



ISOTHERMAL EQUILIBRIUM AND KINETIC STUDIES OF THE USE OF *MANGIFERA INDICA* AND *PHOENIX DACTYLIFERA* SEEDS POWDERS IN WASTEWATER TREATMENT

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ABSTRACT

This research was aimed at studying the possibility of combining Mangifera indica and Phoenix dactylifera seeds powders as coagulants in wastewater treatment. To achieve this, the seeds were cleaned, dried, husk removed and then crushed. The oil was extracted from the crushed seeds using solvent extraction, with the dried cake used as coagulant in treating the wastewater. Initial parameters of the wastewater including BOD and COD were measured before setting out to study the removal of BOD by combining Mangifera indica (MI) and Phoenix dactylifera (PD) seeds powders over concentration ranges of 5-25 mg/l. The experimental data over the studied concentrations ranges of 5-25 mg/l was processed using the Langmuir and Freundlich isotherm models. The Pseudo-first and second order kinetic models were studied from the plots of time versus amount (mg/l) of BOD removed using MI, PD and the combination of the two. The experiment was observed to fit the Freundlich isotherm model based on the Freundlich constants and the coefficient of determination, R^2 . The experiment was observed to have followed the pseudo-second order kinetic model as indicated by the K , R^2 , and Error analyses carried out on the experimental data. In conclusion, it was shown that Mangifera indica was more effective than Phoenix dactylifera for the removal of BOD.

Key words: Mangifera indica, Phoenix dactylifera, Kinetics, Isothermal

INTRODUCTION

Water demand constantly exceeds supply in most parts of the world, and many more areas are expected to experience this imbalance in the near

future. This is partly due to the fact that only 2.5% of the global water is fresh water. Closely following this, is the ever-increasing world population which depletes many major aquifers besides the impact of climate change on these aquifers (Turrall et al., 2011). Water resources are increasingly becoming scarce necessitating the needs for water conservation and water management. At the heart of this, is the ability to utilize water and wastewater effectively. To achieve this goal, wastewater must be treated in an economic manner (Raman and Krishnamoorthy, 2014). The use of chemicals in the coagulation process of water treatment comes with several challenges including cost of chemicals, increase in volume of sludge, depression of the pH of the effluent and potential health implications such as Parkinson and Alzheimer. Bio coagulants have been known to have some advantages over the conventional chemicals such as non-toxicity, bio degradability, low sludge formation, and overall cost reduction in water treatment facilities.

Biochemical Oxygen Demand (BOD) is an important water quality parameter as it gives an indication of amount of organic compounds in water which is commonly used to characterize wastewater (Ajayi et al., 2016; Abdalla and Hamman, 2014). Various studies have been carried out on the use of plant based- coagulants to remove BOD of wastewater (Mutua et al. 2016; Dange and Lad, 2015; Saha and Saha, 2013; Asgari et al. 2013).

Isothermal equilibrium and kinetic studies have

also been carried out by different researchers utilizing bio coagulants in the removal of water contaminants, (Desta, 2013; Volesky, 2001; Aksu, 2002; Adie et al, 2012; Mahiya et al.,2016; Neto et al, 2013; Vijayakumar et al.,2011). The search is still on for a more efficient bio coagulant which can be used for the removal of BOD and treatment of water and wastewater to help in water conservation.

The objective of the research

This study was carried out to investigate the efficiency of combining *Mangifera indica* (Mango) and *Phoenix dactylifera* (Dates) seeds powders as coagulants for the removal of some wastewater quality parameters which include BOD and COD and to study the isothermal and kinetic behaviors of the treatment process.

MATERIALS AND METHODS

Materials

The following materials were used for the experiment. Jar test flocculator (Peterson Candy), Soxhlet apparatus, pH meter (CRISON micro pH 2000), Muffle furnace (S30 2AU), weighing balance (Mettler H31), BOD Bottle, Mortar and Piston, Filter Paper, Stop watch, Magnetic Stirrer and Magnet, Oven. Others are burette, conical flask, pipette, funnel, spatula, Flat bottom flask.

Methods

A. *Mangifera Indica* and *Phoenix Dactylifera* Seeds Preparation

The seeds (Mango and Dates) were sourced locally from Sabon Tasha and Kawo markets in Chikun and Kaduna North local government areas in Kaduna state. They were washed, cleaned and dried under the sun for fourteen days until they were completely dried. The dry clean seeds were then crushed after the removal of the seed coating and separation of the seeds from chaff to reduce their particle sizes to between 2 mm – 70 μ m. Solvent extraction was used to separate oil from the seeds. 200 g of the pre-processed seeds was treated in a multistage counter current process with hexane (500 ml) as the solvent in Soxhlet extractor until the oil

content was reduced to the lowest possible level. The mixture of oil and solvent were then separated by distillation with the cake washed with distilled water, dried in an oven to constant weight. It was then sieved and used as the coagulants.

B. Jar Test

The Wastewater was sampled from the influent of the Ahmadu Bello University Zaria wastewater stabilization pond and its water quality parameters which included BOD and COD were measured, then jar test was carried out to determine the effect of graduated dosages on the measured physicochemical and bacteriological parameters of the wastewater. Jar test was carried out with rapid mixing at 100 rpm for 1 minute and slow mixing at 30 rpm for 30 minutes. Residual turbidity for different combinations of coagulant dosages were then measured in the interval of 60, 120 and 720 minutes. The BOD of the supernatant liquid from the Jar test was taken at different time intervals of 20, 40, 60, 80, 100, and 120 hours. Linear plots of the different equilibrium concentrations, amount of BOD adsorbed at equilibrium and time were used to process and study the isothermal and kinetic processes of the experiment.

C. Isothermal Studies

The Langmuir and Freundlich isotherms were used to study the distribution of metal ions between the liquid phase and the solid phase. The study was carried out at an average constant temperature of 28 °C and a pH of 6.9.

Langmuir and Freundlich Isotherms- The experimental data for the reduction in BOD by dosage with *Mangifera Indica*, *Phoenix dactylifera*, and their combination over the studied concentration range of 5-25 mg/L were processed using the Langmuir and Freundlich adsorption Isotherm models. The adsorption data were represented by these adsorption isotherms. Linear plots were employed to determine the values of Langmuir and

Freundlich constants and a deduction was made base on these constants whether the experimental data followed these adsorption isotherm models. The Langmuir isotherm is represented by Eqn (1).

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \quad (1)$$

where Q_e is the equilibrium amount of adsorbate exchanged by the adsorbent (g/g), C_e is the equilibrium concentration of adsorbate in the solution (g/L), Q_m (g/g) is the maximum uptake of adsorbate exchanged and K_L is the Langmuir constant (L/g). R_L is given by Eqn (2)

$$R_L = \frac{1}{1 + K_L C_o} \quad (2)$$

Where, C_o is the initial concentration of the adsorbate in the mixture. The Langmuir constants Q_m and K_L were evaluated from the linear regression analysis of the plot C_e vs C_e/Q_e ($Q_m = 1/\text{slop}$, $K_L = 1/[Q_m \times \text{Intercept}]$).

The linear form of the Freundlich isotherm model is given by Eqn (3).

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (3)$$

Where K_f is Freundlich constant (L/g) and $1/n$ is the heterogeneity factor. The constants n and K_f were evaluated from the linear regression analysis of the plot of $\ln Q_e$ vs. C_e ($K_f = e^{\text{intercept}}$, $n = 1/\text{slop}$)

D. Kinetic Studies

The Pseudo-first-order and second-order kinetic models- The Pseudo-first-order and second-order kinetic models were tested at different concentrations (amount of BOD removed) in this study to determine which model is in good agreement with experiment q_e (adsorption capacity) value, thus suggesting which model the sorption system follows. The Pseudo-first-order-model can be expressed as in eqn(4).

$$\ln(q_e - q_t) = \ln(q_e) - \frac{K_1 t}{2.303}$$

Where, q_e , q_t (mg/g) are the mass of BOD adsorbed at equilibrium (adsorptive capacity), and mass adsorbed at any time 't', K_1 , (min⁻¹) is the equilibrium constant of the Pseudo-first-order adsorption. The values of K_1 and q_e were determined respectively from the slope and intercept of the plot of $\ln(q_e - q_t)$ versus t ($q_e = e^{(\text{intercept})}$, $K_1 = 2.303 \times \text{slope}$). The pseudo second order is given by Eqn (5).

$$\frac{t}{q_t} = \frac{1}{K_2 2 q_e^2} + \frac{1}{q_e} t \quad (5)$$

Where, K_2 (gmg⁻¹min⁻¹), t , and q_e , are the pseudo-second-order rate constant, time, and adsorption capacity at equilibrium respectively. The value of the q_e is determined from the plot of t/q_t versus t ($q_e = 1/\text{slope}$, $\text{intercept} = 1/K_2 q_e^2 t$)

E. Statistical Evaluation for Adsorption Kinetics for BOD Removal.

The Total Error and Root Mean Square Error statistical expressions were used to evaluate the performance of the kinetic model. Model estimate values were compared with observed experimental values and deductions were made as to the best fit.

a. *Total Error (Err²)*. Total error (in terms of adsorption experiment) is the sum of the squares of the error between the calculated adsorption capacity and the expected adsorption capacity of a kinetic model. This is a measure of variation in value not explained by the values obtained. The lower the value of total error obtained, the higher the accuracy, validity and good fitness of the method (model). Mathematically, Total error is given by Eqn (6)

$$\text{Err}^2 = \sum_{i=1}^n (Y_o - Y_e)^2 \quad (6)$$

Where n, Y_o , Y_e , are the number of data points, obtained experimental adsorption capacity values, and computed adsorption capacity value respectively (for the kinetic models).

b. *Root Mean Square Error (RMSE)*. It is an absolute measure of fit which can be interpreted as the standard deviation of the unexplained variance. It has the useful property of being in the same units as the response variable. Lower values of RMSE indicate better fit. It is represented by Eqn (7):

$$RMSE = \frac{1}{n} \sqrt{\sum_{i=1}^n (Y_o - Y_e)^2} \quad (7)$$

Where n, Y_o , Y_e , are the number of data points, obtained experimental adsorption capacity values, and computed adsorption capacity value respectively (for the kinetic models)

RESULTS AND DISCUSSION

Langmuir Isothermal model- Linear plots of C_e/q_e vs. C_e (Figures 1-3) were employed to determine the values of Langmuir constants Q_m (mg/g) and K_L (L/mg) from the slope and intercept of the plots respectively.

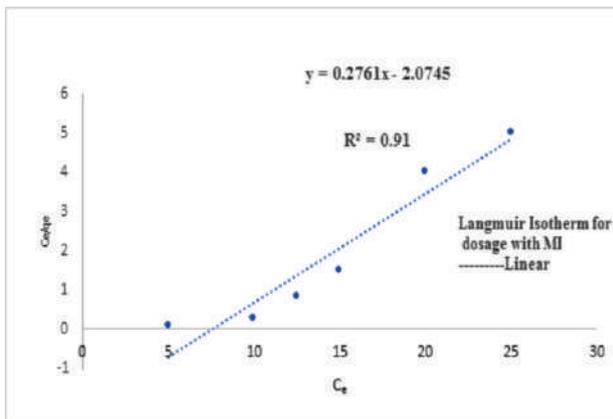


Figure 1 Langmuir Isotherm for Dosage with MI

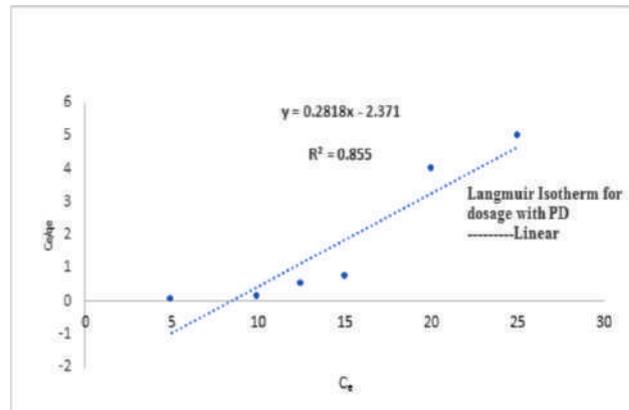


Figure 2 Langmuir Isotherm for Dosage with PD

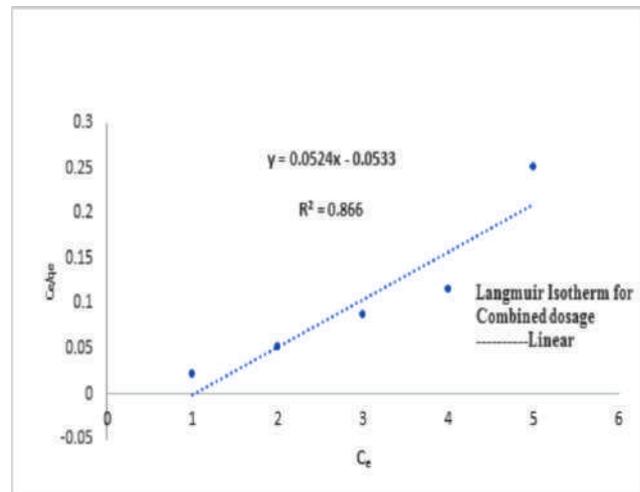


Figure 3 Langmuir Isotherms for Combined Dosage (MI & PD)

Table 1 shows the Langmuir isotherm parameters for dosages with *Mangifera indica*, *Phoenix dactylifera* and a combination of the two in varying proportions. Table 2 shows the separation factor (R_L) for the dosages. The R_L values were calculated from equation 2. The constants K_L and Q_M relate to the energy of adsorption and maximum adsorption capacity (Desta, 2013).

Table 1: Langmuir Isotherm Parameters and R^2

Parameters	MI	PD	Combination (MI &PD)
Q_m	3.62	3.55	19.08
K_L	-0.13	-0.12	-0.98
R^2	0.92	0.86	0.87

Table 2: Separation Factor (R_L) of MI, PD, and Combination (MI & PD) at Different Initial Concentrations

Initial Concentration	R_L Values		
	MI	PD	Combination (MI & PD)
5	2.86	2.50	-0.26
10	-3.33	-5.00	-0.11
12.5	-1.60	-2.00	-0.09
15	-1.05	-1.25	-0.07
20	-0.63	-0.71	-0.05
25	-0.44	-0.50	-0.04

**Initial Concentration R_L Values
MI PD Combination (MI & PD)**

52.862.50-0.2610-3.33-5.00-0.1112.5-1.60-2.00-0.0915-1.05-1.25-0.0720-0.63-0.71-0.0525-0.44-0.50-0.04

From Table 1, it was observed that the combination of the seeds gave a higher value of maximum adsorption capacity Q_M , than either of the individual seeds. The coefficient of determination R^2 , for MI was the highest which shows the degree to which the model fits the experiment. The separation factor R_L , which is indicative of the isotherm shape that predicts whether an adsorption system is 'favourable' or 'unfavourable' (Saswati and Gosh, 2005), obtained for different initial concentrations for MI, PD and the combined dosage were between the values $0 < R_L < 3$. According to Mamdouth *et al.*, (2004) Saswati and Gosh, (2005), R_L values between 0 and 1 indicate favourable adsorption, however, the negative sign on the separation factor for both seeds and their combination, for the different initial concentration indicates that the model is not linear and does not fit into the Langmuir isotherm model (Maarof *et al.*, 2005; Igwe and Abia, 2007; Kiurski *et al.*, 2012;).

Freundlich Isotherm model- The Freundlich isotherm is introduced as an empirical model, where q_e represents the amount adsorbed per amount of adsorbent at the equilibrium (mg/g), C_e represents the equilibrium concentration (mg/L), K_f and n are parameters that depend on the adsorbate and adsorbent (Desta, 2013).

Freundlich equilibrium constants were determined from the plot of $\log q_e$ vs. $\log C_e$ (Figure 4-6), with the intercept = $\log K_f$, and the gradient = $1/n$.

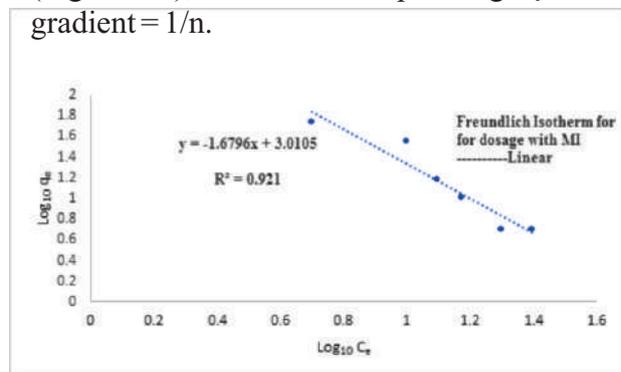


Figure 4. Freundlich Isotherm for Dosage with MI

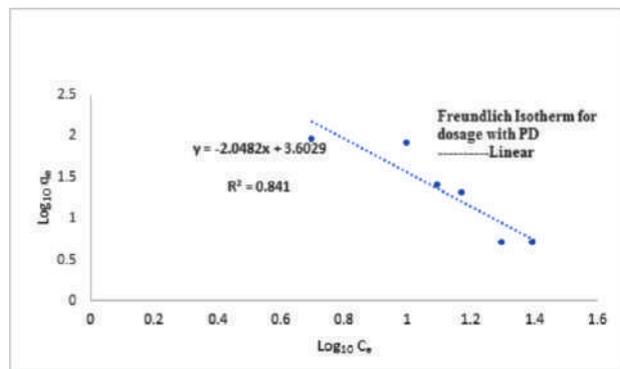


Figure 5 Freundlich Isotherms for Dosage with PD

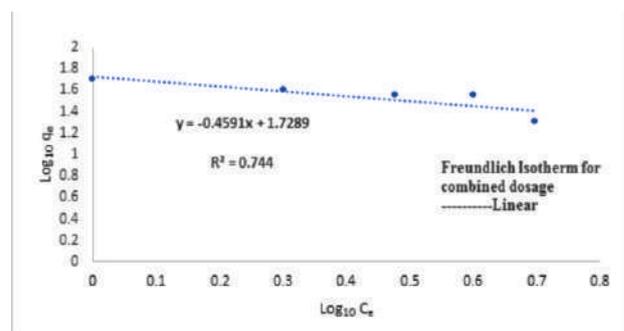


Figure 6: Freundlich Isotherm for Combined Dosage (MI & PD)

Table 4: Freundlich Isotherm Parameters and R²

Parameters	MI	PD	Comb
K _f (mg/g)	0.48	0.56	0.23
n (g/mg)	0.59	0.49	2.18
R ²	0.92	0.84	0.74

The n value indicates the degree of nonlinearity between solution concentration and adsorption as follows: if n=1, then adsorption is linear; if n<1, then adsorption is a chemical process; if n>1, then adsorption is a physical process (Desta, 2013). Values of n (which reflects the intensity of adsorption) ranging between 1 and 10; represents favorable adsorption (Adie *et al.* 2012). The values obtained (Table 4) were in the range of 0<n<1 for both MI and PD, suggesting that the process follows the Freundlich Isotherm model and is a chemical process (Al.Jlil and Latif, 2013; Desta, 2013; Kilic *et al.*, 2014). The combination (MI & PD) gave a value of n>1 which indicates that the experiment was more of a physical rather than a chemical process. The coefficient of determination R², were above 0.7 (70 %) for all the dosages suggesting the high degree to which the experimental data fits the model.

Kinetic Studies

Kinetics is of great significance to evaluate the performance of a given adsorbent and gain insight into the underlying mechanisms. The study provides information about the possible mechanism of adsorption and the different transition states on the way to the formation of the final adsorbate-adsorbent complex and helps develop appropriate models to describe the interactions (Vijayakumar *et al.*, 2011, Adie *et al.*, 2012). The Pseudo-first and second order kinetic models were studied from the plots of time versus amount (mg/l) of BOD removed using MI, PD and the Combined dosages.

Pseudo-First order kinetic model- The Pseudo-first order model is as expressed in equation (4). The values of K₁ and q_e were determined respectively from the slope and intercept of the plot of Log (q_e-q_t) versus t, as shown in Figures 7 - 9 and the values of K₁, q_e and coefficients of determination (R²) are tabulated in Table 5

Pseudo-second order Kinetic Model: The Pseudo-second order model equation is as expressed in Equation (5). The values of K₂ (g/mg/min), and q_e (mg/g), were determined respectively from the intercept and slope of the plot of t/q_t versus t as shown in Figures 10 – 12.

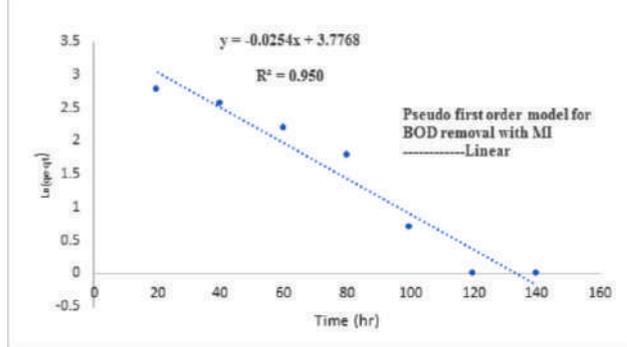


Figure 7 Pseudo first order model for BOD removal with MI

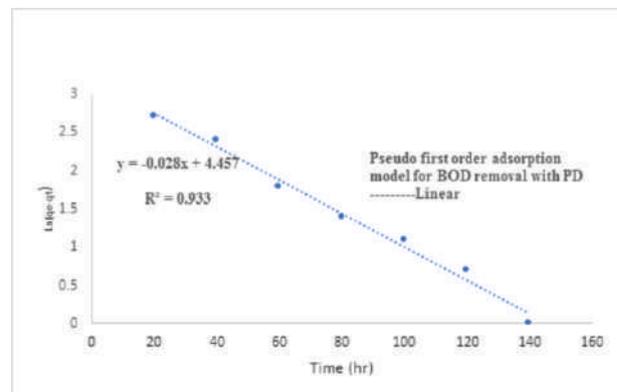


Figure 8: Pseudo first order model for BOD removal with PD

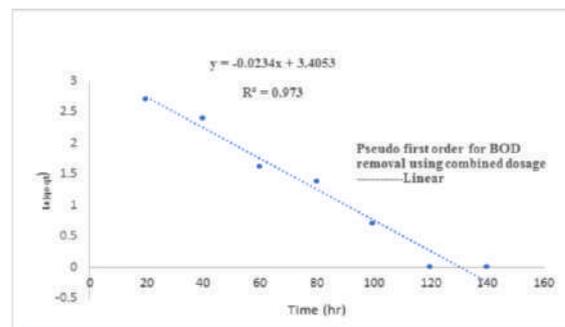


Figure 9: Pseudo first order model for BOD removal with combined dosage

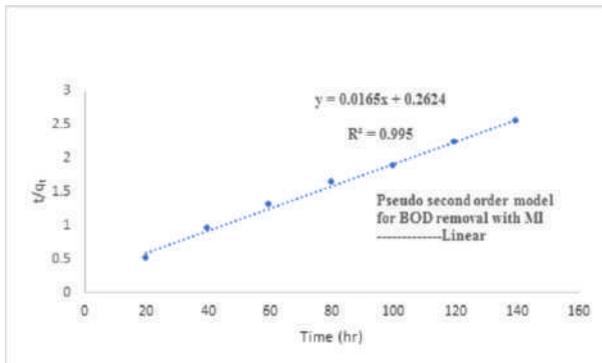


Figure 10: Pseudo second order for BOD removal with MI

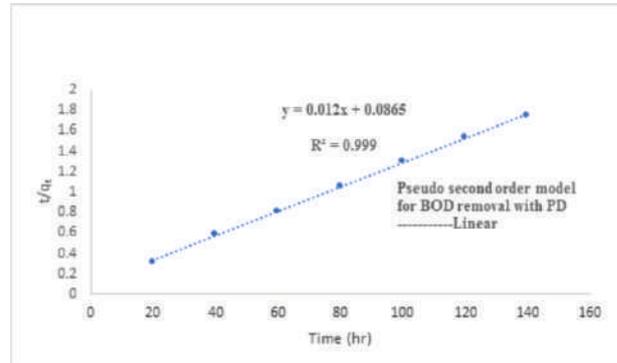


Figure 11: Pseudo second order for BOD removal with PD

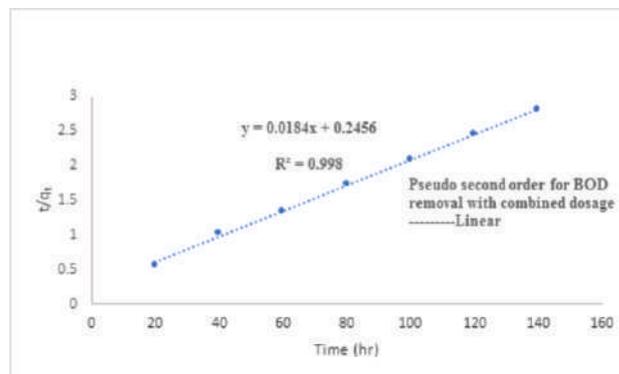


Figure 12: Pseudo second order for BOD removal with combined dosage

Table 5: Kinetic Parameters for BOD removal with MI, PD, and their combination

REACTION ORDER		MI	PD	COMB
Pseudo-first order Parameters	q_e exp. (mg/g)	55	80	50
	K_1 (min^{-1})	0.058	0.064	0.054
	q_e calc. (mg/g)	43.68	86.23	30.12
	R^2	0.95	0.93	0.97
Pseudo-second order Parameters	q_e exp. (mg/g)	55	80	50
	K_2 ($\text{gmg}^{-1} \text{min}^{-1}$)	0.0037	0.0017	0.0038
	q_e calc. (mg/g)	60.61	83.33	54.35
	R^2	0.99	0.99	0.99

From Table 5 it was observed that the calculated value of the adsorption capacity from the Pseudo-second-order models (plots) q_e calc. (mg/g), are closest to the observed experimental values of the adsorption capacity of both adsorbent from experiment q_e exp. (mg/g) for the BOD removal. The coefficients of determination, R^2 are closest to unity for the pseudo-second-order kinetics than the pseudo-first-order kinetic. This suggests that the adsorption system can be better represented by the pseudo-second-order model (Adie *et al.*, 2012). The high correlation coefficient, R^2 for the pseudo-first-order may

indicate that the present system may be followed by pseudo-first-order (Vijayakumar *et al.*, 2011, Neto *et al.*, 2013)

Statistical Evaluation for Adsorption Kinetics for BOD Removal.

Statistical evaluation for the BOD removal with *Mangifera indica*, *Phoenix dactylifera* and their combination was carried out using the Total Error and the Root Mean Square Error (RMSE) as given by equations (6) and (7). Table 6 shows the error parameters for the pseudo-first and second order.

Table 6: Statistical Evaluation for Adsorption Kinetics for BOD Removal with MI, PD and their combination

Reaction Order		MI	PD	COMB
Pseudo-first order Parameters	Total Error (Err ²)	128.14	38.81	395.21
	Root Mean Square Error (RMSE)	11.32	6.23	19.88
	R ²	0.9509	0.9333	0.9733
Pseudo-second order Parameters	Total Error (Err ²)	31.47	11.09	18.92
	Root Mean Square Error (RMSE)	5.61	3.33	4.35
	R ²	0.9954	0.9993	0.9987

From Table 6, it was observed that the pseudo-second-order kinetic model was statistically significant for the BOD removal using *Mangifera indica* and *Phoenix dactylifera* based on the higher correlation coefficients, lower total error and root mean square error values. Generally, the lower the values of Total error and RMSE, the higher the accuracy, validity and good fitness of the model (Hooper *et al.*, 2008, Adie *et al.*, 2012, Samarghandi *et al.*, 2009). This result also agrees with similar studies carried out on the evaluation of performance for BOD removal using statistical tools such as the RMSE, Regression analysis and Total error (Abyaneh, 2014; Pompeu, 2017; Wei, 2013).

CONCLUSION

The use of *Mangifera indica* and *Phoenix dactylifera* seeds powder have great potential for treatment of wastewater. The result of the Isothermal studies suggested that the experiment is a chemical process and fits the Freundlich Isotherm model.

The experiment was observed to have followed the pseudo-second order kinetic model as indicated by the K_2 , R^2 , and Error analyses carried out on the experimental data. The calculated values of the adsorption capacity, q_e (mg/g), from the pseudo-second-order models were found to be 60.61, 83.33 and 54.35 for *Mangifera indica*, *Phoenix dactylifera* and their combination respectively, and were closest to the observed experimental values of the adsorption capacity of both adsorbent from experiment $q_{e, exp}$ (mg/g) for the BOD removal.

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