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ACTIVATED CARBON BASED CANARIUM SCHEWEINFURTHII SHELLS FOR THE REMOVALOF NITRATE IONS FROM AOUEOUS SOLUTION

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ABSTRACT

The activated carbons preparation conditions from canarium scheweinfurthii shells were investigated. The two most influenced factors: the H3PO4 concentration (30 to 60 %), and the activation temperature (300 to 700°C) were studied at constant impregnation ratio of 1/1. The analysis of experimental data showed that, the optimum preparation conditions were achieved for an activation temperature of 700°C and H3PO4 concentration of 30 %. Prior to the preparation, the proximate analysis of the precursor gave, 32.70 %, 2.10 %, 63.34 % and 1.86 % for the fixed carbon, moisture, volatile matter and ash contents respectively. The iodine number was found to be 436.8mg/g and the BET surface area of 397.5 m2/g. According to the batch adsorption study, the maximum quantity of nitrate ions adsorbed was 7.40 mg/g at the following reaction conditions: 0.05 g of adsorbent, 10 mg/L of adsorbate concentration, pH of 2 and 40 minutes of contact time.

KEYWORDS: Canarium schweinfurthii shells, Chemical activation, Activated carbon, Pyrolysis, Adsorption and Nitrates ions.

I. INTRODUCTION

Nitrate pollution of ground and surface waters in Cameroon is becoming an issue of major concern [1]. Nitrates find their way into ground and surface waters through biological fixation, precipitation and anthropogenic activities such as unsewered sanitation in densely populated areas, intensive agriculture and heavy use of nitrogenous fertilizers [2]. Studies carried out in different parts of Cameroon revealed that nitrate ions concentration of groundwater were greater than the World Health Organization acceptable limit (50 mg/L). For example in the Mbanga-Njombe-Penja Banana Plain, the nitrate concentration ranged 0.05 to 146.6 mg/L[3], in the Lake Chad Basin around 1.0 to 300 mg/L[4], in Yaoundé around 0.010 to 161.0 mg/L[5] and up to 94.3 mg/L in Douala [6]. The contaminated water with nitrate ions causes fatal poisoning and methemoglobinemia also known as "blue baby" disease for children [7]. Nitrates ions in water react with amines in the diet forming carcinogenic nitrosamines which causes gastric and esophageal cancers. Nitrate pollution is also known to cause algal blooms and eutrophication. In line with research efforts to tackle and redress nitrate pollution, the valorization of biomass wastes for activated carbon production is a viable and economic option [8, 9]. In this work, we seek to optimize the preparation of activated carbons from *canarium schweinfurthii* shells (burseraceae) nutshell for its utilization for nitrates ion removal from aqueous solution [10, 11]. The nutshells are waste material that are abundantly available in the Western Region of Cameroon and seems to be a good precursor for activated carbons production [7, 12, 13]. Some previous works have been done but not given any information about the charcoal. J. R. Kana and collaborators have done the effect of biochar coal from Canarium schweinfurthii kernel on the production performances of broiler chickens fed a diet containing peanut cake as main plant protein source, but not give any characteristic about this biochar .A.S. Olawale and O.A Ajayi have done the Thermal Activation of Canarium Schweinfhurthi Nutshell and have obtained the BET surface areas of 41 1.99m²/g. U. Bassey and collaborators have done the Adsorption Isotherm, Kinetics and Thermodynamics Study of Cr (VI) ions onto Modified Activated Carbon from endocarp of Canarium schweinfurthii. And not give any characteristic of the activated carbon.. Additionally, as far as our knowledge there are no much works dealing with preparation of activated by chemical activation of *canarium*



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schweinfurthii. Therefore, it is necessary embarked to optimize the preparation conditions of activated carbons from these nutshells and to test their effectiveness in nitrates ions removal from aqueous solution.

II. EXPERIMENTAL PROCEDURE

Activated carbon preparation

The *canarium schweinfurthii* shells were collected from the locality of Bayangam in the west region of Cameroon. They were washed with tap water followed by washing with distilled water, sundried, crushed into particles sizes less than 1.0mm using a mortar and kept in an oven for 24hrs.For the chemical activation procedure, 15.0g of the sample was mixed with 15.0 mL of H_3PO_4 for the concentration of 30, 40, 50 and 60 % followed by carbonization in a Carbolite furnace at the temperature of 300, 400, 500, 600 and 700 °C. The heating rate for the pyrolysis was 10°C/min at constant resident time of 1.0 hr. After the carbonization, the activated carbons obtained were washed with distilled in order to remove the excess phosphoric acid still a neutral pH. Finally, the samples were dried in an oven at 110 °C for 24 hrs, crushed and sieved over an 80 μ m mesh and stored in a dessiccator over silica for further experiments.

Activated carbon characterization

The BET specific surface area, total pore volume and average pore diameters of the activated carbons were determined by Standard Multipoint Techniques of nitrogen adsorption, using a Tristar 3000 V6 08A (unit 1 port 2, serial: 1212) equipment. The Fourier Transform Infrared Spectroscopy (FT-IR) was performed using a BRUKER Alpha-P Spectrometer with ethanol as the solvent. The X-ray powder diffraction (XRD) was carried out using XPert MPD and Philips diffractometer and the SEM analysis was carried out using a FEI/PSEM2, EDX microscope.

Batch Adsorption Studies

Batch adsorption experiments were carried out at room temperature. The nitrate ions solution of 1000 mg/L was prepared by dissolving 7.233 g of KNO₃ in 1000 mL conical flask then completed with distilled water. A solution of 20 mL of KNO₃solution was mixed with different amounts of activated carbon (0.05 to 1.00 g) and the mixture was stirred using a magnetic stirrer at various time intervals(30-90 mins). Each solution was filtered after the reaction time and the nitrate ions concentration was determined in the filtrate using a Techmel UV-visible spectrophotometer model, USA S 23A, at a wavelength of 410 nm. The quantity of nitrate ions adsorbed at equilibrium (Qe, mg/g) and the percentage removal (%R, %) of nitrate ions from aqueous solution was calculated using the equations (1) and (2) respectively:

$$Q_e = \frac{\check{c}_O - c_T}{m} \times V (1)$$

%
$$R = \frac{c_O - c_t}{c_O} \times 100 (2)$$

III. ADSORPTION MODELING

The Langmuir and Freundlich isotherms are the two most well-known isotherms which have been used to model adsorption processes of systems.

Langmuir isotherm

The Langmuir adsorption isotherm model assumes that adsorption occurs at specific homogenous sites within the adsorbent. The linear form for the Langmuir adsorption model is given as the relation below:

$$\frac{Ce}{Qe} = \frac{1}{Q_m K} + \frac{C_e}{Q_m}(3)$$

where,

Qe (mg of adsorbate per g of adsorbent) is the adsorption density at the equilibrium solute concentration, Ce is the equilibrium concentration of adsorbate in solution (mg/L), Q_m (mg of solute adsorbed per g of adsorbent) is the maximum adsorption capacity corresponding to complete monolayer coverage, K is the Langmuir constant related to energy of adsorption (L of adsorbate per mg of adsorbent).

A plot of C_e/Q_e against C_e should give a straight line and K and Q_m can be evaluated from the slope and intercept respectively.



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Freundlich isotherm

The Freundlich adsorption isotherm is an empirical equation used to describe the adsorption in heterogeneous systems. The linear form of the equation of the Freundlich adsorption model is given below: $lnQ_e = lnK_f + \frac{1}{n}lnC_e(4)$

where,

 $K_{\rm f}$ and 1/n are empirical constants depending on the nature of sorbent and sorbate and the temperature, it indicates the strength of bond energy between sorbate and sorbent.

A plot of $\ln Q_e$ against $\ln C_o$ should give a straight line with 1/n as the slope and $\ln K_f$ the intercept.

IV. RESULTS AND DISCUSSION

Iodine number and proximate analysis results

The moisture, ash and fixed carbon contents of the activated carbon were found to be 2.10 %, 5.90 % and 88.64 % respectively. The pH_{PZC} = 2 was obtained by varying the pH of a mixture of NaCl with activated carbon [9, 14, 15]. The iodine number is defined as the milligrams of iodine adsorbed by 1.0 g of carbon when the iodine concentration of the filtrate is 0,02 N based on the procedure established by the American Society for Testing Materials (ASTM D2866-94) [10, 16]. It is a criterion used to determine the amount of active surface sites present on the activated carbon and additionally, it also indicates the degree of porosity of activated carbon. 0.1 g of activated carbon was added to 30 mL of 0.02 N iodine solution. The mixture was magnetically stirred for 3 hrs, after which 10 mL of the filtrate was titrated using 0.005N of sodium thiosulphate pentahydrate (Na₂S₂O₃.5H₂O) solution using starch as indicator.

$$2Na_2S_2O_{3(aq)} + I_2Na_2S_4O_{6(aq)} + 2NaI_{(aq)}$$
(5)

The results obtained are presented in Figure 1, as a function of the acid concentration used for activation (Figure 1a) and the carbonization temperature (Figure 1b). The iodine number increases with the decrease of H_3PO_4 concentration. Hence, an increase in the concentration of H_3PO_4 has a negative effect of the carbon porosity like the obstruction of pores. This trend is similar to results reported by others authors [12, 17]. For the chemical treatment, the iodine number increases with increase in temperature and reaches a maximum value at 700 °C. Hence, the highest iodine number was recorded to be 584.2 mg/g at 700 °C with 30 % of H_3PO_4 . The effect of carbonization time on the iodine number was evaluated at 700 °C. Figure 2 represents a curve of variation of iodine number with respect to the carbonization time. It can be seen that, the maximum development of pores is achieved after 1.0hr, thus, the greatest iodine number obtained.



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Figure 1: Iodine number of activated carbon against the activation temperature (a), Iodine number of activated carbon against the H_3PO_4 Concentration (b).



Figure 2: Determination of optimum carbonization time

BET analysis of the activated carbon

The shapes of adsorption isotherms give qualitative information about the extent of the surface area available for adsorption [9,18, 19, 20, 21]. Figure 3a and Figure 3bshow the N₂ adsorption-desorption isotherms and distribution of pore diameter of the activated carbon. The shape of this isotherm indicates that it is a type IV isotherm, with pores located in the mesopore range. This shape is also similar to type II at low pressure [22, 23, 24]. The BET specific surface area is 397.46 ± 12.94 m²/g.



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Figure 3: N₂-adsorption-desorption isotherm (a) and pore diameter distribution (b)

FT-IR Analysis of the precursor and the activated carbon

The results of the FT-IR analysis of the biomass and the activated carbon are given in Figure 4.For the biomass, the broad band at 3458.9 cm⁻¹depicts the stretching vibration of free OH and indicates the presence of adsorbed water. The band at 2953.92 cm⁻¹can be attributed to the -CH₂ symmetric stretch, the band at 1657 cm⁻¹can be attributed to the deformation bands of water molecules. The C=O bands is very sharp and it can be noticed the difference from the biomass spectra that shows a clear sharp band at 1779 cm⁻¹ that is clearly due to C=O. the sharp band at 1065.52 cm⁻¹ is affected to - CH₂ wagging. The band at stretch of 1265,83cm⁻¹ is -CH3 symmetric bending. From the spectra of the activated carbon, it can be seen that the bands at 3458.9 cm⁻¹, 1065.52 and 1750 cm⁻¹ remained after carbonization procedure. However, the width of the band at 3500 cm⁻¹ is reduced and this is probably due to the effect of phosphoric acid, indicating the removal of water molecules. A strong band at 1100 cm⁻¹ for acyl bands C-O and 1657 cm⁻¹ for the deformation bands of water molecules are also observed.



XRD analysis of the activated carbon

Figure 5 shows the diffused haloes characterized by imperfect development of peaks that appear at 2Θ values between 5.0250 to 69.9750 degrees. There are no significant distinguishable peaks. This spectrum confirms the absence of any ordered crystalline structure and consequently indicates the amorphous nature of this activated carbon. Some previous studies have confirmed this non-arrangement in the activated carbon molecular structure which is an advantageous property for a good adsorbent [25, 26, 27, 28].





Figure 5: The XRD diffractogram of the biomass

SEM Analysis of precursor and activated carbon

The SEM micrograph of biomass and activated carbon are given in Figure 6a and Figure 6b.it can be observed a highly inhomogeneous surface structure of both the activated carbon and precursor. There is also a textural change in the samples before and after carbonization, indication the presence of larges pores on the activated carbon consecutive to the thermal treatment of the precursor.



Figure 6a: SEM of the biomass (a) and of the activated carbon (b)

Batch Adsorption results

Effect of contact time

The effect of contact time was studied at an initial nitrate ions concentration of 10 mg/L (Figure 7). The time was varied from 5 to 60 minutes, with 0.05g of activated carbon. The analysis of Figure 7 reveals that, during the first 5 minutes, there is a rapid increase in the rate of adsorption, which is due to the availability of adsorption sites on the activated carbon. Followed by the decrease in adsorption trend after 5 minutes probably consequence of the insufficient of the energy of adsorption which conduct to the desorption after 5minutes and when the energy is sufficient for the adsorption, the equilibrium time is reached after 40 minutes.



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Effect of pH

The effect of pH was studied by varying the pH from 2 to 12 by maintaining the adsorbate concentration of 10mg/L and contact time of 40 min (Figure 8). The pH of the solution was adjusted using 0.1M of HCL or 0.1 M of NaOH solutions. The low adsorption quantities observed at basic pH is due to the high adsorption competition of OH⁻ ions with respect to nitrate ions and negatively charged surface of the activated carbon. Consequently, at this pH which in turn reduces the attraction of nitrate ions to the adsorbent. Hence, the optimum pH at which maximum adsorption occurred is 2.



Effect of Adsorbent Dose

The effect of adsorbent dose was studied using different adsorbent masses (0.05 to 1.00 g), at the adsorbate concentration of 10 mg/L, contact time of 40 min and pH of 2 (Figure 9). It can be observed a decrease in the quantity of nitrate ions adsorbed as the mass of active carbon is increasing. This trend can be attributed to desorption, which arises as a result of electrostatic interactions between particles of nitrate ions and adsorbent, then agglomeration of the adsorbent particles thereby decreasing the available surface for the adsorption. The mass at which maximum adsorption (30.4 mg/g) occurs is therefore 0.05 g of adsorbent.



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Effect of initial nitrate ions concentration

The effect of initial nitrate ions concentration was studied at different adsorbate concentration (10 to 70 mg/L) and at constant adsorbent dose of 0.05g, contact time of 40 min and pH of 2 (Figure 10). The initial concentration of nitrate ions increase with the increase of quantity adsorbed.



V. ADSORPTION ISOTHERM MODELS

The Langmuir and Freundlich isotherms plots are illustrated in Figure 11and their respective constant parameters are listed in Table 1. The linear correlation coefficients (R^2) of the Langmuir and Freundlich isotherm models are 0.942 and 0.956 respectively. According to these R^2 values obtained, it appears that both models are suitable to explain the equilibrium of nitrates ions adsorption on the activated carbon based canarium schweinfurthii shells. Hence, surface heterogeneity of the activated carbon is in correlation with the surface topography as described by SEM analysis. The 1/n value is less than 1, implying the strong interaction between the activated carbon and nitrate ions [29-32].

Table 1: Langmuir and Freundlich Isotherm parameters					
	\mathbb{R}^2	Qm	Kl	K _f	1/n
Langmuir	0.9423	7.407	0.00326	/	/
Freundlich	0.9562	/	/	0.000266	0.4882

11. 1 .





Figure 11: Langmuir (a) and Freundlich (b) adsorption isotherms

VI. CONCLUSION

The optimization of preparation conditions of activated carbon from *canarium schweinfurthii* shells has been achieved. The activated carbon prepared in optimum conditions was characterized and applied to the removal of nitrate ions in aqueous solution. The experimental results obtained revealed that, the adsorption of nitrates ions onto activated carbon from *canarium schweinfurthii* shells is favorable, with pH being the most determining factor in the uptake of nitrates ions removal. It was established that this activated carbon is efficient in the removal of nitrates ions from aqueous solution.

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