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ORIGINAL ARTICLE

Comparison of Freundlich and Langmuir Isotherms for Adsorption of Methylene Blue by Agrowaste Derived Activated Carbon

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ABSTRACT

Freundlich and Langmuir adsorption isotherms were used to model the equilibrium adsorption data obtained for adsorption of methylene blue on activated carbons produced from different agro wastes. Adsorption process was executed by equilibrating various quantities of activated carbon (0.1-1.0)g with 100cm^3 of methylene blue solution 250mg/l.The Langmuir isotherm parameters X_m and K_l and the Freundlich isotherm parameters K_f and n were determined from the adsorption equilibrium data for the various samples. Correlation coefficients were also determined. Important adsorption factors such as carbonization yield, ash content and percent fixed carbon were determined for each carbon sample. The equilibrium adsorption data fitted well to the two models, but gave a better fit to them Langmuir model, as was evidenced from the higher value of R^2 0f 0.9999.The Langmuir isotherm parameters X_m which measure the monolayer capacity of the adsorbent and the Freundlich isotherm parameter Kf, which indicates the extent of adsorption, follow the same trend and were good measures of the adsorption capacity of the different activated carbon sample. Percent of MB adsorbed was also estimated. Coconut shell carbon with adsorption capacity 76% and fixed carbon 88.4%, gave X_m 0.455 & K_f 0.149 while orange peels carbon of adsorption capacity of 32% and fixed carbon 56.5%gave X_m 0.174& K_f 0.109.Maize cob sample with 56.0%MB adsorption, fixed carbon 74.8% gave X_m 0.325 & K_f 0.138 Carbonisation yield for coconut shell sample was 33.69% while orange peels was 25.11% and for guinea corn stem was 30.50%.These results show that the activated carbon produced from agrowastes, with the exception of orange peel, compare favourably with commercial activated carbon.

Key words: Adsorbents, active carbon, adsorption, adsorption performance, Freundlich and Langmuir parameters

Introduction

Adsorption is a surface phenomenon that occurs when a gas or liquid solute accumulate on the surface of a solid or liquid forming a molecular or atomic film[1] Adsorption has been described as an effective separation process for treating industrial and domestic effluents. It is widely used as effective physical method of separation in order to eliminate or lower the concentration of a wide range of dissolved pollutants (organics or inorganics) in the effluent. [2]The adsorptions of various solutes on a solid remain an active area of research. However finding simple and easily performable experiments to illustrate the quantitative aspects of adsorption can be very difficult.[3]

The application of adsorption in the removal of highly toxic chromium(vi) which exist in many industrial wastewater was studied using various adsorbents. For example,anaturaladsorbent basalt,andesite and rice hulls have been used succesfully[4,5]. The importance of the study drug adsorption has been employed since pharmaceutical adssorbentis normally given to patients after ingestion

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of drug overdose or in the treatment of poisoning.[6] The use of biomaterials as biosorbent for the treatment of waste water as a potential alternative to the conventional treatment method has been confirmed in separate studies. [7,8]The importance of adsorption is seen in the need for choosing effective adsorbent for the adsorption or purification needs. The determination of adsorption capacity is a necessary perquisite for selecting adsorbent.[9] The standard analytical tests such as iodine number, phenol value or surface area estimation have been found to be misleading as predictor of adsorption.[10]The so called 'surrogate' tests were found not to necessarily relate to adsorbent's ability to adsorb the specific impurities in the process stream. [10,11]

Adsorption is usually described through an isotherm. The adsorption isotherm indicates how the adsorbed molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state [12] The adsorption capacity and also the performance of the activated carbon according to Kumal *et al.* [13] were usually predicted from equilibrium sorption isotherm. Potgieter[14] and Ochonogor and Ejikeme[15] also reported that adsorption isotherm is very useful in choosing the best carbon for adsorption or purification need.

Several models describe the process of adsorption. Although many theories of adsorption have been put forward to explain the phenomena of adsorption,[2,14,16] the isotherms of Freundlich and Langmuir had been widely used by several researchers.[12,13] Freundlich and Langmuir isotherms are of course used in adsorption to understand the extent and degree of favorability of adsorption.[17]

Therefore the main objective of this work is to undertake a comparative study of the common adsorption isotherms, Langmuir and Freundlich in the estimation and comparison of quantitative aspect of adsorption, hence relative characterization of various adsorbents. For this present work, activated carbon was selected as a reference adsorbent, being an effective adsorbent material with exceptional high surface area resulting from the large number of cave-like pores.[11,18] Hence additional objective is to produce the activated carbon from indigenous waste materials and study the carbonaceous material effect on the activated carbon performance.

Freundlich isotherm was used to analyse the adsorption efficiency of the activated carbon used in the waste water treatment.[16] Freundlich and Langmuir models was used to study the sorption process in the removal of cadmium ions from aqueous solution with powdered marble waste as adsorbent.[17] The sorption behaviour of forchlorfernuron in soil have been monitored, a good fit to linear and Freundlich isotherm was observed.[19] Nerst and Freundlich model was used to study the adsorption mechanism of an active carbon in textile and tannery effluents.[9] The sorption of zinc by clay soil under varying pH conditions has also been studied and the data were modeled to Freundlich isotherm to evaluate the sorption. [20] Similarly in the study of the removal of chromium from aqueous solution with wheat bran as adsorbent, Langmuir isotherm was used as analytical technique for the adsorption and related parameters.[21]

Materials and Methods

The materials used as adsorbent for these series of experiments were indigenous waste materials found locally in abundance. These are orange peels, coconut shell, maize cob, guinea corn stem and used tyre. The raw carbonaceous materials were analysed for carbonization yield. The active carbons obtained were also analysed for ash content and percent fixed carbons. These are factors which would influence the performance of the active carbon as adsorbent.

Production of activated carbon:

Three major processes were used in the production of activated carbon: carbonization, activation and purification [18, 24] Carbonization was carried out in a specially constructed burning chamber. It was designed to limit air supply .Each raw sample, weighing 100g, was first dried in the oven at 100^{o}C for one hour. The dried sample was charred in the burning chamber. The charred product was allowed to cool down to room temperature and weighed. This was then ground with a mortal and pestle and sieved to a workable particle size range.[18]The sample at this stage was purified in 0.5M Hydrochloric acid solution rinsed with distilled water and dried in the oven for one hour. Activation was carried out in a muffle furnace, using orthophosphoric acid as activating reagent. [18, 22] Exactly 25g of each carbonized product was weighed was placed in a beaker containing 500 cm^3 of 1.0M solution of orthophosphoric acid. The content of the beaker was mixed thoroughly and heated until it formed a paste. A crucible was cleaned and heated to a constant weight. The paste was then transferred into the crucible and this latter was placed in the furnace and activated at 500^{o}C for two to three hours. The activated product was cooled down to room temperature washed with distilled water and then dried in the oven at 100 ^{o}C for several hours.The percent fixed carbon of each activated carbon was determined by heating a known weight in a furnace at 900 ^{o}C for four hours. [22,24].

Determination of Adsorption Equilibrium Points:

A batch adsorption test was run using methylene blue (MB) a cationic basic organic dye. The absorbance of MB was measured using a spectronic 20 spectrophotometer at 650nm wavelength. Adsorption measurement for each carbon sample was made under equilibrium condition over a period of time of one hour.[11,18] The adsorbent- adsorbate equilibrium points were generated by contacting various quantities of activated carbon samples(0.1-1.0)g with 100cm^3 of MB stock solution containing 250mg/l.The mixture was agitated on a mechanical shaker for one hour, followed by filtration using What man filter paper (No1.)The filtrate containing the residual concentration of MB was determined spectrophotometrically.

Data Analysis:

Two basic approaches were used in interpreting the experimental result for adsorptive capacity. The percent of MB adsorbed (Adsorption efficiency%) was determined for each sample of activated carbon at the same equilibrium points as follows[21,22,23]

$$\% \text{ MB adsorbed} = \frac{(Ci - Cf) \times 100}{Ci}$$

where Ci pre-adsorption concentration of the solution
and Cf post-adsorption concentration of the solution

Isotherm data were analysed using Langmuir and Freundlich adsorption equations. The final expression of both equations is discussed in literature[3,11,23]. The two equations contain two adjustable parameters and different constants were generated[10,23] The Langmuir and Freundlich parameters were determined and correlation coefficients were calculated.

The linear form of Freundlich isotherm is

$$\text{Log } x = \text{LogK} + 1/n \text{ LogC}$$

where x is the amount adsorbed per gram of the adsorbent, C is the equilibrium concentration, while K and 1/n are constants. K is a function of energy of adsorption and temperature and is a measure of adsorptive capacity 1/n determines intensity of adsorption. [3,23]

The Langmuir isotherm in linear form is given as:

$$1/X = 1/X_m \,.\, K_L + 1/C + 1/X_m$$

where X_m is the monolayer capacity and K_L indicates a binding constant which is related to the heat of adsorption.

All the constants are specific to test conditions and the adsorbent type. [3]

The plots of 1/x versus 1/c and Log X versus Log C were made to test the Langmuir and Freundlich adsorption models respectively. In each case related respective constants were determined .Approximate specific surface areas were also calculated from Langmuir constant Xm using the expression S= X_m .N.a, where N is Avogadro' no and a is the area of adsorbing molecule (which for MB is 120Å) [14,24,26]

Results and Discussion

The percent yield of carbonization of the raw samples shows guinea corn stem(23.30),being the least of them, followed by orange peel (25.11), maize cob (30.50), coconut shell (33.69),while used tyre (40.79) has the highest of the samples.The results agree with the published results on the same or related materials such as in the work of Mkayula and Matunbo,in which the yield was 28.0% for cashew nut shells,32,8% for coconut shells and 20.2% for rice husks.[11]. Adediran and Nwosu obtained 38.4% for used tyre,45.3% for cow hoof and 11,6% for plantain peels.[25]

The results of percentage ash and percentage fixed carbon are presented in table 1,show that the used tyre has the least percentage ash content (6.74%) and highest carbon content(93.26%),followed by maize cob carbon (25.22% ash content and (74.78%) carbon).However orange peels carbon was the least in percentage fixed carbon(56,55%) with high ash content of 43.45%.In a related work Ogbonnaya obtained for palm kernel shell carbon (95.51%)fixed carbon,(4.49%)ash content and for cow bone carbon (95.64%) fixed carbon (4.36%) ash content. The adsorption efficiency based on MB remove for each carbon sample is also presented in table 1. The adsorption in terms of MB removed from solution is seen to be directly related with the percentage fixed carbon. Those samples with high fixed carbon also exhibited high adsorption efficiency and vice versa.

The adsorption isotherms data (Table2)were fitted to both the Freundlich and Langmuir isotherm equations. The typical graphical representations of the linearised plots are shown in figure 1 and 2 for adsorption of methylene blue on coconut shell activated carbon (csc) respectively typical for adsorption of methylene blue on coconut shell activated carbon(csc).The results shown that the adsorption process could be described well with both Freundlich and Langmuir isotherms. The experimental equilibrium data fitted well to both equations with correlation coefficient values greater than 0.9 as reported in a related work.[6,27] Although both isotherms describe the adsorption process very well, Langmuir isotherm for each sample of carbon adsorbent fitted the behaviour better with higher correlation coefficient recorded for each sample as shown in table 3.The Langmuir model was also found to give a better fit in the adsorption of phenol by water hyacinth ash.[3]This better fit of equilibrium data to the Langmuir isotherm suggest monolayer coverage of MB on the carbon adsorbent.[3,18]

The Langmuir and Freundlich constants determined from the slopes and intercepts of the respective plot are summarized in table 3. Although the constants are specific to test conditions and adsorption type, the results indicate comparable assessment of carbon adsorbents prepared and investigated.The constant Xm in the Langmuir isotherm which represents a practical limiting adsorption capacity is useful in comparing adsorption of different adsorbents performance. The value of Xm relates directly with MB adsorbed from solution, for example UTC which removed much of MB gave high Xm,while the reverse was the case for OPC and GSC.The values of Freundlich constant K_f in the result indicate the extent of MB removal, hence a measure of adsorption capacity as Langmuir Xm. Thus CSC and UTC having large Xm of 0.455 and 0.442 respectively also have high K_f of 0.148 and 0.137 respectively, while OPC with low Xm of 0.256 also has a low K_f of 0.118 therefore Langmuir Xm and Freundlich K_f do indicate and compare adsorption performance, but from the Langmuir Xm the specific surface area can be estimated. In one report different adsorbents weregraded using Freundlich adsorption parameter,K_f.[25] In the study of adsorption of chlorpheniramine, The Freundlich parameter K_f was used in the grading the adsorbents,with the lower grade talc powder giving 1.91 while the best grade activated charcoal gave 4.68.[6] Also in the kinetics equilibrium modeling of nickel adsorption by cassava peels the equilibrium data were fitted to Freundlich and Langmuir isotherms which gave K_f 1.85 and Xm 4.74 respectively, both constants indicating adsorption capacity.[12]

The adsorption parameter n in the Freundlich isotherm which measures preferential adsorption of one adsorbate to other, the intensity of adsorption compared directly with Langmuir constant K_L a factor which relate to heat of adsorption and affinity to the binding site.[12,28,29] The two constants K_L and n were observed to be proportionally related for each sample of carbon adsorbent. High value of n indicates a strong bond between the adsorbent and the adsorbate a desired parameter in dyeing technology. [9]The value of n is >1 for each sample (table 4).A similar result was obtained in the adsorption of MB from aqueous solution on the surface of wool fiber cotton fiber. [23]Also in another related work 0.504 was obtained as 1/n.[12] The Langmuir constant X_m is found related to the specific surface areas. [14,24,26]The estimated surface areas in comparison with the adsorption performance of the samples are presented in table 4. The results obtained are found to be in the same range with activated carbon from similar materials. [11,22]Coconut shell carbon with the highest adsorption efficiency of 76.0% has the estimated area of 923m^2/g while orange peels carbon with the lowest 32.0% adsorption efficiency has the area 353 m^2/g

With the exceptionion of activated carbon produced from orange peels, the quality of activated carbon produced as adsorbent was found to be comparable with that of commercial ones.For example samples of activated carbon from coconut shells and used tyre as in table 1 and 3 gave high adsorption efficiency Table 1 & 3. They were followed by the samples from maize cob and guinea corn stem, while orange peels carbon gave a low result. The results of these samples in this study are in the same range with those reported for activated carbons from similar and related raw materials [1116,22,25].

Summary and Conclusion:

In this study both Freundlich and Langmuir isotherms have been used to describe and illustrate adsorption process. The application of the constants in the evaluation and comparison of quality and

Table 1: Ash Content, % Fixed Carbon & Adsorption Efficiency %

Activated Carbon Samples	Ash Contents%	Fixed Carbon%	Adsorption Efficiency %
Orange peel Carbon (OPC)	43.45	56.55	32.0
Guinea Corn Stem Carbon (GSC)	32.70	67.30	51.0
Maize Cob Carbon (MCC)	25.22	74.78	56.0
Used Tyre Carbon (UTC)	6.64	93.26	68.0
Coconut Shell Carbon (CSC)	11.6	88.46	76.0

Table 2: MB Adsorption on Coconut Shell Carbon (CSC)

Post Adsorption [MB] x10(mg/l)	MB Adsorbed (mg)	Act.Carbon Dosage (mg)	MB Adsorbed per unit Mass of Carbon(g/g)	Langmuir Data		Freundlich Data	
c			x	1/c	1/x	Log c	Log x
2.00	23.00	100	0.230	0.500	4.348	-0.301	-0.638
1.00	24.00	150	0.160	1.000	6.250	0.0	-0.796
0.55	24.45	250	0.097	1.818	10.309	-0.260	-0.132
0.42	24.58	300	0.082	2.381	12.195	-0.377	-0.086
0.36	24.64	350	0.071	2.777	14.085	-0.444	-1.149
0.33	24.70	400	0.062	3.333	16.129	-0.523	-1.208
0.26	2474	450	0.055	3.846	18.182	-0.585	-1.260
0.24	24.76	500	0.049	4.167	20.408	-0.620	-1.310
0.21	24.79	550	0.045	4.762	22.222	-0.678	-1.347

Preadsorption [MB] = 250 mg/l

Table 3: Langmuir and Freundlich Parameters for adsorption of MB on CSC

	Freundlich Data		Langmuir Data		Freundlich constants		Langmuir constants		Correlation coefficients	
SAMPLE	SLOPE 1/n	Log K	SLOPE 1/XmK	$1/X_m$.	n	K	X_m.	K	Freundlich	Langmuir
GSC	0.513	-0.927	3.640	3.91	1.950	0.118	0.259	1.061	0.9834	0.9996
MCC	0.660	-0.861	3.819	3.08	1.515	0.138	0.325	0.806	0.9967	0.9993
UTC	0.703	-0.880	4.777	2.26	1.422	0.132	0.442	0.474	0.9914	0.9987
OPC	0.271	-0.961	2.121	5.76	3.686	0.109	0.174	2.709	0.9548	0.9993
CSC	0.739	-0.828	4.235	2.20	1.353	0.149	0.455	0.519	0.9962	0.9995

Table 4: Summary of the Estimated Specific Surface Area Compare with Adsorption Efficiency.

Activated Carbon Samples	Specific Surface Area m^2/g	Adsorption Efficiency %
Orange peel Carbon (OPC)	353	32.0
Guinea Corn Stem Carbon (GSC)	519	51.0
Maize Cob Carbon (MCC)	659	56.0
Used Tyre Carbon (UTC)	899	68.0
Coconut Shell Carbon (CSC)		76.0

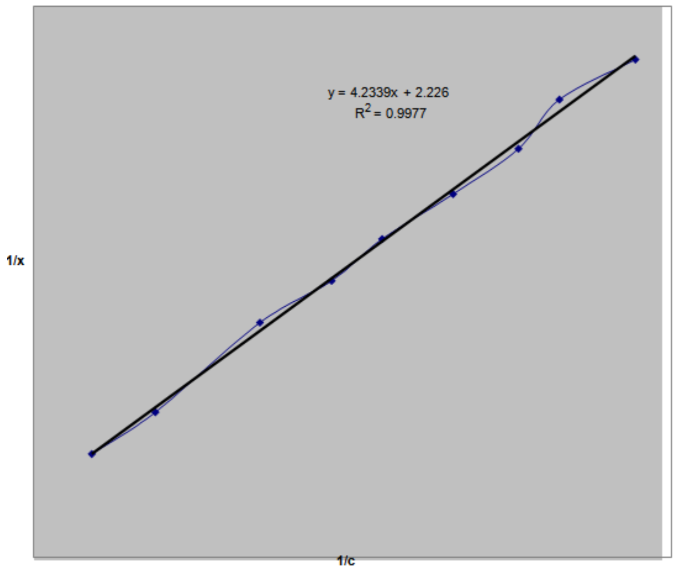

Fig. 1: Application of Langmuir equation to the experimental data points determined for the adsorption of MB on the coconut shell carbon (csc)

The straight line represent a linear regression fit to the result
x is the mass of MB per unit mass of activated carbon.
c is the concentration after adsorption
R is the regression co-efficient

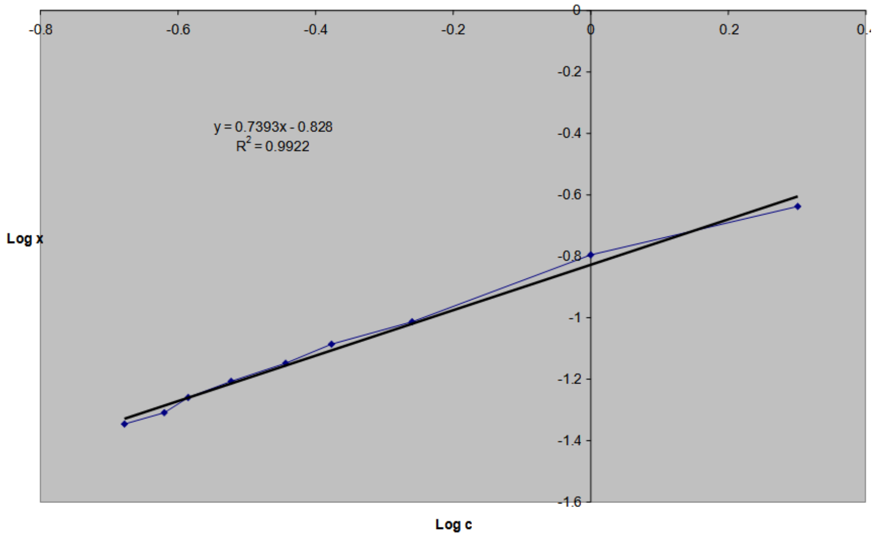

Fig. 2: Application of Freundlich equation to the experimental data points determined for the adsorption of MB on the coconut shell carbon (csc)

The straight line represent a linear regression fit to the result
x is the mass of MB per unit mass of activated carbon.
c is the concentration after adsorption
R is the regression co-efficient

capacity of adsorption was also demonstrated. The experimental data in the adsorption studies were fitted to Freundlich and Langmuir equations to determine the extent and degree of favourability of adsorption. In addition Langmuir Xm affords comparison f adsorption performance and the estimation of specific surface area.

The various adsorption parameters obtained for the activated carbon produced from waste materials showed that are good adsorbents with the exception of that from orange peels.

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