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EQUILIBRIUM AND KINETICS OF COLOUR ADSORPTION FROM TEXTILE WASTEWATER BY A NOVEL ADSORBENT

G. D. AKPEN, M. I. AHO AND M. H. MAMWAN

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ABSTRACT

Activated carbons were produced from a local variety of mango seed by carbonizing the inner seed before activation with $ZnCl_2$ using an impregnation ratio of 1:4. Laboratory batch studies were conducted in order to generate data for kinetic and equilibrium modelling of adsorption of colour by the produced mango seed endocarp activated carbon (MSEAC) for the purpose of determining the mechanism and rate controlling steps of the adsorption process. Langmuir, Freundlich and Tempkin Isotherms were adopted. While the kinetic models used were pseudo first order kinetic, pseudo second order kinetic, Intra-particle diffusion and Elovich models. The pseudo second order model gave the highest coefficient of determination (R^2) of 0.999 (indicating goodness of fit) compared to other kinetic models tested. This suggests that chemisorption is the rate-limiting step in this biosorption system. Langmuir isotherm fitted experimental data better compared with the other tested models(R^2=0.9987), implying that the adsorption process is based on a monolayer adsorption**.** Chi-squared test performed on all the models confirmed the goodness of fit of the generated data to the Langmuir and pseudo-second order over the other isotherm and kinetic models because they had the least caculated Chi- squared (χ^2_{Cal}) values of 5.2 and 3.2 respectively. It was recommended that these two models are reliable tools for predicting the mechanism and rate of adsorption of colour from textile wastewater by the produced MSEAC.

KEYWORDS: Mango seed endocarp, activated carbon, colour, kinetics, isotherms, Textile wastewater.

INTRODUCTION

Activated carbon has been used worldwide as an effective adsorbent for removing colour and chemical contaminants from water and waste water. Currently, Nigeria imports commercial carbon at high cost for water treatment which puts significant burden on the water treatment budget. A review of literature revealed many attempts on the use of agricultural wastes/by-products as adsorbents for water/wastewater treatment. The adsorption of phosphate by Terminalia catappa based activated carbon was studied by Ochonogor *et al*. (1999). Activated carbon was produced from Rosa canina and applied in the removal of dye from wastewater (Gurses *et al*., 1999). Rice husk and corn-cobs based activated carbon was produced and characterized by Aloko and Adebayo (2007) and used to remove phosphates from water. Jatropha husk activated carbon was used by Namasivayam *et al*. (2007) for the removal of anions, heavy metals, organics and dyes from water. High removal efficiencies were achieved by mango seed shell activated carbon in the removal of colour and phenol from wastewater (Akpen *et al*. 2011, 2014].

However, scanty information is available on the use of mango seed endorcarp as adsorbent, though the potential for its use has been recognised by some researchers (Elizalde-Gonzalez & Hernandez-Montoya, 2007, Kwagher and Adejoh, 2012). In Nigeria, mango seed litter around streets especially in the suburban areas and they constitute environmental nuisance. The utilization of these seeds for wastewater treatment will have cost saving and waste management advantages. The aim of this study is to investigate the mechanism and nature of adsorption of colour from wastewater by mango seed activated carbon. The preparation, characterization and the optimum conditions for the removal of colour by MSEAC are reported elsewhere (Akpen *et al*., 2017).

MATERIALS AND METHODS

Production of Mango Seed Endocarp Activated Carbon (MSEAC)

Mango seeds were collected from Mangar District of Bokkos Local Government Area of Plateau State, Nigeria, air-dried and broken to expose the endocarp which was further air dried. The air dried mango seed endocarps were carbonized in a muffle furnace at a temperature of 500^0C for 2 hours and allowed to cool for 3 hours before activation with $ZnCl_2$ solution. The $ZnCl_2$ solution was prepared by dissolving 100 g of anhydrous $ZnCl_2$ in 100 mL of distilled water. 400g of the

G. D. Akpen, Civil Engineering Department, University of Agriculture, Makurdi, Nigeria.
M. I. Aho, Civil Engineering Department, University of Agriculture, Makurdi, Nigeria.
M. H. Mamwan, Civil Engineering Department, University of Agriculture, Makurdi, Nigeria.

carbonized material were mixed with the prepared $ZnCl_2$ solution to obtain an impregnation ratio of 1:4.That is 1 part of $ZnCl_2$ to 4 parts of carbonized mango seed endocarp by mass.

Adsorption studies

Batch kinetic studies were carried out by adding a fixed amount of MSEAC of 0.8 g (size 150 µm-850 µm) into 500 mL flasks containing 100 mL of textile wastewater obtained from Angel Spinning Textile Industry, Kano. The flasks were agitated in a flocculator at 90 rpm at room temperature (± 34^0C) at preset time intervals of 10 min, 20 min, 30 min, 40 min, 50 min, 60 min and 70 min respectively. The initial concentration of colour, pH, and carbon dose adopted were respectively 512 Pt-Co units, 7.6 and 0.8g. Colour concentrations were measured by a DR 2000 UV spectrophotometer (HACH Company, USA) according to the standard method specified in the manual. The amount of colour adsorption per unit mass of adsorbent, q_t (Pt-Co/g) was calculated according to Equation (1):

$$q_t = \frac{(C_o - C_t)}{W} V. \qquad \text{......................................} \qquad (1)$$

where C_0 and C_t (Pt-Co) are the liquid-phase concentrations of colour at initial and at preset time, t respectively. V (L) is the volume of the solution and W (g) is the mass of dry adsorbent used.

The procedures of equilibrium experiments were basically identical to those of kinetic tests except that different initial concentrations of 215, 302, 399, 427, 450, 512 and 550 Pt-Co units of the coloured textile wastewater of pH 7.6 were adopted. The time to reach equilibrium adopted was 40 minutes. The amount of equilibrium adsorption, q_e (Pt-Co /g), was calculated using Equation (2):

$$q_e = \frac{(C_o - C_e)}{W} V. \qquad \text{..} \qquad (2)$$

where C_e (Pt-Co) is the liquid-phase concentration of colour at equilibrium.

Adsorption Isotherm Modelling

The equilibrium adsorption isotherm is one of the most important approach to understand the mechanism of adsorption systems. The Langmuir (1918), Freundlich (1906) and Temkin (Temkin and Pyzhev, 1940) isotherm equations were used to interpret the experimental data. Langmuir isotherm theory is based on the assumption that adsorption is on a homogeneous surface, i.e., the surface consists of identical sites, equally available for adsorption and with equal energies of adsorption, and that the adsorbent is saturated after one layer of adsorbate molecules forms onto the surface The linearized form of the Langmuir adsorption isotherm equation is expressed as in Equation (3):

$$\frac{c}{q} = \frac{c}{q_n} + \frac{1}{K_a q_n}. \qquad \text{....................................} \qquad (3)$$

where, q = mass of solute adsorbed/mass of adsorbent; q_n = mass of solute adsorbed/mass of adsorbent for a complete monolayer, c = concentration of solutes in solution; and K_a = constant related to enthalpy of adsorption (Langmuir (1918). The Langmuir constants; are obtained by plotting c/q against c. The slope, a =1/ q_n and intercept, b = 1/ K_a q_n.

The Freundlich isotherm equation is an empirical expression that encompasses the heterogeneity of the surface and the exponential distribution of sites and their energies and is expressed as:

$$q = K_f C^{\frac{1}{n}}. \qquad \text{..} \qquad (4)$$

In linearized form, Equation (4) is given by Equation (5) thus:

$$\text{In}\, q = \text{In}\, K_f + \frac{1}{n} \text{In}\, C. \qquad \text{....................................} \qquad (5)$$

where, C is the equilibrium concentration of the solute (mg/L) and q is the equilibrium
adsorption capacity (mg/g). K_F and 1/n are constants representing the adsorption capacity and the intensity of the adsorption respectively and can be calculated from the linear plot of In *q* versus In *C*. The slope (1/n) measures the surface heterogeneity. Heterogeneity becomes more prevalent as 1/n gets closer to zero (Valix *et al*., 2004).

The Temkin isotherm equation assumes that the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent-adsorbate interactions, and that the adsorption is characterized by a uniform distribution of the binding energies, up to some maximum binding energy. The linearized Temkin isotherm is given in Equation (6):

$$q = B\text{ln}\, K_T + B\text{ln}\, C. \qquad \text{....................................} \qquad (6)$$

where, B = RT/b. K_T is the equilibrium binding constant (L/mg) corresponding to the maximum binding energy and b is a constant related to the heat of adsorption. R is the gas constant (8.314 J/mol K) and T is the absolute temperature (K). A plot of q versus ln C enables the determination of the isotherm constants K_T and b from the intercept and slope respectively.

Adsorption Kinetics Modelling

The kinetics of adsorption data was processed to understand the dynamics of adsorption process in terms of the order of rate constant. The rate constants of chemical adsorption of colour from the textile wastewater were determined using the pseudo-first order, pseudo-second order, Elovich (Chien and Clayton, 1998), and the intra-particle diffusion (Weber and Morris, 1963) models.

The pseudo-first-order rate expression based on solid capacity is generally expressed as follows (Lagergren, 1898):

$$\frac{dq_t}{dt} = K_1(q_e - q_t). \qquad \text{.......................................} \qquad (7)$$

where, $\frac{dq_t}{dt}$ is the rate of adsorption, q_e is the amount of adsorbate adsorbed at equilibrium (mg/g), q_t is the amount adsorbed at any time t (mg/g), k_1 is the rate constant of first order adsorption (l/minutes). After integration and applying boundary conditions, t = 0 to t and q_t = 0 to q_t; Equation (7) becomes:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t. \quad \dots\dots\dots\dots\dots \quad (8)$$

Values of adsorption rate constant (k_1) for the adsorbate are determined from the straight line plot of log (q_e – q_t) against t. A high value of the coefficient of determination is an indication that the rate of removal of the adsorbate by the adsorbent follows the pseudo-first-order equation.

The pseudo-second-order equation is also based on the sorption capacity of the solid phase. It predicts the behaviour over the whole range of data. Furthermore, it is in agreement with chemisorption being the rate controlling step and is expressed as (Ho *et al*., 2000):

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2. \quad \dots\dots\dots\dots\dots \quad (9)$$

where, k_2 is the rate constant of second order-adsorption (g/mg.min). $\frac{dq_t}{dt}$, q_t, q_e and t are as defined earlier. For the same boundary conditions as in Pseudo-first order model, the integrated form of Equation (9) becomes:

$$\frac{1}{q_e - q_t} = \frac{1}{q_e} + k_2 t. \quad \dots\dots\dots\dots\dots \quad (10)$$

Equation (10) can be rearranged to obtain:

$$q_t = \frac{t}{\frac{1}{k_2 q_e^2} + \frac{t}{q_e}}. \quad \dots\dots\dots\dots\dots \quad (11)$$

Equation (11) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t. \quad \dots\dots\dots\dots\dots \quad (12)$$

If the initial sorption rate, h (mg/ g. minutes) at t = 0 is defined as:

$$h = k_2 q_e^2. \quad \dots\dots\dots\dots\dots \quad (13)$$

Then replacing $k_2 q_e^2$ in Equation (12) with h, we obtain:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}t. \quad \dots\dots\dots\dots\dots \quad (14)$$

Equation (14) does not have the disadvantage of the problem with assigning an effective q_e. If pseudo-second order kinetics is applicable, the plot of t/q_t against t of Equation (14) should give a linear relationship from which q_e and h values will be determined from the slope and intercept of the plot respectively and there is no need to know any parameter beforehand (Ho and McKay, 1998). k_2 is then determined from Equation (13).

The Elovich model is mainly applicable for chemisorption kinetics. The equation is often valid for systems in which the adsorbing surface is heterogeneous (Sivakumar and Palanisamy, 2009). The Elovich model is generally expressed as:

$$\frac{dq_t}{at} = \alpha e^{-\beta q_t}. \quad \dots\dots\dots\dots\dots \quad (15)$$

To simplify the Elovich equation, Chien and Clayton (1998) assumed that αβt>>1 and integrating Equation (15) for the boundary conditions, qt = 0 at t = 0 and q_t = q_t at t = t yields in linear form:

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t. \quad \dots\dots\dots\dots\dots \quad (16)$$

where, α is the initial adsorption rate (mg/g minutes) and β is the desorption constant and is related to the extent of surface coverage and the activation energy for chemisorptions (g/mg). q_t, q_e and t are as defined under Equation (7). A plot of q_t against ln t gives a linear trend with a slope of (1/ β) and an intercept of 1/ β ln (α β).

The most commonly used technique for identifying the mechanism involved in the adsorption process is by using intra-particle diffusion model as proposed by (Weber and Morris, 1963) thus:

$$q_t = k_d t^{1/2} + I. \quad \dots\dots\dots\dots\dots \quad (17)$$

where, K_d is the intra-particle diffusion rate constant. q_t and t are as defined under Equation (7). If intra-particle diffusion occurs, then a plot of q_t against $t^{1/2}$ will be linear and the line will pass through the origin if intra-particle diffusion was the only rate limiting parameter controlling the process. Otherwise, some other mechanism such as external mass transfer is also involved. Values of I give an idea about the thickness of the boundary layer.

Model Verification

To test the reliability of the developed models for prediction of the adsorption process, a chi-squared (χ^2) test was performed according to Equation (18) thus:

$$\chi^2 = \sum_{i=1}^{N} \frac{(O_i - e_i)^2}{e_i} = \sum \left| \frac{(\text{Observed} - \text{Expected})^2}{\text{Expected}} \right|. \quad \dots\dots\dots \quad (18)$$

where, N = independent observations in the sample and k =1 is the population parameter. N = 7 for both the kinetic and equilibrium data. The results of the chi-squared test are presented in Table 4.

RESULTS AND DISCUSSIONS

Characteristics of Textile Wastewater

The results from the characterization of Textile waste water are presented in Table 1. The concentrations of BOD and COD were higher than values specified by National Environmental Standards Regulation and Enforcement Agency (NESREA). Other parameters analysed were within acceptable limits specified by NESREA except, the five-day dissolved oxygen concentration.

Table 1: Characteristics of Textile Wastewater

Wastewater parameter	**Value**	**NESREA standards**
pH	7.6	6-9
Colour [Pt-Co units ≡ mg/L]	512	25
Chloride [mg/L]	134.6	600
Total dissolved solid, TDS [mg/L]	176.8	500-1500
Suspended solid, SS [mg/L]	314	30
Total Solid, TS [mg/L]	490.8	2000
Dissolved oxygen - initial [mg/L]	5.0	4.0
Dissolved oxygen – 5 day [mg/L]	1.4	Not <2
BOD [mg/L]	214	20
COD [mg/L]	428	80
Total coliform [MPN/100 mL of H_2O]	550	400

Adsorption Isotherms Results

The equilibrium adsorption isotherm is one of the most important data to understand the mechanism of adsorption systems. Values of all the isotherm constants as obtained are shown in Table 2 along with the values of the coefficient of determination (R^2). The Langmuir model fitted the experimental data better than the other isotherm models given the higher values of R^2 obtained. Conformation of the experimental data to the Langmuir isotherm indicates the formation of a monolayer of colour molecules at the outer surface of the mango seed endocarp activated carbon. It further implies that, the adsorbed molecules cannot migrate across the surface or interact with neighbouring molecules (an indication of chemisorption). Similar observations were reported by Bello *et al*. (2010) on the adsorption of acid orange 10 dye onto treated sawdust and Namasivayam and Kavitha (2002)] on the adsorption of Congo red dye on treated sawdust from coir pith.

Adsorption Kinetics Results

The intra-particle diffusion plot presented in Fig.1 indicates that the linear plot for a wide range of contact times did not pass through the origin. This deviation from the origin may be due to the variation of mass transfer in the initial and final stages of adsorption as reported by (Sivakumar and Palanisamy, 2009). This indicates that although intra-particle diffusion was involved in the adsorption process, it was not the sole rate-controlling step. This also confirms that adsorption of colour on the adsorbent was a multi-step process, involving adsorption on the external surface and diffusion into the interior. The kinetic parameters for the adsorption of colour from the textile wastewater were calculated as presented in Table 3. The coefficient of determination (R^2) for the pseudo-second-order kinetic model was higher than the other kinetic models tested implying that the adsorption system studied obeys the pseudo second-order kinetic model. This suggests that the rate-limiting step in these biosorption systems may be chemisorptions involving valence forces through sharing or exchange of electrons between adsorbent and the adsorbate as reported by (Ho and McKay, 1999).

Table 2: Isotherm parameters for the removal of colour by MSEAC.

Equilibrium isotherm	Equilibrium constants/parameters	Goodness of fit (R^2)
Langmuir isotherm $c/q = c/q_n + k_a/q_n$	q_n = 588.23 [Pt-Co/g] K*a*= $8.5x10^{-7}$ [$Pt\text{-}Co^{-1}$]	0.998
Freundlich isotherm $Inq = Ink_f + 1/n\ Inc$	n = 10.08 K_f = 386.75	0.4994
Temkin isotherm q= BInA + BInC	A = 10.54 [kJ] B = 43.09 [J]	0.5698

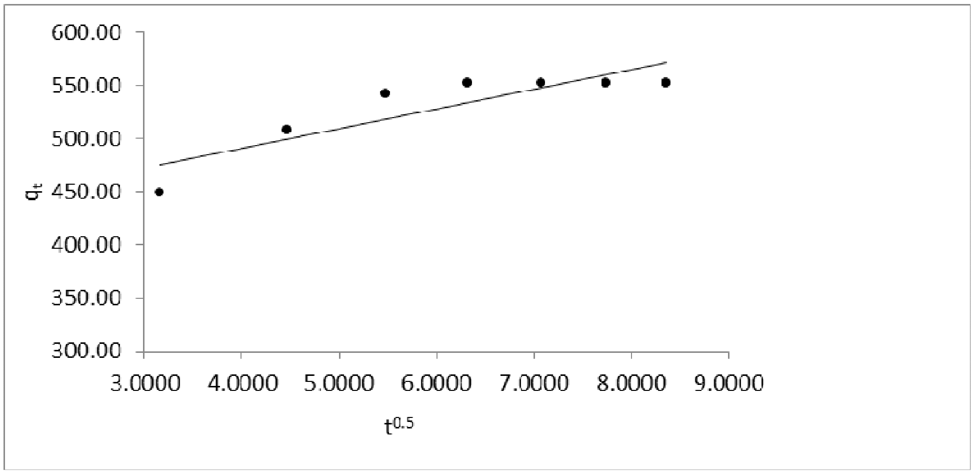

Fig.1: Intra-particle diffusion model

Table 3: Kinetic model parameters for the removal of colour by MSEAC

Kinetic model	Rate constants	Goodness of fit (R^2)
Pseudo-first order model	K_1= 0.002993 [min^{-1}] q_e = 138.36 [Pt-Co g^{-1}]	0.818
Pseudo-second order model	K_2 = 0.00025 [g Pt-$co^{-1}$$min^{-1}$] q_e = 1000 [Pt-Co g^{-1}] h = 250 [Pt-Co $g^{-1}$$min^{-1}$]	0.999
Intra-particle diffusion model	K_d = 18.55 [min^{-1}] I = 417	0.764
Elovich model	β = 0.0187 [g Pt-co^{-1}] α =31844.2 [Pt-Co $g^{-1}$$min^{-1}$]	0.871

Results of Chi-Squared Test:

The results of Chi-squared test on the various isotherm and kinetic models are presented in Table 4. Langmuir isotherm had the least calculated chi- squared (χ^2_{Cal}) value of 5.2 among the isotherms studied, implying it's the best model because it fits the experimental data better than the other models. The Pseudo-second order model emerge the best kinetic model with χ^2_{Cal} value of 3.2. Results of the chi-squared test is in line with the R^2 values observed in Tables 2 and 3.

Table 4: Chi-squared test

Model	χ^2_{Cal}	χ^2_{Tab}	Remarks
Temkin isotherm	12.7	12.6	$\chi^2_{Cal} > \chi^2_{Tab}$, hence, model not reliable for prediction
Langmuir isotherm	5.2	12.6	$\chi^2_{Cal} < \chi^2_{Tab}$, hence, model reliable for prediction
Freudlich isotherm	60952.4	12.6	$\chi^2_{Cal} >> \chi^2_{Tab}$, hence, model not reliable for prediction
Pseudo-first order model	4.7	12.6	$\chi^2_{Cal} < \chi^2_{Tab}$, hence, model reliable for prediction
Pseudo-second order model	3.2	12.6	$\chi^2_{Cal} < \chi^2_{Tab}$, hence, model reliable for prediction
Intra-particle diffusion model	6674.8	12.6	$\chi^2_{Cal} >> \chi^2_{Tab}$, hence, model not reliable for prediction
Elovich model	149.9	12.6	$\chi^2_{Cal} > \chi^2_{Tab}$, hence, model not reliable for prediction

CONCLUSION

Activated carbons were produced from a local variety of mango seed by carbonizing the endocarp before activation with $ZnCl_2$ using an impregnation ratio of 1:4. Laboratory batch studies were conducted in other to generate data for kinetic and equilibrium modelling of the adsorption of colour by the produced MSEAC in other to determine the mechanism and rate controlling steps of the process. The equilibrium models used included; Langmuir isotherm, Freundlich isotherm and Tempkin isotherm. While the kinetic models used were; first order kinetic, second order kinetic, Intra-particle diffusion, and Elovich models.

The pseudo-second order model gave the highest goodness of fit (R^2) of 0.999 compared to the other kinetic models tested. This implies that chemisorption is the rate controlling step. Langmuir isotherm fit experimental data with high value of goodness of fit (R^2) of 0.998 compared to the other tested isotherm models, which implies that the adsorption process is based on a monolayer adsorption. Chi-squared tests performed on all the models confirm the reliability of the Langmuir and pseudo-second order models over the other isotherm and kinetic models respectively. The results indicate that activated carbon prepared from mango seed endocarp is suitable as adsorbent material for adsorption of colour from textile wastewater. The Langmuir and pseudo-second order kinetic models considered in this study should be used as reliable tools for predicting the adsorption of colour from textile wastewater by the produced MSEAC. Column studies on the produced MSEAC should be carried out to determine the suitability of the MSEAC in column reactors.

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