Methylene Blue Adsorption Kinetics Investigation by Coconut Shell Activated Carbon Adsorbent Using Fractional Power, Avrami and Bangham Models

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Abstract–Methylene blue is not easily biodegradable, and its persistence in water can contribute to long-term contamination if not properly treated. Numerous adsorption kinetic study successes were achieved utilizing activated carbons of coconut shell, especially the Pseudo-First and Pseudo-Second order models with little/no regards to the unique behavior described by the Avrami, Bangham and the Fractional Power kinetic models. Herein, the parameters in those models, such as $k_{AV} \& n_{AV}$; $K_f \& \alpha$ and; k & v, respectively, were determined to describe the sorption mechanism. Avrami model fitting results in $n_{AV} = 0.72537$ (< 1), suggesting a complex adsorption process which does not follow the first-order kinetic; but a k_{AV} of 0.24926 obtained, signifies a positive adsorption rate. A 'v < 1' obtained deviates from the first-order but shows that the Fractional Power model best described the Coconut Shell- Activated Carbon Methylene blue bio-sorption process at constant adsorption capacity of 15937.8 mg/g. Findings show that Bangham kinetic model does not fit the experimental data for MB sorption from aqueous solution. MB exposure issues like gastrointestinal discomfort, skin and eye irritation, and imbalances in the populations of various species in aquatic ecosystems due to impacts on their growth, reproduction and survival, may be solved using a very effective adsorbent, such as the CS-AC used here.

Keywords- Methylene blue dye, Coconut shell, Avrami model, Bangham model, Fractional power kinetic

NOMENCLATURE

FP	Fractional Power.
RSM	Response Surface Methodology.
MB	Methylene Blue.
CS	Coconut Shell.
AC	Activated Carbon.

I. INTRODUCTION

In practice, researchers often choose a kinetic model based on the observed behavior of the bio-sorption process and validate the selection by comparing model predictions to experimental data. Fractional Power (FP), Avrami, and Bangham bio-

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sorption kinetic models are three distinct approaches used to describe the kinetics of bio-sorption processes. Amongst them, a sigmoidal shape, making it suitable for bio-sorption processes with a gradual decrease in the adsorption rate over time, characterizes Bangham model. Avrami model incorporates an exponential term in addition to a power-law term, offering versatility in capturing a variety of bio-sorption mechanisms surface adsorption and diffusion-controlled (including processes) [1], [2]. On the other hand, the FP model uses a power-law relationship with a fractional exponent [3], providing flexibility in describing kinetics with non-integer reaction orders. The appropriateness of a model depends on the specific characteristics of the bio-sorption system under investigation. While isotherm studies focus on equilibrium conditions [4]-[8], describing the distribution of sorbate between the bio-sorbent and the solution at a specific time, kinetic studies visualize the rate at which sorption occurs over time. Kinetic studies are often conducted first to understand the fundamental behavior of the bio-sorption process. Once key parameters are identified, response surface methodology (RSM) can be employed for systematic optimization [9], [10]. In some cases, kinetic information can be integrated into the optimization process. For example, kinetic constants derived from experimental data can be used as input parameters in the optimization model. Now, majority of the existing kinetics of methylene blue (MB) removal from wastewater using coconut shell (CS) activated carbon (AC) as adsorbent, as an example, had already been studied. Backed only with an isotherm study, Olafadehan et al. (2012) demonstrated the effectiveness of CS-AC in treating brewery wastewater.

CS-AC is a low-cost adsorbent for wastewater purification [12]–[15], also studied in a fixed bed column [16]–[18] and sometimes, a two-stage adsorber (Wong et al., 2022). MB, a synthetic dye with a distinctive blue color, is used in various

industrial applications, including textile dyeing [20]-[22], laboratory testing, and as a stain in medical and biological Table 1: Bio-sorption Process Conditions procedures. While it serves its intended purposes, the presence of MB in water can be a cause for concern due to several reasons, including its toxicity, health risk and the loss of water aesthetic quality. Adsorption [23], [24], chemical precipitation, advanced oxidation processes and biological treatment are major MB removal techniques. Thus, variants of the coconut parts or its derivative have also been applied in several sorption processes involving CS. As such variants or derivative, one has shell [25], charcoal [26]-[27], choir [28]-[31], fiber (Al-Aoh et al., 2016; Zhang et al., 2018), AC [34]-[35], dreg [36], leaves [37]–[39], tree bark [40], spent grated coconut [41], coating/modifications [42]–[45], hydrochar [46], biochar [47] and additives [48]- [49]. In addition, the removal of other dye types from water had been examined using CS [50], [51].

Study conducted by Shah & Parmar (2018) show that AC derived from CS has 5.5 pH, 2% moisture content, 10.7% ash content, 0.5 g/cm3 bulk density, 1227 mg/g iodine number, 249 mg/g MB number and 936 m²/g specific surface area. Also, optimization of operating variables for the production of AC from CS was investigated by Liang et al. (2020), reporting a lesser iodine value of 698.37 mg/g. None among published articles between 2000-2024 employed the FP, Bangham and the Avrami kinetic model to explain the adsorption mechanism of MB dye by CS-AC. However, Pseudo First order, Pseudo Second order [30], [38], [54]-[56], Weber & Morris intraparticle diffusion [57]-[60], Natarajan & Khalaf, Bhattachara & Venkobachar [61], Bangham [62], [63], Mekay et al. and the liquid film diffusion [62] kinetic had been studied for this particular adsorbent and adsorbate.

This study wishes to estimate the kinetic parameters from the three models described, to study the behavior of the adsorbent at 10, 20, 30, 40, 50 and 60 mins contact time. Previously, Khuluk et al. (2019) carried similar study at 40, 60 and 80 min equilibrium time, but studied the isotherm only. Abdullah et al. (2017), Ismail et al. (2015) and Foo & Hameed (2012) studied the effect of acid and/or base treatment to MB sorption using CS-AC and; Yasin et al. (2007) and Gimba et al. (2000) investigated the adsorption of MB unto potassium hydroxide treated CS-AC and iron and calcium chloride activated powdered CS-AC, respectively.

Thermodynamic studies previously conducted [70], highlights the energy requirements during MB adsorption using variety of CS parts. Though, the present study is only interested in MB removal from aqueous solution, it suffices and will serve as a guide towards carrying out similar studies on plastic industry, tannery, cosmetic industry, photographic, laboratory, printing, paper, pharmaceutical, dye manufacturing and leather industry wastewaters, which may contain the dye. Thus, the order and mechanism of the adsorption process may be understood, contribution of the 3 different kinetic models to the overall adsorption behavior is assessed and process conditions for efficient removal of MB from aqueous solutions can be optimized.

II. METHOD

A. Process Condition

Procedure for the adsorption of MB unto CS-AC were set, as shown in Table 1. Method followed in the preparation of AC from CS in this study, was partly in accordance with Shaheed et al. (2015), Das et al. (2015), Sulyman & Sulyman (2018) and Rangari & Chavan (2017).

Condition	Measurement
CS-AC Dosage	0.2g
Contact Time	10-60 min
Temperature	303 K (30°C)
Initial MB Concentration	100 mg/L
Agitation Speed	250 rpm
Volume of Solution	50 L
AC Particle Size	63-125µm

Activating agent used was sodium hydroxide (NaOH), as described in Mercileen et al. (2023), Cazetta et al. (2011) and Chong & Tam (2020). During MB adsorption by CS-AC, Wong et al. (2013) utilized a particle size of 150 μ m larger than the size employed in this study.

B. Model Fitting

Constant parameters in Bangham model (Equation 1) [63], Avrami (Equations 2 & 3) [78], [79] and the FP kinetic model (Equations 4 & 5) [2] were determined using experimental kinetic variables in Table 2.

$$\log\log\left(\frac{C_i}{C_i - q_t M}\right) = \log\left(\frac{K_f M}{2.303 V}\right) + \alpha \log t \tag{1}$$

$$q_t = q_e [1 - \exp(-k_{AV} t^{n_{AV}})]$$
(2)

$$\ln\left[\ln\left(\frac{q_e}{q_e-q_t}\right)\right] = n_{AV}\ln k_{AV} + n_{AV}\ln t \tag{3}$$

$$q_t = kt^{\nu} \tag{4}$$

$$\log q_t = \log k + v \log t \tag{5}$$

Where, C_i = initial concentration of dye solution (mg/L), V = volume of dye solution (L), M = mass of CS adsorbent (g), q_t = quantity of dye adsorbent at time, t (mg/g), α (< 1) and K_f = constants obtainable from slope and intercept of Equation 1, respectively. k_{AV} = Avrami adsorption kinetic constant $(min^{-n_{AV}})$, n_{AV} = Avrami model exponent of time related to the change in mechanism of adsorbent. k = FP rate constant, and v = constant that is usually less than unity if adsorption kinetic data fits well into power function model. Unknown parameters were determined by plotting relevant variable/term data in Table 2 based on the linearized form of the Bangham (Equation 1), Avrami (Equation 3) and the FP (or Equation 5) model. However, before any other step, the maximum adsorption capacity of CS-AC for MB, $q_e \pmod{g}$ was determined by running a user-defined regression analysis in Origin Pro 2018 using Equation 2 (a nonlinear model). Initial guesses for q_e , k_{AV} and n_{AV} was made to allow the statistical analysis software estimate their new set of value based on good fit or high coefficient of determination (R^2) of the predicted and experimental q_t vs. contact time plot. Linear version of the depiction is re-illustrated using Equation 3 and comparison was made as to the best kinetic parameter estimated between nonlinear method of solution and graphical/linear results. Thus, a plot of $\ln\left[\ln\left(\frac{q_e}{q_e-q_t}\right)\right]$ againt $\ln t$ was carried out,

taken/using q_e value obtained from regression analysis. Relevant graphical representation was also produced to analyze the Bangham and FP model. Essentially, q_t was calculated using Equation 6 [80]–[83].

$$q_t = \frac{(C_i - C_e)V}{M} \tag{6}$$

Where, M, C_i , C_e and V are as defined earlier.

Table 2: Axis Data for Parameter Determination

t (min)	$C_e \text{ (mg/L)}$	$q_t (\mathrm{mg/g})$	$q_t (mg/g)$	ln t	$\ln\left[\ln\left(\frac{q_e}{q_e-q_e}\right)\right]$	$\left(\frac{1}{1}\right)$	log t
		Expt.	Prdct.		Expt.	Prdct.	
10	68.52	7870	11699.01	2.3025851	-0.38447	0.280967	3.895975
20	41.61	14597.5	14153.55	2.9957323	0.906554	0.783756	4.164278
30	38.3	15425	15093.93	3.4011974	1.234452	1.077868	4.188225
40	38.02	15495	15511.09	3.6888795	1.276271	1.286544	4.190192
50	37.82	15545	15711.93	3.912023	1.309159	1.448405	4.191591
60	36.72	15820	15814.08	4.0943446	1.590695	1.580656	4.199206
t (min)	log t	$q_t imes 10^3$ (mg/	/g)	$\log q_t$	$Log \log \left(\frac{C}{C_i}\right)$	$\left(\frac{z_i}{q_t M}\right)$	
10	1	7.87		3.895975	-2.16177		
20	1.30103	14.5975		4.164279	-1.89049		
30	1.47712	15.425		4.188225	-1.86617		
40	1.60206	15.495		4.190192	-1.86418		
50	1.69897	15.545		4.191591	-1.86275		
60	1.77815	15.82		4.199207	-1.85502		

III. RESULTS AND DISCUSSION

A. Avrami Model Parameters

Figure 1a was obtained as a result of defining the Avrami kinetic model as a user-defined model in Origin Pro by selecting the Orthogonal Distance Regression (Pro) iteration algorithm. For the linearized plot (Figure 1b), q_e must be known and is cautiously taken from nonlinear regression conducted.



Figure 1: Avrami Kinetic Parameter from (a) Nonlinear Regression and (b) Graphical Method

Normally, when $\ln \left[\ln \left(\frac{q_e}{q_e - q_t} \right) \right]$ is plotted against $\ln t$ for the Avrami biosorption kinetic model, a straight line with a slope equal to the Avrami exponent ' n_{AV} ' and a y-intercept equal to $\ln t$ is obtained [84]. Similarly, using the experimental and predicted $\ln \left[\ln \left(\frac{q_e}{q_e - q_t} \right) \right]$ data, new sets of k_{AV} and n_{AV} can be calculated from straight-line equations shown in Figure 1b. The Avrami parameter values for the adsorption of MB by CS-AC adsorbent is shown in Table 3.

Table 3: Avrami MB Adsor	ption Kinetic Parameters
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Parameter	Nonlinear Method	Graphical Method		
		Predicted Data	Experimental	
			Data	
$q_e (\text{mg/g})$	15937.83593	15937.83593	15937.83593	
k_{AV} (*)	0.24926	3964.522	0.089377	
n _{AV}	0.72537	0.0249	1.0046	
R^2	1.0000	0.9363	0.8861	

*Unit depends on the unit of time

As expected, predicted $\ln \left[\ln \left(\frac{q_e}{q_e - q_t} \right) \right]$ values vs. lnt gives an almost perfect fit ($R^2 = 0.9363$) than the experimental data with $R^2 = 0.8861$, for similar actual and predicted $q_e = 15937.84$ mg/g. Often, the graphical method of finding kinetic parameters utilizes experimental x- and y-axis data and not the predicted ones. As such, Avrami parameters obtained following the nonlinear and experimental data are close. Value of k_{AV} = 3964.522 obtained from the use of predicted data graphical method is too high and odd, even though the fit is better. It does not satisfy the corresponding value of $n_{AV} = 0.0249$ obtained, because normally, if $n_{AV} = 0$ or $\cong 0$, the plot of $\ln \left[\ln \left(\frac{q_e}{q_e - q_t} \right) \right]$ against ln t will be a horizontal line with a slope of zero. Therefore, natural logarithm of the ratio $\frac{q_e}{q_e - q_t}$ remains constant with time. This suggests that the biosorption process follows a zero-order reaction, where the rate of the reaction is independent of the concentration of the MB. The adsorption occurs at a constant rate until equilibrium is reached. This observation strikes out estimates form the predicted data obtained graphically, leaving the experimental graphical result and the nonlinear regression estimates. Researchers will find the R 'PUPAK Package' containing model fitting functions for linear and non-linear adsorption kinetic and diffusion models developed by Magalong et al. (2022), a better and easier way of predicting bio-sorption kinetic parameters in the future. Since the Orthogonal Distance Regression algorithm gives a higher R^2 value of unity, implying a better fit compared to the experimental graphical parameters in Table 3, it may be adopted as the most accurate Avrami parameter values for MB CS-AC bio-sorption. If $n_{AV} = 1$ in the Avrami model, the plot of $\ln\left[\ln\left(\frac{q_e}{q_e-q_t}\right)\right]$ against ln t will be a straight line with a slope = k_{AV} , and this suggests first-order kinetics in the Avrami model, almost in agreement with $n_{AV} = 1.0046$ obtained empirically. But $n_{AV} < 1$ for this study, suggests a complex adsorption process that does not strictly follow first-order kinetics, as obtained via regression. When $n_{AV} = 2$, it is a second-order reaction. Rate constant ($k_{AV} = 0.24926 \ min^{-n_{AV}}$) reflects the speed of the MB adsorption process, where a positive value, indicates a positive rate of adsorption. In a study conducted by Kuete et al. (2020), $k_{AV} = 0.213 \ min^{-1}$ closer to this study and $n_{AV} = 0.562$ was realized during thymol blue adsorption by Garcinia cola nut shells.

B. Fractional Power Kinetic Parameters

k and v FP kinetic parameters, as determined using the nonlinear FP model (or Equation 4) and the linearized model (or Equation 5) also explains the behaviour of the CS-AC biosorbent.



Figure 2: FP Model Parameter Determination by (a) Regression and (b) Graphical Method

The fact that 'v' < 1 [86], implies that the adsorption kinetic data determined via the two approaches in Table 4, fits well into the power function model. However, since \mathbb{R}^2 is higher, as observed under the graphical technique (0.7405), the corresponding k and v values at which it occurs is the most accurate prediction.

Table 4: Estimated FP Model Parameters

Parameter	Nonlinear Technique	Graphical Technique
k (mg/g min)	5390.83675	4090.722
v	0.27972	0.3566
\mathbb{R}^2	0.70377	0.7405

Important derivations from this analysis are as follows: (1) the adsorption of MB on CS-AV follows a FP kinetic model, indicating a deviation from classical first-order kinetics, (2) adsorption rate (k) is relatively high, suggesting a rapid adsorption process and (3) the model moderately fits the experimental data (\mathbb{R}^2 values around 0.7). Generally, a value of v between 0 and 1 suggests fractional-order kinetics, as obtained in Kuete et al. (2020) and Mozaffari et al. (2020). The higher the value of v, the more the kinetics deviate from classical first-order kinetics. Here, both techniques provide similar values for v, indicating a fractional-order adsorption process. In addition, a higher k value points to faster adsorption rate. In that case, the following assumptions of the FP model are satisfied:

(1) A homogeneous system where the concentration of MB is uniform throughout the CS-AC.

(2) A single-component adsorption based on the assumption that only MB is involved in the process.

(3) A constant temperature (in this case, 30°C) throughout the bio-sorption process,

(4) No external mass transfer resistance from the bulk solution to the CS-AC surface.

(5) The model assumes well-defined initial conditions (Table 1) for the process.

(6) A negligible effect between MB molecules on the CS-AC surface.

C. Bangham Kinetic Parameters

It is discovered that q_t data in Table 2 are too large to give a meaningful value of $Log \log \left(\frac{C_i}{C_i - q_t M}\right)$. Hence, $q_t \times 10^3$ was the re-scaled value used to estimate the Bangham model kinetic parameters. So, a plot of $Log \log \left(\frac{C_i}{C_i - q_t M}\right)$ against $\log t$ is shown in Figure 3.



Figure 3: Bangham Kinetic Plot for MB Biosorption via CS-AC

Even so, the plot of $Log \log \left(\frac{C_i}{C_i - q_t M}\right)$ against $\log t$ is not linear as expected, showing that the Bangham model is not an appropriate description of the adsorption kinetics in this system. Kavitha & Namasivayam (2007) obtained a perfect linear plot at 35°C using coconut coir as adsorbent to remove MB, as also realized in Inyinbor et al. (2016) at $\mathbb{R}^2 > 0.9$ using a different adsorbate-adsorbent system. The model parameters in question (Table 5) were then computed from slopes and intercepts of Equation 1 plotted.

Table 5: Bangham Kinetic Parameters for MB Sorption using CS

Parameter	Value
K _f	2.045662
α	0.3608
\mathbb{R}^2	0.7408

Rate constant, K_f associated with the Bangham kinetic model is dependent on the order of the reaction while α represent the fractional attainment of equilibrium or the proportionality between the rate of adsorption and the driving force. Specific range of K_f values considered "high" or "low" is contextdependent and can vary for different adsorbate-adsorbent systems. Researchers may compare K_f values with those obtained for other adsorbate-adsorbent systems or under different conditions to assess relative adsorption rates; such as in Kavitha & Namasivayam (2007). Prajapati & Mondal (2020) obtained $K_f = 2.35$ and $\alpha = 0.74$ at 100 mg/L MB C_i sorbed by modified CS. An $\alpha = 0$ can be interpreted as the initial stage of adsorption where the process has not yet reached equilibrium. Values within the range: $0 < \alpha < 1$ may suggest that the adsorption process is reaching equilibrium, and the dimensionless $\alpha = 0.3608$ represents the fraction of equilibrium attained. And lastly, $\alpha = 1$ indicate complete attainment of equilibrium. R² measures how well the experimental data fits the Bangham kinetic model and an R² of 0.7408 infer a reasonable fit. Overall, the occurrence of undefined logarithmic terms can suggest that the model fit may not be appropriate for the entire adsorption process and the $q_t \times 10^3$ values resulting in Figure 3 are just exploratory. That is, the Bangham [10] assumption that the rate of adsorption is directly proportional to the number of available adsorption sites on the adsorbent material or first-order kinetics is not satisfied. Other [11] assumptions similar with '1' and '3' in FP model is also in agreement. In addition, the model assumes that the rate of adsorption remains constant throughout the adsorption process. This is a simplification, and in reality, the adsorption rate might change as the CS-AC bio-sorbent becomes saturated.

IV. CONCLUSION

Moderate success had been achieved in finding the best kinetic model amongst Avrami, FP and Bangham models, that describes the adsorption of MB from aqueous solution using CS-AC. For thorough and accurate representation of the reaction kinetics, reaction order from Bangham (α) and Avrami model (n_{AV}) were determined in the process via nonlinear regression or graphical method, as appropriate. It was discovered that very large q_t obtained in this study is not supported by the Bangham kinetic model and when it was rescaled for ease of interpretation, an α value of 0.3608 described MB CS-AC system as an adsorption process approaching equilibrium. In this study, FP best describe the sorption of MB, favored by k = 4090.722 mg/g min, v = 0.3566 and $\mathbb{R}^2 =$ 0.7405. The Avrami nonlinear parameter estimate also suggest that the empirical data fits the model prediction. The analysis result almost abides by the assumptions of both FP and Avrami models. To know whether the bio-sorption is pore diffusion dependent at the particle size range (63-125 μ m) exploited, the intra particle diffusion kinetic model may be used. Elovich, Boyd and the general order biosorption kinetics is still not investigated for MB removal using CS adsorbent.

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