Physical Science International Journal



24(1): 1-13, 2020; Article no.PSIJ.54102 ISSN: 2348-0130

The Kinetics of the Adsorption Process of Cr (VI) in Aqueous Solution Using Neem Seed Husk (Azadirachta indica) Activated Carbon

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Authors' contributions

This work was carried out in collaboration among all authors. Author AAD designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors WLD and NYP supervised the work. Authors BM, MAA, DI, UASZ and MSM managed the analyses of the study. Author AAD managed the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/PSIJ/2020/v24i130169 <u>Editor(s)</u>: (1) Dr. Manuel Malaver de la Fuente, Universidad Maritima del Caribe, Av. El Ejercito, Catia La Mar, Estado Vargas, Venezuela. (2) Dr. Roberto Oscar Aquilano, School of Exact Science, National University of Rosario (UNR), Rosario, Physics Institute (IFIR) (CONICET-UNR), Argentina. <u>Reviewers:</u> (1) Muhammmad Raziq Rahimi Kooh Chern Yuan, Universiti Brunei Darussalam, Brunei. (2) Manohar V. Lokhande, Sathaye College, India. (3) H. Y. He, Shaanxi University of Science and Technology, China. Complete Peer review History: <u>http://www.sdiarticle4.com/review-history/54102</u>

Original Research Article

Received 24 November 2019 Accepted 27 January 2020 Published 06 February 2020

ABSTRACT

The aim of this research is the study of physico-chemical properties activated carbon prepared from agricultural by product such as Neem Seed Husk (NSH) which is abundantly available in our environment The Activated Carbon (AC) was prepared using H3PO4 as activating agent and carbonized at 300°C for two hours. The results shows that the activated process was successful and can compete favorably with commercial activated carbon. The prepared activated carbon was characterized using Fourier Transform Infrared Spectrophotometer (FT-IR), Scanning Electron Microscopy (SEM), Energy Dispersion X-ray (EDX) and Thermogravimetric Analysis (TGA). The percentage removal of Cr(VI) increased with increase in process parameters such as adsorbent

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dose, time and temperature while there was a decrease with increase in pH and Initial concentration. It was established from the results that activated carbon produced from Neem seed husk has adsorption capacity which could remove 99.75% Cr (VI) at optimum process conditions (pH-2.0, Cr(VI) concentration-10 mg/L, adsorbent dose 0.5 g/L, Temperature 70 c and contact time 30 mins.). Thus, the adsorption method using activated carbon produced from biomass was used effectively for removing Cr(VI) in a stock solution, seems to be an economical and worthwhile alternative over other conventional methods, because of it availability, low price and multipurposes. The adsorption data fitted well into Freundlich and Langmuir with correlation coefficient (R2) 0.9522 and 0.9403 respectively. The kinetics of the adsorption process was tested through pseudo-first-order and pseudo-second-order models. The pseudo-second-order kinetic model provided the best correlation for with (R2) 0.993, while the pseudo-first-order was found to be 0.928. The study provided an effective use of low-cost activated carbon as a valuable source of adsorbents for the removal of Cr(VI) ions from aqueous solution.

Keywords: Activated carbon; neem seed husk; fourier transform infrared spectrophotometer; adsorption process; pseudo-second-order models.

1. INTRODUCTION

Today, large quantities of agricultural by products are lying waste littering and polluting the environment which can be used for activated carbon (AC) production instead of synthetic AC usually prepared from petroleum sources which are expensive and non-biodegradable and therefore pose problems of environmental pollution after use.

In developing countries the demand for AC are met by importation in large quantity at high cost depleting their foreign reserves [1]. However, various types of precursors are used mainly in producing AC which are generally fossil based hydrocarbon like bitmus, coal and lignite while natural biomass is important as it is the most abundant renewable raw material [2].

In recent time, a number of agricultural and forest by products have been increasingly attracting attention as an alternatives for AC production due to their low cost, sustainability and availability [3]. Neem (*Azadirachta indica*) is a plant with lot of potentials which is widely spread in tropical countries and common in northern part of Nigeria [4,5].

Activated carbon is prepared using two methods (physical and chemical activation processes). Chemical activation method is frequently used in preparing AC and the activating agents used mainly are $ZnCl_3$, K_2CO_3 , NaOH and H_3PO_4 [6]. The use of AC in industries is very important and requires a solid having a very developed porous structure. Researchers have reported that organic matter (Biomass) is effective for the

preparation of such AC with highly porus structure [7].

This research focus to explore the feasibility of Neem seed husk based AC for the removal of Cr (VI) from ageous solution and also to optimize the absorption conditions that will ensure high Cr (VI) uptake. AC prepared from various raw materials exhibits good capacity for removal of Cr (VI) in tannery effluent, stock solution and waste water but no report to the best of my knowledge on use of Neem seed husk AC have appeared for removal of Cr(VI).

2. MATERIALS AND METHODS

The raw samples were obtained from National Research Institute for Chemical Technology Zaria and around Basawa Village, in Kaduna State.

2.1 Reagents

All reagents used were of analytical grade which are as follows: phosphoric acid, distilled water, deionized water and potassium dichromate (VI) and acetone.

2.2 Analytical Equipment and Apparatus

The equipment's used are; Fourier Transform Spectroscopy (FTIR-8400S). UV-Spectrophotometer (model JENWAY 6300), TGA (Perkin Elmer Thermal Analysis), SEM, Vacuum pump/Buchner funnel, Oven (Thermostat oven Weighing DHG-9023A), Muffle Furnace, Balance, pH Meter, Sieve, Beakers, Measuring cylinder, Conical Flask, Petri Dishes, Pestle and Mortar, Trays, Crucibles, Tongs, Water Bath (Grant JB Series), Spatula.

2.3 Sample Preparation

The raw Neem Seed Husk (RNSH) was washed with deionized water and dried at 120° C for 4 h. The dried raw material was then grounded to particle size of 125 to 250 µm. The particle size was determined using a laboratory test sieve.

2.4 Chemical Activation and Carbonization

Thirty grams of the ground raw sample was soaked in 150 ml of 50% phosphoric acid at 30°C for 48 h. The mixture was filtered out and the activated sample was carbonized in a muffle furnace at 300°C for 2 h in nitrogen atmosphere. After cooling the carbonized material then it was washed with 200 ml hot distilled water, and dried for 2 h at 120°C. The dried carbon was then weighed to determine percentage yield. This procedure can be use to prepare both Powdered Activated Carbon (PAC) and Granular Activated Carbon (GAC) [8].

2.5 Characterization of Raw Samples (Elemental Analysis)

The following physico-chemical tests were carried out to characterize the Raw Neem Seed Husk (RNSH) and Activated Neem Seed Husk (ANSH) using standard methods of AOAC (1984).

2.6 Preparation of Stock Solutions

Activating solutions of H_3PO_4 , in addition for the heavy metal solution of potassium dichromate (VI), for the adsorption tests were prepared in stock solutions of up to 1000 mg/L.

2.7 Investigation of Adsorption Potentials of Activated Carbon from Biomass

In investigating the adsorption potentials of activated carbon produced from biomass, the effect of adsorbent dose (0.1–0.5 g/L), contact time (10-50 min), pH (2.0-11), initial concentration (5–25 mg/L) and temperature ($30 - 70^{\circ}$ C) on the percentage removal of potassium dichromate (VI) were examined.

2.8 Chromium (VI) Analysis

The percentage removal of Chromium (VI) and amount adsorbed (mg/g) were calculated using the following equations;

$$\% Removal = \frac{c_i - c_e}{c_i} \times 100$$
 (1)

$$q_e = \frac{(C_0 - C_e)V}{W}$$
(2)

2.9 Langmuir Isotherm

The Langmuir Isotherm is used to obtain the maximum adsorption capacity produced from complete monolayer average of adsorbent surface. The isotherm equation gives the fractional coverage (θ) in the form as given in equation 3 and 4:

$$\theta = \frac{q_e}{q_m} = \frac{bc_e}{1+bc_e} \tag{3}$$

$$C_e/q_e = 1/bq_m + (1/q_m)C_e$$
 (4)

Which shows that a plot of (C_e/q_e) vs C_e should yield a straight line if the Langmuir equation is obeyed by the adsorption equilibrium. The slope and the intercept of this line then give the values of constant q_m and b.

2.10 Freundlich Isotherm

For adsorption from solution, the Freundlich isotherm is expressed as given by equation (5):

$$q_e = k_f \, C_e^{nf} \tag{5}$$

Where, k_f ($Mg^{1-1/n}g^{-1}$) in the Freundlich constant, which indicates the relative adsorption capacity of the adsorbent related to the bonding energy and nf is the heterogeneity factor representing the deviation from linearity of adsorption and is also known as Freundlich coefficient. The Freundlich coefficients can be determined from the plot of $\log q_e$ versus $\log c_e$ on the basis of linear form of equation is given by equation (6):

$$Log q_e = Log K_f + np \ Log \ c_e \tag{6}$$

2.11 Adsorption Kinetics

The kinetics of removal of Cr (VI) is explicitly explained below using pseudo first order and second – order models

2.11.1 Pseudo first order kinetics

The non – linear form of pseudo first – order equation is given by equation 7:

$$\frac{d_{qt}}{d_t} = k_{ad} \left(q_e - q_t \right) \tag{7}$$

Where, q_e and q_t are the amounts of Cr (VI) adsorbed ($Mg g^{-1}$) at equilibrium time at any instant of time, t respectively, and K_{ad} (1 min⁻¹) in the rate constant of pseudo first – order adsorption operation. The integrated rate law after application of the initial condition of $q_t = 0$ at t = 0, becomes a linear equation given by equation (8):

$$Log (q_e - q_t) = \log q_e - Kad t/_{2.303}$$
(8)

2.11.2 Second – order kinetics

Application of the second order kinetics has to be tested with the rate equation given by equation (9):

$$\frac{d_{qt}}{d_t} = K_2 (q_e - q_t)^2$$
 (9)

3. RESULTS AND DISCUSSION

3.1 Characterization of Adsorbent

The Table 1 shows the physical properties of the produced activated carbon and the raw samples before and after activation.

The physicochemical parameters of the raw produced biomass compared to the activated carbon shows that the activation was successful. The above process parameters are clear indications that the produced absorbent can compete favorably with commercial activated carbon. For instance the ash content of the raw samples ranges from 6.53-12.97% while the produced absorbent ranges from 1.02-8.95% showing a decrease in the ash content, which is a measure of inorganic residue, left after the organic matter has been burnt off. Other literatures reported ash content of 3.91% (Garba, 2014) also ash content from 6.56-18.70% for different particles of raw neem seed and activated neem were reported [4,5], reported also 3.39% was [9]. The moisture content of the raw samples ranges from 5.26-7.18% whereas the produced 4.36-5.70% absorbent was which when compared to the literature tend to fall within the recommended range of 3.02-10.01% [4,5]. The lower the moisture percentage, the better the yield and quality of the activated carbon [10]. The observed bulk density was in the range of 0.41-0.57 g/cm³. This is similar to Olive waste cake and Peach stones which were reported as 0.551 g/cm³ and 0.56 g/dm³ respectively [11].

3.2 FT-IR Analysis

The FTIR spectroscopy can provide basic spectra of activated carbons, especially for determination of types and intensities of their surface functional groups. The spectra of raw and activated neem seed husk are presented in Figs. 1 & 2.

According to the results, it can be observed that there was disappearance of bands when comparing the raw material spectrum with the activated carbons spectra, indicating that the chemical bonds were broken during the carbonization process followed bv the activation. The FT-IR spectra for the raw Neem seed husk has shown absorption band at 1249.91 cm⁻¹ ,1435.09 cm⁻¹ which indicates the presence of C-C, C-N and C-O. Stretching absorption band at 1712.85 cm⁻¹ is assigned to carbonyl C=O present in esters, aldehydes, ketone groups and acetyl derivatives. After activation the intensities of the absorption band increases and there is a new adsorption band at 1026 cm⁻¹ indicating the presence of C-C, C-O and C-N. When comparing the two spectra, we can observed that deep modification take place in the wave number range from 1700-750 cm⁻¹ which is similar to a deep modification in the wave number range from 1700-1000 cm⁻¹ as reported [11].

3.3 SEM-EDX Characterization

Scanning Electron Microscopy (SEM) is widely used to study the surface morphology, including the pore structure, surface structure and pore arrangement on a material surface [2].

The SEM images of the activated Neem seed husk showed an irregular and well developed porous structure indicating relatively high surface area. The external surface of the activated carbon has cracks, crevices and some grains in various sizes in large holes. The availability of pores and internal surface is requisite for an effective adsorbent [9].

The presence of carbon in the sample is further illustrated by EDX micrograph, which shows the presence of C, O, N and P, and of small amount of Ag, Si, S, Al, Na, Mg, Ca, Fe, and K. On the basis of these facts, it can be concluded that the prepared activated carbons from Neem seed husk present an adequate morphology for potassium dichromate (VI) adsorption.

3.4 Thermogravimetric Analysis (TGA)

This is a technique use in thermal analysis to measure the mass of a sample as it is heated,

cooled or held at constant temperature in a defined atmosphere. Below is the result of activated carbon produced from biomass of different samples.

Parameters	Ash content (%)	Moisture Content (%)	% Yield	Bulk Density (g/cm ³)
RNSH	12.97	6.03	-	-
ANSH	8.95	4.36	47.97	0.53
07.5				
97.5 	1285- 1295- 12	2407.24-2283.79-		941.29- 887.28- 887.28- 87.28- 702.11>
3988.06 33988.06 3780.05 3749.74 3780.60 3780.60 3780.60	3348.54 348.54 3348.54 3117.0 3117.0 3117.0 2824.18 2862 2824.18		1519.96-	249.91
		1112.85- <	1435.09-	
82.5	3500 3000 2	500 2000 1750	1500 1	250 1000 750

Table 1. Physical properties of activated carbon and raw sample

Fig. 1. Raw neem seed husk



Fig. 2. Activated neem seed husk

Table 2. EDX table for ANSH

Element	Element	Element	Atomic	Weight
Number	Symbol	Name	Conc.	Conc.
6	С	Carbon	76.87	69.34
8	0	Oxygen	17.08	20.52
7	Ν	Nitrogen	3.52	3.71
15	Р	Phosphorus	0.79	1.85
47	Ag	Silver	0.11	0.92
14	Si	Silicon	0.35	0.74
16	S	Sulfur	0.23	0.56
13	Al	Aluminium	0.27	0.55
11	Na	Sodium	0.25	0.42
12	Mg	Magnesium	0.21	0.39
20	Ca	Calcium	0.13	0.38
26	Fe	Iron	0.08	0.32
19	K	Potassium	0.10	0.28

From the chromatogram In Fig. 4, the descending TGA thermal curves indicate the occurrence of weight loss. The thermal stability of the activated carbon samples were tested by measuring the mass loss during a heating ramp rate at 30 to 905°C at 10/min. High mass loss occurred at temperature of range 100 to 250°C which is mainly attributed to the elimination of water absorbed in the pores of the carbon. In the range 300 to 400°C no considerable mass loss occurs for the treated activated decomposition carbon but the of the material continues. At 600°C there is change in atmosphere from Nitrogen to Oxygen. From 950°C combustion of carbon 650 to continues and inert inorganic residues are obtained. This is similar to the findings of Marshahida, et al. [12].

3.5 Adsorption Studies

Batch equilibrium studies:

Effect of adsorbent dose: The effect of the of adsorbent dosage on the adsorption potassium dichromate (VI) was carried out within the adsorbent dosage of 0.1, 0.2, 0.3, 0.4 and 0.5 g of activated carbon produced from ANSH as shown in Fig. 6. It can be observed that the adsorption efficiency increases with increased in adsorbent dosage from 0.1 to 0.5g of ANSH have percentage removal of 89.4%. Similar observation has been reported by Michael, et al. (2018) At a higher dosage, there is limited availability of adsorbing species for the relatively larger number of surface sites or surface area on the adsorbent [9].

Effect of pH: The effect of solution pH was studied between pH 2 to 11 with 0.5 g of an adsorbent dose and the results is shown above in Fig. 7. The results showed that adsorption was

maximum at pH of 2.0 and removal was not favored at pH above 2.0. This is related to the findings of Michael, et al. (2018). However, Merly, et al. (2013) reported that the adsorption of Cr(VI) from tannery effluent decreased with increasing pH from 2 to 5. The removal of Cr (VI) is pH dependent because the surfaces of activated carbons are negatively charged at certain pH [13]. Therefore, in this study all subsequent adsorption experiments were carried out at pH 2.0 where the highest adsorption was attained.

Effect of Concentration: The effect of initial concentration was carried out at various concentrations from 5 to 25 mg/L, 0.5 g adsorbent and pH 2. Fig. 8 shows the adsorption of Cr (VI) from the produced activated carbon in which the percentage removal of the metal ion decreased with the of increased in initial concentration the solution. This is assumed to be due to driving force of i.e concentration gradient (Shehu, et al. 2017). The trend in this finding can be explained as follows: at higher concentration, most of the Cr (VI) was left unabsorbed due to saturation of the adsorption sites [10].

Effect of Time: Contact time is a very important parameter in adsorption processes. It determines the equilibrium time of the adsorption process. The characteristics of activated carbon and its available adsorption sites affects the time needed to reach equilibrium [9]. The adsorption was studied at 10 to 50 min contact time using 0.5 adsorbent dose, at pH 2 and 5 mg/l Cr (VI) solution. The results above shows that the adsorption capacity increased with increase in contact time for ACN. This shows that the adsorbent is yet to attain equilibrium perhaps with increase in time the equilibrium can be attain.



Fig. 3. SEM for ANSH at different Nano-Space 100, 80 and 50



Fig. 4. Thermogravimetric analysis of ANSH



Fig. 5. Calibration curve for Chromium (VI)



Fig. 6. Efficiency of Cr (VI) Removal in relation to Adsorbent Dose of Activated Carbon from ANSH



Fig. 7. Effect of pH on chromium (VI) adsorption at 0.5 g adsorbent dose

Effect of Temperature: The effect of temperature was studied by conducting the experiment at different temperatures of 30 to 70°C and optimum conditions; 5 mg/L of Cr (VI) concentration, contact time of 60 min, fixed pH of 2 and adsorbent dose of 0.5 g, the result obtain is shown above temperature versus percentage removal. From the result it is observed that with

an increase in temperature the percentage removal increases, this is generally in agreement with the findings of Danbature, et al. [14].

3.6 Isotherm Studies

The adsorption isotherm gives a picture of the distribution of adsorbate species between

aqueous and solid phase - over the adsorbent surface at an equilibrium state. To design a batch sorption process, the experimental data should be fitted to different types of isotherm models. In this work, the adsorption isotherm study was carried out by using two isotherm models namely Langmuir and Freundlich.



Fig. 8. Effect of Initial Concentration; Adsorbent dose 0.5 g and pH 2



Fig. 9. Effect of contact time; Adsorbent dose of 0.5 g, pH of 2 and initial concentration of 5 mg/L



Fig. 10. Effect of Temperature; Contact time 60 min, Adsorbent dose of 0.5 g, pH of 2 and initial concentration of 5 mg/L



Fig. 11. Langmuir isotherm for ANSN



Fig. 12. Freundlich isotherm for ANSH



Fig. 13. Pseudo first-order ANSH

The Langmuir isotherm data has been linearized using the Langmuir equation and is plotted between Ce/q_e versus Ce. The Langmuir constant q_m , which is a measure of the monolayer

adsorption capacity of ANSH as 1.896 and the regression correlation coefficient R^2 is obtained as 0.9403 which indicates a good agreement between the experimental values and isotherm

parameters and also confirms the monolayer adsorption of Cr(VI) onto ANSH. The K_L is an indication of the level of interaction between the adsorbate and the absorbent [1]. The adsorption of the molecule is unfavorable if K_L >1, linear when K_L=1 and favorable for 0< K_L<1 or irreversible for K_L<0.

The applicability of the Freundlich isotherm is analyzed based on adsorption of heterogeneous surface using the same equilibrium data of Cr(VI) adsorption on ANSH. Freundlich constants K_f and n obtained by plotting the graph between logCe and logq_e. The values of K_f and n are 0.280 and 1.452 for ANSH respectively. The regression correlation coefficient obtained from Freundlich was found to be 0.9522 for ANSH. This favors Freundlich model.

3.7 Kinetics Studies

The kinetics of adsorption provides the rate of sorbate uptake onto the activated sorbent within

the equilibrium contact time. The pseudo-firstpseudo-second order order and kinetic models were implemented to evaluate the rate constant of the adsorption process. The were fitted experimental data with the aforementioned models and linear regression analyses were carried out and the constants were calculated. The linear plots of pseudo-firstorder was log (ge - gt) against t (minutes) gives the slope as the rate of reaction, k_1 and intercept of log ge. The linear plots of pseudo-second order was t/qt against t (minutes) gives 1/qe(cal) as the slope and 1/k2qe 2 as the intercept, where k₂ (g/mg-minutes) is the rate constant of the second-order adsorption.

The linearity of the plots with R^2 values that are very close to unity is an indication that the adsorption process followed both pseudo-first order and seconder order kinetics models [1]. The summary of the kinetic parameters for pseudo-first order and second order models where shown in Table 4.



Fig. 14. Pseudo second-order ANSH

Table 3. Isotherma	I constants and	regression	coefficients
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Adsorption isotherm		ANSH
Langmuir	R^2	0.9403
	K _L (L/g)	0.551
	q _m (mg/g)	1.896
Freundlich	R^2	0.9522
	K _F	0.280
	Ν	1.337

Adsorption kinetics		ANSH
Pseudo-first-order	R^2	0.928
	$K_1 \min^{-1}$	0.051
	q _e (g/g)	-0.211
Pseudo second-order	R^2	0.993
	K ₂ (g/mg/min)	0.419
	$q_e(g/g)$	0.512

Table 4. Kinetics constants and regression coefficients

4. CONCLUSION

Based on the detail experimental investigation the activated carbon was prepared using H₃PO₄ agent (chemical activation) activating as from Neem seed husk, the percentage yield was found to be 47.97%, Bulk density of the activated carbons prepared is ANSH- 0.53 g/cm³, the ash content of the raw samples ranges from 6.53-12.97% while the produced absorbent ranges from 1.02-8.95% showing a decrease in the ash content, the moisture content of the raw samples ranges from 5.26-7.18% whereas the prepared activated carbon was 4.36-5.70%. the FT-IR, SEM and TGA results were found to be promising as reported in various literatures, ANSH could remove 99.75% Cr (VI) at optimum process conditions (pH-2.0, Cr(VI) concentration-10 mg/L, adsorbent dose 0.5 g/L, Temperature 70°C and contact time 30 mins.). The adsorption data fitted well into Freundlich with correlation coefficient (R^2) 0.9522 while the correlation coefficient (R²) of Langmuir was 0.9403. The kinetics of the adsorption process was tested through pseudo-first-order and pseudo-second-order models. The pseudosecond-order kinetic model provided the best correlation (R²) 0.993 while the pseudo-firstorder was 0.928.

However, activated carbon produced from Neeem seed husk would be useful in tannery industry and water treatment plant because of its outstanding absorption efficiency. Thus, this study could add economic value to agricultural by products in Nigeria, reduce disposal problems, and offer an economic source of AC for various industries using AC.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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> Peer-review history: The peer review history for this paper can be accessed here: http://www.sdiarticle4.com/review-history/54102