2012). There was an increase in τ as bed height increased and decreased as flow rate and initial concentration increased, this result is in agreement with several literature, (Yahaya et al. (2011); Yahya and Odigure, 2015).

Table 2: Yoon-Nelson model constants for the adsorption of Cr (VI) ions on SPP.

Q(cm ³ /min)	L(cm)	Co(mg/dm ³)	τ (min)	k_{yn}	q_o (mg/g)	R ²
0.5	2	5	5643	0.0005	14108	0.8194
0.5	4	5	12818	0.0001	32045	0.0274
0.5	6	5	1752300	0.000001	430750	0.0003
1.5	6	5	2316	0.0009	17373	0.7562
2.0	6	5	3729	0.0002	37290	0.1003
0.5	6	15	29511	0.00008	221334.4	0.0152
0.5	6	30	9584	0.0003	143760	0.4145

A.5.2) Adams-Bohart model

This continuous adsorption model was established on the basis of surface reaction theory and it assumes that equilibrium is not instantaneous therefore, the rate of the sorption is proportional to the fraction of sorption capacity still remaining in the adsorbent (Pal et al., 2013). The Adams-Bohart equation is given as:

$$\frac{C_t}{C_o} = \exp(k_{AB}C_o t - k_{AB}N_o \frac{Z}{U}) \tag{2}$$

where C_o , C_t (mg/dm³) are the column’s inlet (influent) and outlet (effluent) concentration of Cr (VI) ions ion respectively. k_{AB} (dm³/mg .min) is the kinetic constant, U (cm/min) is the linear velocity often represented as the ratio of the volumetric flow rate to the column section area, Z (cm) is the bed height

of the adsorbent in the column and N_o (mg/dm^3) is the saturation concentration. The values of N_o and k_{AB} can be obtained from the intercept and slope of the linear plot of $\ln(\frac{C_o}{C_t})$ against time (mins). After applying the equation to the experimental data for the breakthrough curve, the time of breakthrough was then obtained. The values of N_o and k_{AB} were calculated and presented in Table 3. From the Table 3, values of the rate constant k_{AB} decreases and N_o increases as the bed height increases, but with increment in flow rate and initial concentration, there was an increase in the rate constant k_{AB} but a decrease in N_o . This is due to the saturation of adsorbent's active sites which is in agreement with the result reported by Saadi et al. (2013) and Sekhula et al. (2012).

Table 3: Adam-Bohart model constants for the adsorption of Cr (VI) ions on SPP.

Q (dm^3/min)	Z(cm)	C_o (mg/cm^3)	N_o (mg/dm^3)	k_{AB} ($\text{dm}^3/\text{min.g}$)	R^2
0.5	2	5	33.99	0.00008	0.8233
0.5	4	5	37.34	0.00002	0.0273
0.5	6	5	3028.44	0.0000002	0.0003
1.5	6	5	16.89	0.00012	0.7726
2.0	6	5	73.30	0.0002	0.0932
0.5	6	15	146.94	0.0000053	0.0195
0.5	6	30	92.65	0.00001	0.4130

A.5.3) Bed-Depth Service Time (BDST) model

This model was simplified from the Adam-Bohart model, which states that bed height (Z) of a column is a function of service time (t) and the influent concentration. The equation is expressed as Equation 3.

$$t = \frac{N_o Z}{C_o U_o} - \frac{1}{K_a C_o} \ln\left(\frac{C_o}{C_t} - 1\right) \quad (3)$$

The column service time is the time taken to reach a certain percentage removal at each bed height (Mohan and Sreelakshmi, 2008). The plot of service time against bed height at a flow rate of $0.5 \text{ cm}^3/\text{min}$ (Figure 6) was linear ($R^2 = 0.9643$) indicating BDST model correlated with the experimental data.

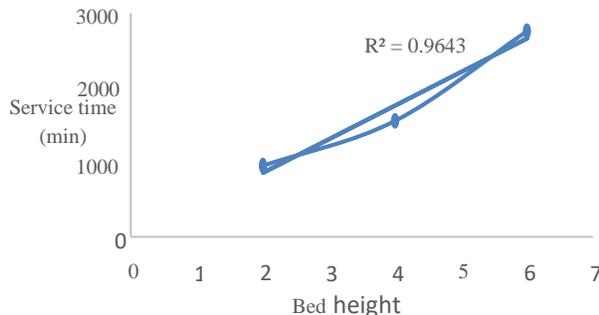


Figure 6: BDST model plot for Cr (VI) ions adsorption by SPP (flow rate = $0.5 \text{ cm}^3/\text{min}$ and initial concentration = $5.0 \text{ mg}/\text{dm}^3$)

The bed adsorption capacity, N_o , in BDST model is determined from the slope of the plot of service time, t, against the bed height, Z, where the influent concentration C_o and linear velocity of solution through the column U_o is assumed to

be constant during the column operation. The bed capacity, N_o , throughout the adsorption process was changing with time and thus used to predict the performance of the bed, if there was a change in the initial solute concentration, C_o to a new value of solute concentration. As the bed height increases, the mass transfer zone also increases and thus, allowing the adsorbate molecules enough time to diffuse deeper into the adsorbent (Malkoc and Nuhoglu, 2006).

The rate constant, K_a , calculated from the intercept of BDST plot, indicates the rate of Cr (VI) ions ion transfer from the solution to the surface of SPP (Vijayaraghavan et al., 2005). N_o and K_a were calculated to be $4.259 \text{ mg}/\text{dm}^3$ and $0.01045 \text{ dm}^3/\text{mgmin}$ respectively. The BDST model parameters can be useful to scale up the process for other flow rates without further experimental run.

B. Comparison with other Adsorbents

Table 4: Comparison of agricultural wastes adsorption capacity in the removal of Cr (VI) ions with other low-cost adsorbents.

Metal	Low-cost adsorbent	Q (mg/g)	References
Cr (VI)	Acid modified sweet potatoe peel, SPP	2.45	This study
	Soy hulls	7.286	Blanes et al., 2016
	Date Pits	1.853	Albadarin et al., 2013
	Tea Waste	2.074	Albadarin et al., 2013
	Almond shell	3.40	Pehlivan and Atun, 2008
	Walnut shell	2.28	Park et al., 2008
	Groundnuts	5.88	Agarwal et al., 2006

The adsorption capacity obtained for the modified SPP in the removal of Cr (VI) are given on Table 4. Although, the value extracted was low in comparison with other biosorbents studied, the study has demonstrated its potentials which could serve as a database for further study.

IV. CONCLUSIONS

This research work has the following conclusions:

- Use of acid modified sweet potato peels as a potential adsorbent for the removal Cr (VI) ions from aqueous solution in a fixed bed continuous system has been demonstrated.
- The effect of flow rate, bed height and initial metal ions concentration influenced the amount of Cr (VI) ions adsorbed and determines the breakthrough time. The column efficiency increased as the bed height and initial metal ions concentration increased but decreased as the flow rate increases.
- Morphological study of SPP using SEM-EDAX observed changes on the surface of SPP before and after adsorption process and the appearance of Cr (VI) ions, showed that the surface morphology of SPP is favourable for the binding of the Cr (VI) ion.

- d) FT-IR spectroscopy analysis indicated that the carboxylic groups, carbonyl and hydroxyl groups were responsible for the adsorption of the Cr (VI) ions from the effluents.
- e) Column kinetics models were applied to evaluate the experimental data. The models adequately described the adsorption of Cr (VI) ions ion on the acid modified SPP with BDST model having the best fit with the experimental data with high regression correlation coefficients, R^2 , value of 96.43%.
- f) The bed capacity and rate constant were 4.259 mg/dm^3 and 0.01045 dm^3/mgmin respectively.
- g) The various results indicated that sweet potatoes peel can be used as a biosorbent for the adsorption of Cr (VI) ions.

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