

## Adsorption Of Recalcitrant Basic Blue 3 Dye Using Coffee Husks Powder: Equilibrium And Kinetics Studies

Wycliffe C. Wanyonyi, Gilbert K. Cheruiyot, Kiplimo J. Joyce

Department Of Mathematics, Actuarial And Physical Sciences, University Of Kabianga, P.O. Box 2030, 20200 Kericho, Kenya

### Abstract

Coffee Husks Powder (CHP) was evaluated for potential application in adsorptive removal of recalcitrant Basic Blue 3 (BB3) dye from aqueous solution. Basic Blue 3 dye is among extensively used colorant in textile industries to impart color while posing serious ecological challenges and health related risks. Batch adsorption experiments were designed ascertain the influence of pH, dye concentration, adsorbent dose, and contact time in dye removal by CHP. Dye removal per unit mass of CHP increased with increase in contact time, dye concentration, fine particle size and low adsorbent dose. Equilibrium results showed that at least 96% of the 8.6374 mg/l dye could be removed within 10 minutes by a mass of 0.5g at optimum pH of 6. At room temperature, both Langmuir and Freundlich isotherms were found applicable in all concentrations studied indicating physisorption and chemisorption. Kinetic behavior of adsorption was better described by Pseudo-Second order implying chemisorption nature of the process. Coffee husks powder displayed great potential in adsorptive removal of BB3 dye in aqueous solutions and proves useful in curbing pollutions of colored wastewaters.

**Keywords:** Basic Blue 3 Dye, Coffee Husks Powder, adsorption, kinetics, equilibrium

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### I. Introduction

Synthetic dyes are largely employed to colour products in industries such as pharmaceuticals, cosmetics, textiles, construction, ink, plastic, food, leather, rubber, paper etc. It is estimated that over  $7 \times 10^5$  tons of over 10,000 different dyes and pigments are produced globally and its market is skyrocketing each year exceeded over US \$11 billion by 2008 [1], [2], [3]. Textile industry for example consumes huge amount of dye and approximately 1-2% of dyes stuffs are lost during production. Additionally, about 10-15% of dyes are discharge as effluent during dye applications [2], [4]. In aquatic environments, wastewaters containing dye pollutants are tough to remove and pose grave dangers to aquatic life, humans and world's economy which largely dependent on dwindling water resource. Dyes are toxic, carcinogenic and mutagenic which makes them unsafe to flora and fauna. Moreover, the presence of the dyes in water even at low concentrations hinder photosynthetic activity by preventing light penetration thereby worsening the quality of water, limiting gas solubility and destroying aesthetic value of water bodies [5]. Despite this negative health and environmental effect, organic dyes such as BB3 are increasingly being used in industries without proper wastewater treatment.

Basic blue 3 (BB3) ( $C_{20}H_{26}ClN_3O$ ) IUPAC name: 3,7-bis(diethylamino)phenoxazin-5-ium chloride; Molecular mass of 359.89 g/mol; molecular structure Fig. 1) is an oxazine cationic dye majorly employed in the textile industries for direct printing crylic carpet, dying wool, acrylic blended fabric and silk [6], [7]. Disposal of industrial effluents containing BB3 dye causes life threatening effects since it is highly toxic and hazardous to aquatic life [7]. Furthermore, direct exposure causes skin and eye irritation/damage, acute toxicity upon inhalation, gut irritations, genetic mutations and cancer [8]. Treatment of wastewaters containing BB3 dye before disposal is therefore critical.

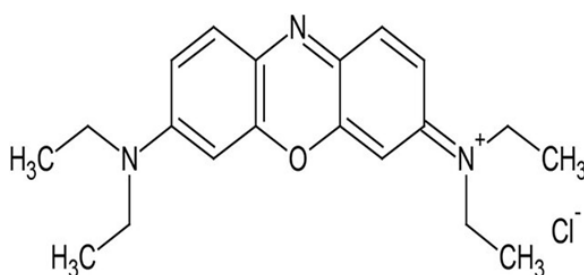


Fig 1. Chemical structure of Basic Blue 3 dye.

Different techniques have been employed in removal of dyes from aqueous solutions including physical, chemical and biological treatment with fungi, algae or activated sludge. However, some of these methods experience a number of limitation including costly, poor performance, long period of processing and production of high sludge quantity [9]. Adsorption has been applauded as a better method for wastewater treatment owing to its, uncomplicatedness, ease of use, low cost, highly efficient, less operation time and high prospect of dye and adsorbent recovery [10]. Activated carbon has for long time been used as the most efficient adsorbent for several industries but its exorbitant cost and loss of huge amount during regeneration has limited its usage [11]. This has stimulated many scholars to search for freely available, effective and biodegradable adsorbents such as *Eichhornia crassipes* roots, macadamia seed husks, rambutan (*Nephelium lappaceum*) peel, tea waste, wheat husk, garlic peel, fig leaf, grass waste, Pomegranate Fruit Peel etc [7], [10], [12], [13], [14], [15], [16].

Coffee husks are the major by-product obtained from a coffee scrub, a cash crop that is cultivated in over 80 countries across the world. During processing of coffee beans, huge amounts of coffee husks are generated and discarded polluting the environment. Efforts to find an alternate method to utilize Coffee husk in solving environmental pollution challenge is limited. This study therefore explores the efficacy Coffee husk in adsorptive removal of BB3 dye. They are locally available, cheaper, non-toxic which are massively generated as by-products in numerous coffee mills globally.

## II. Materials And Methods

### Materials

Fresh coffee husks were sourced from Roret coffee factory in Kericho County, Kenya. The biomass were washed, rinsed and dried in oven set 60° C for 24 hours before crushing using Lab Ball mill -Lasany; S/No: 20160301201. Resultant powder was sieved through different sieves and stored in plastic container with a tight lid for subsequently used in batch sorption experiments. Basic blue 3 dye (analytical grade) was obtained from Ranbaxy Fine Chemicals Ltd, Kenya. 0.0025 g of the dye powder was dissolved in a 250 ml volumetric flask containing double distilled water to yield a stock solution of 10 mg/l. Various dilutions were made from the prepared stock solution for adsorption experiments.

### Batch adsorption experiments

Batch adsorption experiments were performed in triplicate in a 250 mL Erlenmeyer flask placed on an orbital shaker (Thermolyne-type 65800) set at optimal shaking rate of 140 rpm [10]. Except for dosage studies, adsorbent mass of 0.5g in 50 ml dye volume was used at 25°C for 120 minutes on a shaker at 140 rpm. A working solution of 8.6374 mg/l BB3 dye solution was always used and absorbance measurements were taken at every 10 minutes intervals on Hallo-Rb- 10 UV/Vis spectrometer at pre-scanned wavelength ( $\lambda_{\max}$ ) of 654 nm. Dye concentrations ranged from 0.8637 mg/l to 8.6374 mg/l was used to investigate effect of dye initial concentration. To study effect of dose on adsorption; adsorbent mass of 0.1, 0.25, 0.5, 0.75, 1.0, 1.25 and 1.5 g were used. Different particle sizes of 0.15 to 0.3 mm, 0.3 to 0.6 mm, 0.6 to 1.18 mm, 1.18 to 2.36 mm, 2.36 to 4.75mm were also evaluated for their dye uptake efficiencies. pH adjustments from pH 2 to pH 12 of the adsorbate solution were achieved while 0.1M NaOH and 0.1M HCl was used to adjust pH as required. Effect of temperature was investigated at a temperature range of 25° C to 85° C on Grant JB2 water bath (S/No: A 17379). After every 10 minutes, aliquots were drawn from the mother solution filtered into rectangular cuvettes and the absorbance measurements taken at  $\lambda_{\max}$  654 against a blank solution using (HALO RB- 10 UV-Vis) spectrophotometer. The amount of dye adsorbed per unit mass of the adsorbent  $q_e$ (mg g<sup>-1</sup>) and the extent of adsorption (%) was calculated using equ.1.

$$q_e = \frac{V(C_o - C_e)}{W} \quad (1)$$

Where  $C_o$  and  $C_e$  are the initial and the equilibrium concentrations (mg L<sup>-1</sup>) of BB3 dye, V is the volume of solution (L) and W is the amount of CHP used (g). Percentage dye removal (%) was calculated using equ.2.

$$\% \text{ Removed} = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

Adsorption kinetics was determined using different initial BB3 dye concentrations at 25°C by altering the contact time. 0.5 g of CHP of particle size 0.15 - 0.3 mm were mixed with 50 mL BB3 dye in a 250 mL conical flask agitation at 140 rpm. After 10 min time interval, 3 mL were withdrawn, from the conical flask, residual BB3 dye concentration determined and then returned back in the conical flask to restore the initial volume.

## III. Results And Discussion

### Effect of Contact Time

Contact time regulates equilibrium kinetics, underscores the stability of the sorption process and provides an outlook of the general cost considered when designing the sorption system for upscaling wastewater treatment [17], [18], [19]. Contact time influence on BB3 dye sorption was performed at 25°C and the findings displayed in Fig 2. There was a significant steady rise in dye removal within the first 10 minutes. CHP proved efficient in

adsorbing up to 95.27% of BB3 at the equilibrium. Initial high adsorption rate was due to vacant active surface sites available for occupation by adsorbate molecules but as time elapsed beyond 10 minutes dye removal sharply slowed down since the system had attained equilibrium and due to exhaustion of the adsorptive sites [20].

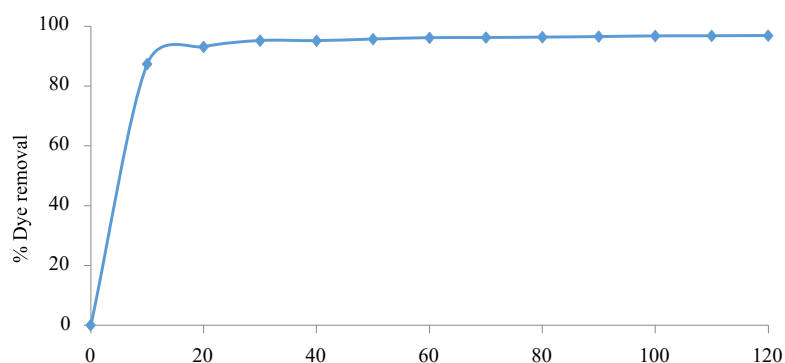


Fig 2. Effect of contact time on BB3 adsorption of CHP (Dose: 0.5 g, 8.6374 mg/l, 50 ml)

### Effect of BB3 Initial Dye concentration

The initial dye concentration effect on adsorption BB3 dye is shown in Fig. 3. The result showed that the amount of dye adsorbed rose when dye concentration increased at all tested concentrations. This can be accredited to an increasing concentration gradient acting as an increasing driving force to overcome all mass transfer resistances of the dye molecules between the aqueous and solid phase, leading to a rising equilibrium adsorption until saturation is achieved [3]. Similarly, adsorption process is dependable on the concentration of the dyes, at very low BB3 dye concentration, fractional adsorption is low and the adsorption process is not dependent on the initial dye concentration. It was further noted that faster saturation of active adsorption sites on the surface of CHP when high dye concentration is used could rationalize this observation. Similar findings have been observed in adsorption of crystal violet dye from aqueous solution using *Rhizophora mucronata* stem-barks [18]:

### Effect of Particle size on BB3 adsorption

Dye uptake from aqueous solution is directly influenced by the available active surface area on the adsorbent. The influence of particle size on adsorptive removal of BB3 dye was investigated using initial dye concentration of 8.6374 mg/l at different particle sizes of the CHP and the results displayed in Fig.4. The result reveal that smaller particle sizes (0.15 – 0.3 mm) proved to be more effective with over 97.6% dye removal. This could be attributed to smaller particle sizes that afford a large surface area which results in an increased dye uptake. This findings corresponds with similar results obtained in the adsorption of crystal violet dye on coffee husks [21].

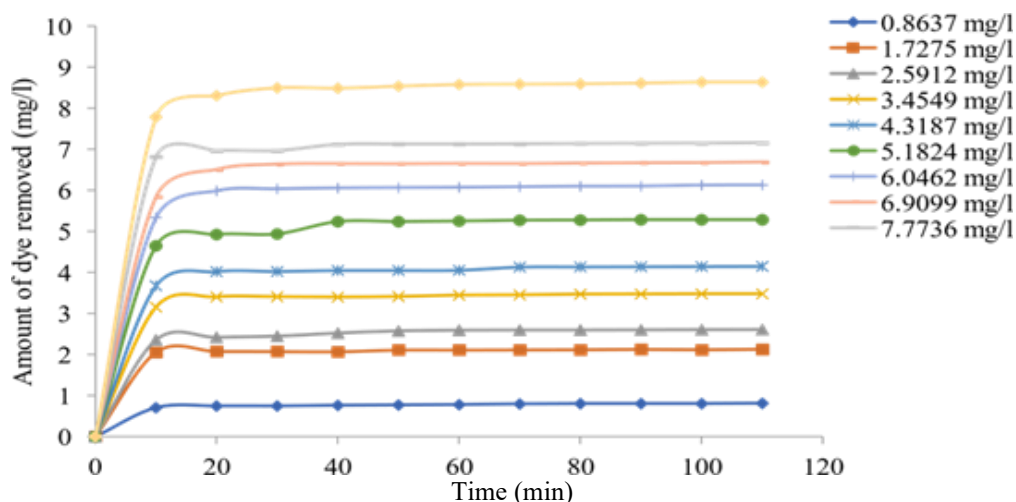


Fig 3. influence of initial concentration on BB3 dye uptake amounts (CHP: 0.5 g; dye conc. range: 0.8637-8.6374 mg/l, 50 ml; temp: 25 °C)

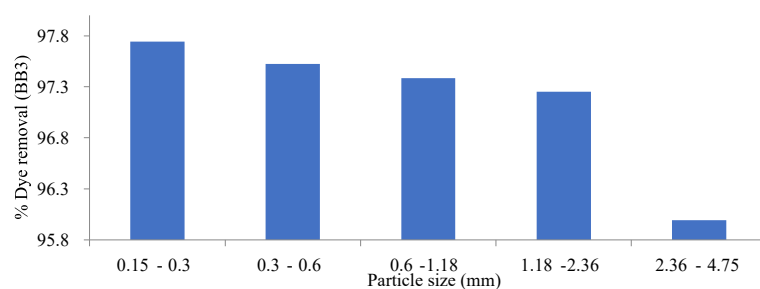


Fig 4. Effect of particle size on adsorption of BB3 Dye. (CHP: 0.5 g; dye: 8.6374 mg/l, 50 ml; temp: 25 °C)

### Effect of pH on adsorption of BB3 Dye

pH plays a perilous role in regulating the charge on adsorbent surface, the magnitude of adsorbate ionization and dissociation of functional groups on the adsorbent [22]. Effect of pH on adsorption BB3 dye was investigated at a pH range of between pH 2 to pH 12 and results presented in Fig 5. CHP exhibited maximum adsorption efficiency at pH 7 where 0.908 mg/g. Initially dye uptake was lower from pH2 but progressively improved upon pH adjustment towards value of 7 then slowed down. At pH value of 2, active adsorbent's active groups get protonated and repulsed cationic BB3 molecules. However, as the pH of solution tend to be neutral (pH 7) these surface groups revert back to their initial state and remain active in adsorption. Above this rendered the adsorbent deprotonated and adsorbent-adsorbate interaction become low.

### Effect of Temperature on adsorption of BB3 Dye

Temperature performs a central role in adsorption since it lessens the viscosity of the solution and increases the speediness of adsorbate diffusion over the exterior layer and interior pores of the adsorbent material [23]. Findings revealed gentle increase amounts in mg/g of BB3 dye adsorbed when temperature of the solution was elevated from 25°C to 85 °C as displayed in Fig. 6. This relates to increased mobility and subsequent dilution of dye ions onto particle's binding group. Additionally, this can be attributed to stronger intermolecular forces between the active binding spaces on the CHP surface and the BB3 dye particles than those between the solvent and dye particles [7].

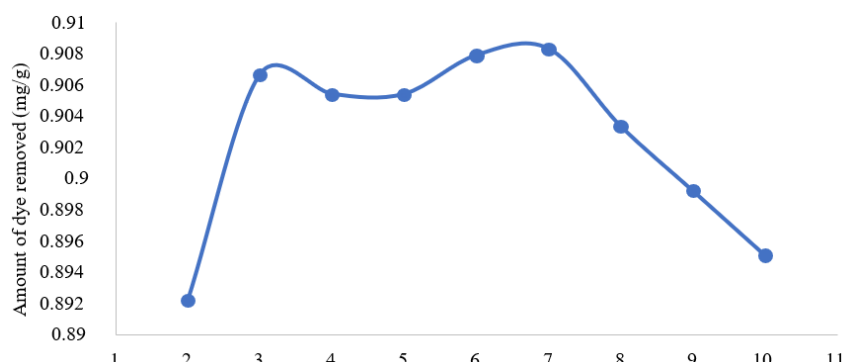


Fig 5. BB3 dye removal rate dependence on pH (CHP: 0.5 g; 8.6374 mg/l, 50 ml; temp: 25 °C)

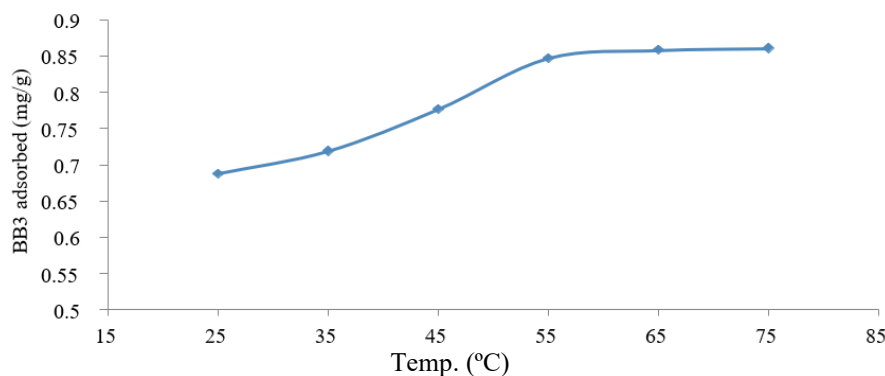


Fig.6. Effect of temperature on adsorption of BB3 (CHP: 0.5 g; 8.6374 mg/l, 50 ml; particle size: 0.15-0.3 mm)

## Kinetic Studies

The experimental adsorption kinetic data were modeled using pseudo-first order [24] and pseudo-second order [25]. Pseudo first and second models are broadly applied in adsorption processes to determine controlling mechanism of adsorption process such as adsorption surface, chemical reaction and/or diffusion mechanisms [26]. The pseudo-first order kinetic model can be presented by equ.3.

$$\text{Log}(q_e - q_t) = \text{log} q_e - \frac{tk_1}{2.303} \quad (3)$$

Where  $k_1$  is the rate constant of pseudo-first-order adsorption,  $q_e$  and  $q_t$  are the amounts of BB3 dye adsorbed by CHP at equilibrium and at time,  $t$ , respectively. Plot of  $\log(q_e - q_t)$  against time ( $t$ ) resulted in curves with lower correlation coefficients ( $R^2 < 0.70$ ). Pseudo-first-order mechanism was found unsuitable for describing sorption of BB3 since the calculated values for  $q_e$  from the graph did not match with the experimental values. Similarly, experimental data were subjected to pseudo-second-order kinetic model to assess sorption kinetics of BB3 dye on CHP. The pseudo second-order model is given by the equ 4.

$$\frac{t}{q_t} = \frac{1}{k_2} \cdot \frac{1}{q_e^2} + \frac{t}{q_e} \quad (4)$$

Where  $k_2$  is the rate constant for pseudo-second-order adsorption ( $\text{gmg}^{-1}\text{min}^{-1}$ ),  $q_e$  and  $q_t$  are the amount of BB3 dye adsorbed ( $\text{mg g}^{-1}$ ) at equilibrium and at time  $t$  (min) respectively. The plot of  $t/q_t$  vs  $t$  of eqn.4 gives a linear line, from which,  $q_e$  and  $k_2$  can be calculated from the gradient and intercept of the plot. Fig. 7 present pseudo second - order kinetics graph for the adsorption of BB3 dye by CHP. The experimental data had a good fit to Pseudo-second-order kinetics model with higher correlation coefficient values ( $R^2 > 0.99$ ). The rate constants, anticipated equilibrium uptakes and the corresponding correlation coefficients for the tested concentrations were determined and presented in Table 1. It is evident that the calculated and experimental values of  $q_e$  ( $\text{mg/g}$ ) were comparable. The findings suggest that the overall rate adsorption process is controlled by chemisorption process involving valence forces through sharing or exchange of electrons between dye and adsorbent [22].

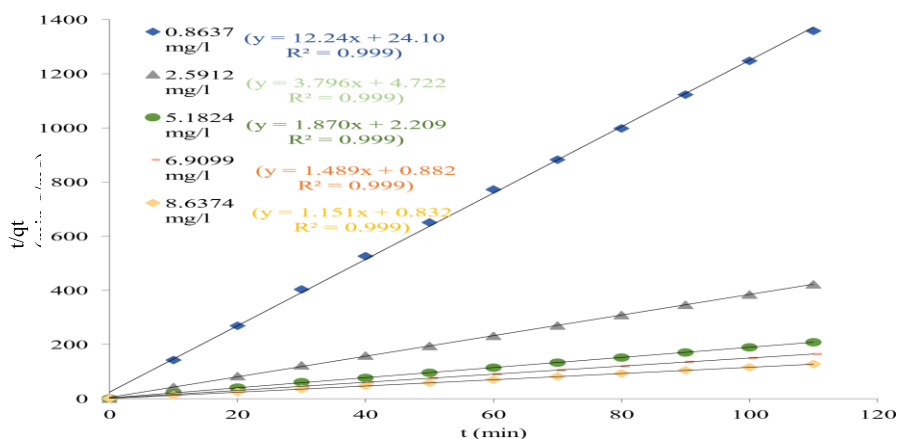


Fig. 7: Pseudo-second order kinetics plot for the adsorption of BB3 dye on CHP

Table 1: Pseudo- second order parameters for BB3 on CHP

Dye	Concentration (mg/l)	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	$k_2$ (g/mg.min)	$R^2$
BB3	0.8637	0.081	0.082	0.001	0.999
	2.5912	0.261	0.263	0.045	0.999
	5.1827	0.529	0.535	0.205	0.999
	6.9099	0.669	0.672	1.286	0.999
	8.6374	0.864	0.869	1.445	0.999

## Equilibrium Studies

Experimental adsorption data were subjected to Langmuir [27] and the Freundlich [28] adsorption isotherms to evaluate adsorption of BB3 dye on CHP. Langmuir isotherm model deduces that the adsorptions occur at specific homogeneous sites on the adsorbent and is used successfully in many monolayer adsorption processes. The linearized forms of Langmuir isotherm is given by equ.5.

$$\frac{1}{q_e} = \frac{1}{bC_e q_0} + \frac{1}{q_0} \quad (5)$$

Where  $C_e$  ( $\text{mg/l}$ ) is the dye concentration at equilibrium,  $q_e$  ( $\text{mg/g}$ ) is the amount of dye adsorbed per unit mass of adsorbent at equilibrium time,  $q_0$  ( $\text{mg/g}$ ) and  $b$  ( $\text{l/mg}$ ) are the Langmuir constants related to maximum monolayer adsorption capacity and energy of adsorption that is affinity of binding sites respectively. These values are determined from the slope and intercept, respectively, of the plots of  $1/q_e$  versus  $1/C_e$ . Further, separation factor which is defined below was also applied to get insights of the isotherm.

$$R_L = \frac{1}{1+bC_0} \quad (6)$$

Where  $C_0$  (mg/l) is the initial dye concentration and  $b$  (l/mg) is the Langmuir constant. Four  $R_L$  scenarios are possible for any adsorption:  $R_L > 1$  (unfavorable),  $R_L = 1$  (linear),  $R_L = 0$  (irreversible) and  $0 < R_L < 1$  (favorable) [29]. Fig. 8 represent a plot of  $1/q_e$  verses  $1/C_e$  for the adsorption of BB3 onto CHP and the correspondent Langmuir isotherm constants are tabulated in Table 2. The experimental data showed a good fit to Langmuir model with high correlation coefficient ( $R^2 = 0.999$ ) indicating that the model was appropriate to describe the adsorption process of BB3 onto CHP. Langmuir constant related with adsorption energy of the system was at minimal suggesting spontaneous nature of the process while linearity was confirmed by separation factor  $R_L = 1$ .

Similarly, experimental adsorption data were subjected to Freundlich isotherm model. The model assumes multilayer adsorptions on heterogeneous adsorbent surface. The linearized form of the equation is given by equ.7:

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \quad (7)$$

Where  $q_e$  (mg/g) is the amount sorbate per unit weight of adsorbent,  $C_e$  (mg/l) is dye equilibrium concentration,  $k_f$  (l/g) is Freundlich constant, representing the adsorption capacity and  $n$  is the Freundlich exponent that depicts the adsorption intensity (dimensionless). The magnitude of the exponent,  $1/n$ , gives an indication of the favorability of adsorption. Values of  $n > 1$  represent favourable adsorption conditions [29].  $k_f$  and  $n$  values are tabulated from the intercept and gradient of the plot of  $\ln q_e$  against  $\ln C_e$ . Fig. 9 shows a plot of  $\ln q_e$  verses  $\ln C_e$  for the adsorption of BB3 dye onto CHP and the corresponding Freundlich isotherm constants tabulated in Table 2. The results suggest that BB3 dye was favourably adsorbed by CHP with high values of the correlation coefficient ( $R^2 = 0.998$ ) indicate that the Freundlich isotherm best suited for the adsorption of BB3 dye on CHP. It is noteworthy to point out that experimental data fitted well both Langmuir and the Freundlich adsorption isotherms models indicating that both models are suitable for describing the adsorption equilibrium of both the dyes in the studied concentration ranges at room temperature (25 °C). The results indicate a phenomenon of single and multilayered sorption of BB3 dye on CHP.

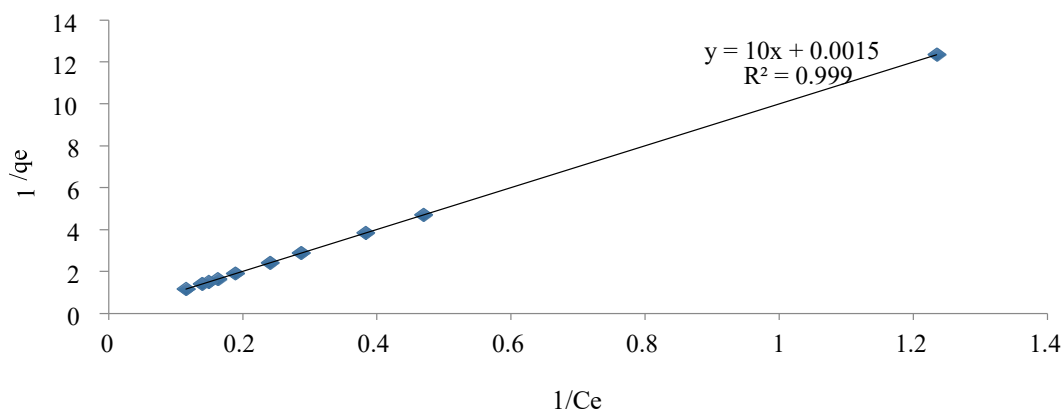


Fig 8. Langmuir Isotherm plot for BB3 dye adsorption

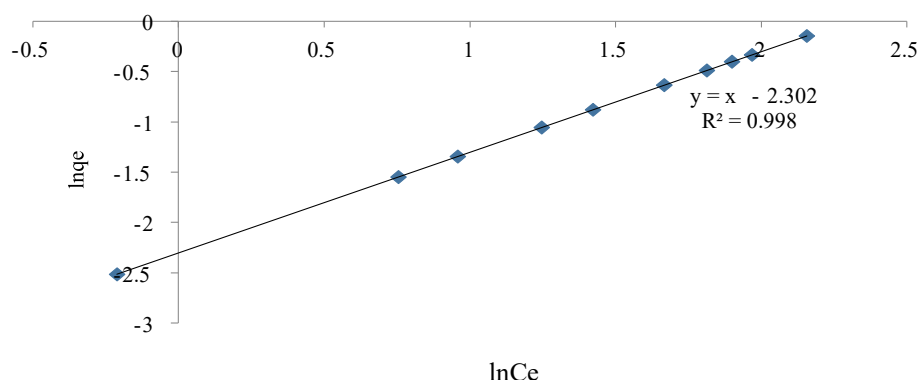


Fig 9. Freundlich Isotherm plot for BB3 adsorption

Table.2. Langmuir and Freundlich isotherm constants for adsorption of BB3 dye on CHP

Langmuir adsorption isotherm			Freundlich adsorption isotherm			
$q_0$	$b$	$R^2$	$R_L$	$K_F$	$n$	$R^2$
0	0.0002	0.999	0.999	9.994	1	0.998

#### IV. Conclusion

This study revealed that CHP is indeed feasible, inexpensive adsorbent for the adsorption of BB3 dye wastewater with over 97% dye removal at 25 °C. Batch studies consideration of operating conditions including contact time, biosorbent dose, particle size, initial concentration, pH and temperature. The results revealed that smallest particle sizes, highest dosages and highest initial concentrations provided better condition for maximum adsorption. Langmuir and Freundlich isotherm models were found to better explain adsorption of BB3 dye on CHP indicating both monolayer and multilayer adsorption process. Further information derived from kinetic studies revealed pseudo-second order kinetic model was in better agreement compared to pseudo-first order kinetic model. Chemisorption therefore dominated the adsorption although physisorption through van der-Waals was also a possible mechanism of adsorption. These findings demonstrated that CHP is a suitable and effective adsorbent for removing BB3 from wastewater. The fact that it is cheap and abundantly in from coffee industry give it advantage as an inexpensive adsorbent.

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