

ADSORPTION ISOTHERM STUDIES OF BOD, TSS AND COLOUR REDUCTION FROM PALM OIL MILL EFFLUENT (POME) USING BOILER FLY ASH

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ABSTRACT: Palm oil is one of the two most important vegetable oils in the world's oil and fats market. The extraction and purification processes generate different kinds of waste generally known as palm oil mill effluent (POME). Earlier studies had indicated the possibility of using boiler fly ash to adsorb impurities and colour in POME treatment. The adsorption treatment of POME using boiler fly ash was further investigated in detail in this work with regards to the reduction of BOD, colour and TSS from palm oil mill effluent. The amount of BOD, colour and TSS adsorbed increased as the weight of the boiler fly ash used was increased. Also, the smaller particle size of 425µm adsorbed more than the 850µm size. Attempts were made to fit the experimental data with the Freundlich, Langmuir and Dubinin-Radushkevich isotherms. The R² values, which ranged from 0.8974–0.9898, 0.8848–0.9824 and 0.6235–0.9101 for Freundlich, Langmuir and Dubinin-Radushkevich isotherms respectively, showed that Freundlich isotherm gave a better fit followed by Langmuir and then Dubinin-Radushkevich isotherm. The sorption trend could be put as BOD > Colour > TSS. The apparent energy of adsorption was found to be 1.25, 0.58 and 0.97 (KJ/mol) for BOD, colour and TSS respectively, showing that sorption process occurs by physiosorption. Therefore, boiler fly ash is capable of reducing BOD, Colour and TSS from POME and hence could be used to develop a good adsorbent for POME treatment.

Keywords: Adsorption, Wastewater, Isotherm, Boiler fly ash, POME.

INTRODUCTION

The palm oil is of tremendous economic and social importance in Nigeria. The palm oil industry apart from being a major foreign exchange earner for Nigeria in the past has also been identified as a great source of water and land pollution. According to Majid *et al.*, (1999) [1], Palm oil mills produce crude palm oil and palm kernel as their main products. The milling process also generates considerable amounts of co-products/waste such as empty fruit bunches (EFB), palm oil

mill effluent (POME), palm fiber and palm shell. The palm oil mill effluent, the final liquid discharged after extracting the oil from the fresh fruit bunch, contains soil particles, residual oils and suspended solids but only 5% of dry matters. Palm oil sludge is the material that remains after decanting the palm oil mill effluent. Both the effluent and sludge pollute streams, rivers or surrounding land when discharged without proper treatment. These effluents cause serious depletion of dissolved oxygen and kill fish, prawns and crabs, which are important sources of food and nutrients.

The effluent discharged from an oil mill is still objectionable and the situation in many countries is quite different and much attention has been given to the subject of effective disposal [2]. The quantity of POME produced is about 60% for every tonne of fresh fruit bunches (FFB) processed [3]. Apart from the sludge water itself, which amounts to about 300 kg per tonnes of bunches milled (or about 1.5 tonnes per tonne of palm oil), there are also about 175kg of sterilizer condensate and between 40 and 140kg of effluent from hydro cyclone or clay bath separators per tonne of bunches [4]. The total amount of effluent is therefore more than half a tonne per tonne of bunches or 2.5 tonnes per tonne of oil produced in a typical mill [5]. Hence, an average of 30 tonne FFB per hour mill will generate about 18 tonnes of effluent per hour. In milling 20 tonnes of bunches per hour, more than 200 tonnes of effluent may be discharged over 24 hours and this may contain up to a tonne of oil and 9 tonnes of dissolved or suspended solids. This effluent has a biochemical oxygen demand (BOD) of about 20,000 mg/L or 20% for 5 days, which is extremely high.

Fibre and shell are termed as by-products of the oil mill and they are used as boiler fuel to produce steam for electricity generation for the mill and domestic consumption within the mill complex. Boiler fly ash is produced in palm oil mills from the burning of the fiber and shell. Every tonne of FFB produces about 4kg to 6kg of boiler ash. This porous ash, which contains about 0.28% - 1.33% phosphorus, 1.02% - 4.31% potassium, 0.39% - 3.24% calcium and 0.29% - 2.60% magnesium [6], can be used as a soil conditioner. Currently, this under utilized waste is mainly used as landfill.

Adsorption of heavy metals from aqueous solution by fly ash from palm oil mill and other sources has been reported [7-12]. In view of the maximizing waste utilization and with the prospect of Federal Environmental Protection Agency (FEPA) imposing more stringent BOD limits on palm oil mill, an adsorption method using boiler fly ash may need to be introduced. Therefore, in this study, we investigated the use of boiler fly ash for the reduction of biochemical oxygen demand (BOD), Total suspended solid (TSS) and colour from palm oil mill effluent (POME). Also, adsorp-

tion isotherms were used to correlate and model the adsorption studies.

MATERIALS AND METHODS

Materials

Samples of effluent water were collected from a palm oil mill in Okigwe, Imo State Nigeria. Boiler fly ash was also collected from the same mill. All reagents used were of analytical reagents grade and were used as purchased without further purification.

METHODS

Sample Collection and Adsorbent Preparation

Samples of effluent water were collected in different containers of about 5 liters in volume. The containers were properly washed and rinsed with the effluent before collection to avoid contamination and dilution. The containers were labeled showing time of collection and temperature at collection.

Boiler fly ash was also collected from the same mill. To do this, a large quantity was collected using polyethylene bags. The boiler fly ash was first washed with distilled water and then air-dried. The particle size distribution was determined using a Fritsch sieve shaker. This properly sieved the boiler fly ash into two particular mesh sizes of 425 μ m and 850 μ m for the purpose of the experiment. Therefore, two samples of boiler fly ash were obtained and placed in different sample containers and labeled according to their mesh sizes (425 μ m and 850 μ m). The specific surface area of the fly ash was determined using the methylene blue absorption test (MBT) method described by Santamarina *et al* (2002) [13]. The moisture con-

tent of the sample was measured by heating the fly ash in an oven at 135°C for two hours [1]. The fly ash was activated by soaking in dilute (2% v/v) HNO₃ over night. It was then rinsed with distilled water and air dried, ready for the experiments. The reason for soaking in nitric acid was to wash off soluble contaminants and activate the boiler fly ash by opening up the pores in readiness for sorption process.

Determination of Specific Surface Area (S_{AA})

The specific surface area of the adsorbent was determined using the methylene blue absorption test (MBT) method [13]. 2.0g of each adsorbent was suspended in 200mL of deionized water and 10ppm of methylene blue solution was added to the adsorbent suspension and agitated in a shaker for 2 h. There after, it was kept for 24 hr to reach equilibrium, after which 10ml aliquot was taken and centrifuged. The centrifuged aliquot was then analyzed using a UNICAM – 8700 UV/Visible spectrophotometer to determine the amount of methylene blue that remained in solution. Also, 20, 30, 40, 50, 60 and 70 ppm solutions of methylene blue were then added sequentially and the previous steps repeated. A graph of concentration of methylene blue added versus the amount of absorbed methylene blue (not shown) was used to identify the point. The specific surface area was calculated from the amount of absorbed methylene blue at the optimum point of cation replacement as follows:

$$\text{Specific surface area (m}^2 \text{ g}^{-1}\text{)} = \frac{\text{mMB} \times \text{Av} \times \text{AMB}}{(319.98) \times (1/\text{Ms})} \quad (1)$$

Where;

mMB = Amount of methylene blue absorbed at the point of complete cation replacement.

Ms = Mass of adsorbent

Av = Avogadro's number, 6.02×10^{23}

AMB = Area covered by one methylene blue (assumed to be 130 \AA^2)

Sorption Experiments

Effect of weight of fly ash

The adsorption experiments were conducted in a batch system by preparing various amounts of fly ash (10, 30, 40 and 60g) of the two different mesh sizes in series of 300ml wastewater. The various mixtures of fly ash and wastewater were allowed to stand with continuous stirring for 60 mins contact time, at a constant temperature of 30°C and pH of 6.4. After the contact time of 60 mins had elapsed, the mixture was centrifuged at 3000 rpm and the supernatant analyzed for BOD, TSS, colour and pH by the same methods described below.

A control sample was prepared by filtering the wastewater without the fly ash. The filtrates were then analyzed for BOD, TSS, and colour as described later. The reductions of these parameters as consequence of adsorption were calculated by difference in the initial concentrations and that remaining in the filtrates.

Effect of Initial Concentration

To investigate the effect of initial concentration of the wastewater on the adsorption process, various initial concentrations were obtained by diluting the wastewater with distilled water. Initial concentrations of 10%, 30%, 40%, 50%, 60% and 70% were used. 30 grams of the boiler fly ash of 800 μ m size was put into 300ml wastewater of different concentrations and allowed to stand with continuous stirring for 60 minutes contact time. After 60 minutes contact time, the mixtures were centrifuged at 3000rpm for 10 mins and the supernatant analyzed for BOD, TSS, colour and pH by the same methods described below.

Analyses of Various Parameters

The pH of the solution was determined using pH meter (HACH 51750 – 60) [14]. The pH meter was first standardized using a buffer solution of about 7.0 and then the other solutions were analyzed for their various pH values.

All samples were analyzed for the other parameters as described in the standard methods for the Examination of water and wastewater [15] and standard methods for water and effluents analysis [16].

RESULTS AND DISCUSSION

The particle sizes of the fly ash used were 450µm and 850µm. The specific surface area of the boiler fly ash calculated from the methylene blue absorption test method was 241.4m²g⁻¹ for

850µm and 280.6m²g⁻¹ for 425µm. The fly ash was found to have moisture content of 1.7%.

Sorption Capacity

The experimental results for the adsorptive removal of BOD, colour and TSS using boiler fly ash are presented. The pH of the palm oil mill effluent was found to be 6.4. The amount adsorbed was calculated from equation (2) below:

$$q_e = C_o - C_e \quad (2)$$

here q_e is the amount adsorbed in mg/g for BOD and TSS and in Hz for colour adsorption; C_o is the initial concentration and C_e is the residual amount (concentration) after sorption process

Figure 1 shows the amount adsorbed for BOD, colour and TSS from POME as weight (g) of boiler fly ash was varied for particle size of 425µm, while that for particle size of 850µm is shown on Figure 2.

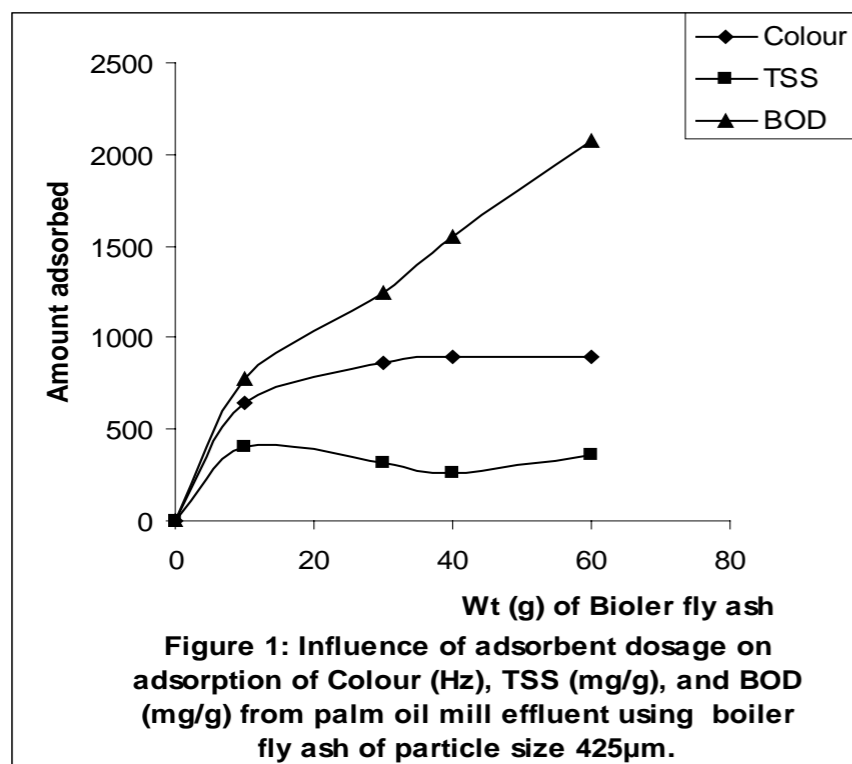


Figure 1: Influence of adsorbent dosage on adsorption of Colour (Hz), TSS (mg/g), and BOD (mg/g) from palm oil mill effluent using boiler fly ash of particle size 425µm.

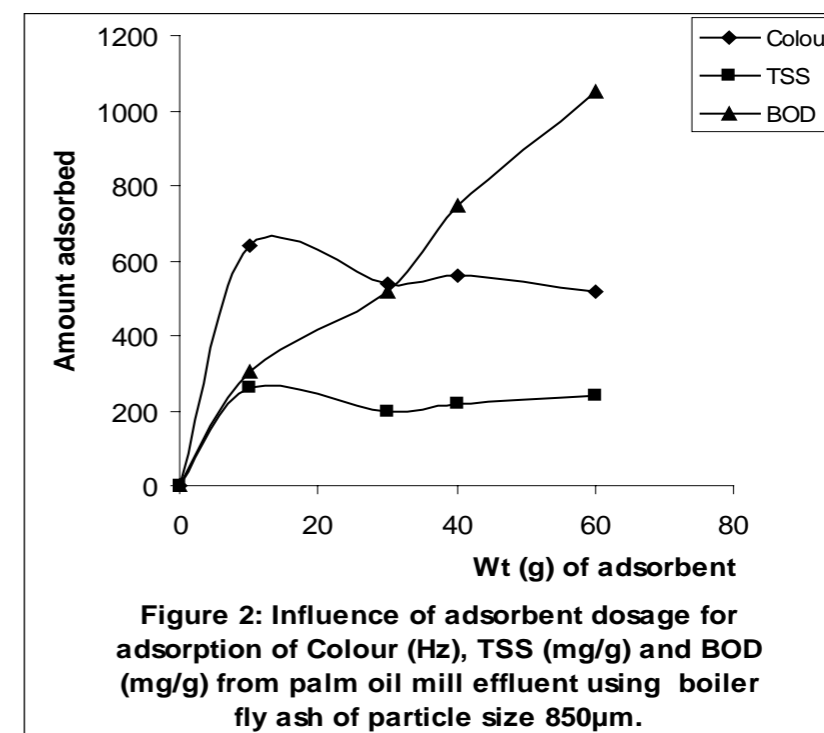
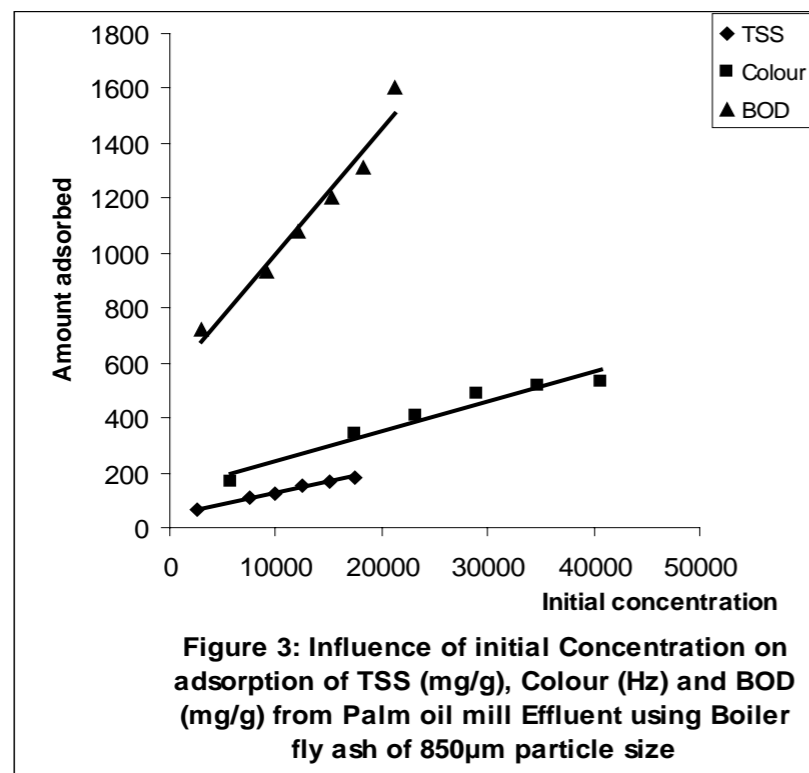


Figure 2: Influence of adsorbent dosage for adsorption of Colour (Hz), TSS (mg/g) and BOD (mg/g) from palm oil mill effluent using boiler fly ash of particle size 850µm.

From these figures, it can be seen that for BOD, both for 425µm and 850µm sizes of boiler fly ash, the amount adsorbed increased steadily as the weight of fly ash was increased. For TSS there are little changes and for colour (850µm particle size), the amount sorbed decreased as the amount of fly ash increased. Also, it can be seen that particle size of 425µm gave higher sorption capacity than particle size of 850µm.

The amount adsorbed as initial concentration increased for TSS, BOD and colour is shown on Figure 3. In each case, the amount adsorbed increased as the initial concentration of each of the parameters increased. The amount of BOD adsorbed was higher followed by colour and then TSS. The linear regression equations for each of the parameters and the regression coefficient are shown as equations 3, 4, and 5 for BOD, colour and TSS respectively. It could be seen that all the R² values are high showing good correlation. The R² value for TSS was highest, followed by BOD and then that for colour.

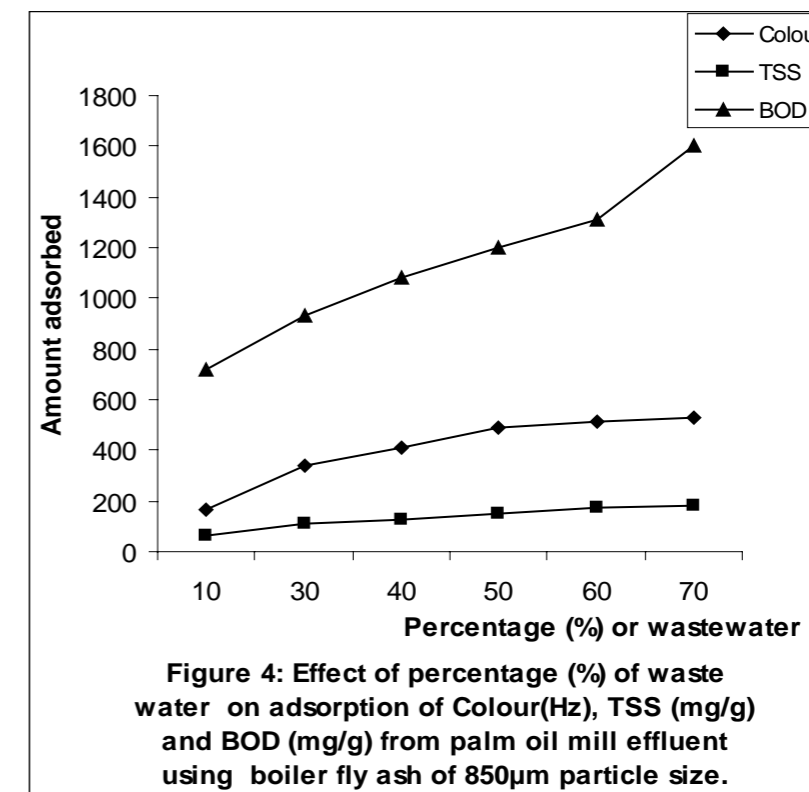


$$y = 0.0458x + 536.43 \quad (R^2 = 0.9668) \quad (3)$$

$$y = 0.0108x + 137.5 \quad (R^2 = 0.9422) \quad (4)$$

$$y = 0.0081x + 46.571 \quad (R^2 = 0.9962) \quad (5)$$

The amount adsorbed against the percentage of wastewater for BOD, colour and TSS from palm oil mill effluent using boiler fly ash is shown on Figure 4. It could be seen that the amount adsorbed increased as percentage of wastewater was increased. The amount of BOD adsorbed was highest, followed by colour and then TSS.



In all, it is clear that the boiler fly ash was capable of reducing the concentrations of BOD, TSS and colour from palm oil mill effluent (POME). The adsorptive removal of these contaminants could be explained based on the modes of sorption of materials or contaminants on adsorbents. The adsorptive removal of contaminants could be attributed to two main terms; intrinsic adsorption and coulombic interaction [17]. The coulombic term results from the electrostatic energy of interactions between the adsorbents and adsorbates. It can also be observed from the adsorption of cationic species versus anionic species on adsorbents [17]. The intrinsic adsorption of the materials is determined by their surface areas, which can be observed by the effect of different sizes of adsorbents. The smaller particle size of 425µm has a higher adsorption. Also, both factors can interact, thereby influencing the adsorption capacity. The specific surface area (S_{AA}) of an adsorbent is the ratio of its surface area to its mass [18]. It has been reported that the surface area has

a great effect on the sorption capacities of adsorbent [19]. Thus, increase in specific surface area increases sorption capacity.

The trend of this sorption capacity could be put as BOD > colour > TSS. This trend could be explained based on solubility and diffusion processes. BOD is defined as the amount of dissolved oxygen needed to break down the carbonaceous component of the waste. This means that BOD involves a dissolved component of wastewater. Also, colour is a consequence of dissolved component of the waste but total suspended solid (TSS) is not. Diffusion takes place before adsorption of the contaminants, most dissolved contaminants diffuse faster than suspended particles, hence will be adsorbed more. Therefore, in our opinion, this is the reason for the sorption trend. The pH could be seen as a resultant effect of the interactions of the other parameters. That is, changes in pH of the wastewater are dependent on the changes in the hydrogen ion concentration which in turn is dependent on the dissolved components of the wa-

stewater. Also, it has a fixed range of values, that is, the pH scale.

Sorption Isotherm

When an adsorbent is in contact with the surrounding fluid of a certain composition, adsorption takes place and after a sufficiently long time, the adsorbent and the surrounding fluid reach equilibrium. Generally, an adsorption isotherm is an expression or diagram, which provides information about the fraction of a surface that is covered by adsorbed molecules in equilibrium at constant temperature as a function of pressure or concentration [20]. Adsorption isotherms are described in many mathematical forms, some of which are based on a simplified physical picture of adsorption and desorption, while others are purely empirical relationship intended to correlate the experimental data in simple equation with two or at most three empirical parameters, the more the number of empirical parameters, the better the fit between experimental data [21]. Three isotherms; Freundlich, Langmuir and Dubinin–Radushkevich isotherms were chosen to analyze the adsorption process. The Freundlich and Langmuir isotherms are the earliest and simplest known relationships.

The Freundlich isotherm model was chosen to estimate the adsorption intensity. It is an empirical equation after Freundlich in 1926, used to describe the isotherm data by;

$$Q_e = K_F (C_e)^{1/n} \quad (6)$$

Where K_F and n are empirical constants [22]. Equation (6) may be linearized by taking logarithms as follows:

$$\log Q_e = \log K_F + 1/n \log C_e \quad (7)$$

Where;

K_F = Constant in Freundlich isotherm

n = Freundlich exponent

C_e = Equilibrium liquid phase concentration

Q_e = Equilibrium solid phase concentration

The plot of $\log Q_e$ against $\log C_e$ is linear and constants K_F and n were evaluated from the intercepts and slopes respectively. The Freundlich isotherm plots are shown on Figure 5 for TSS, BOD and colour. The regression equations and the

coefficient of regression (R^2) are shown as equations 8, 9 and 10 for BOD, Colour and TSS respectively.

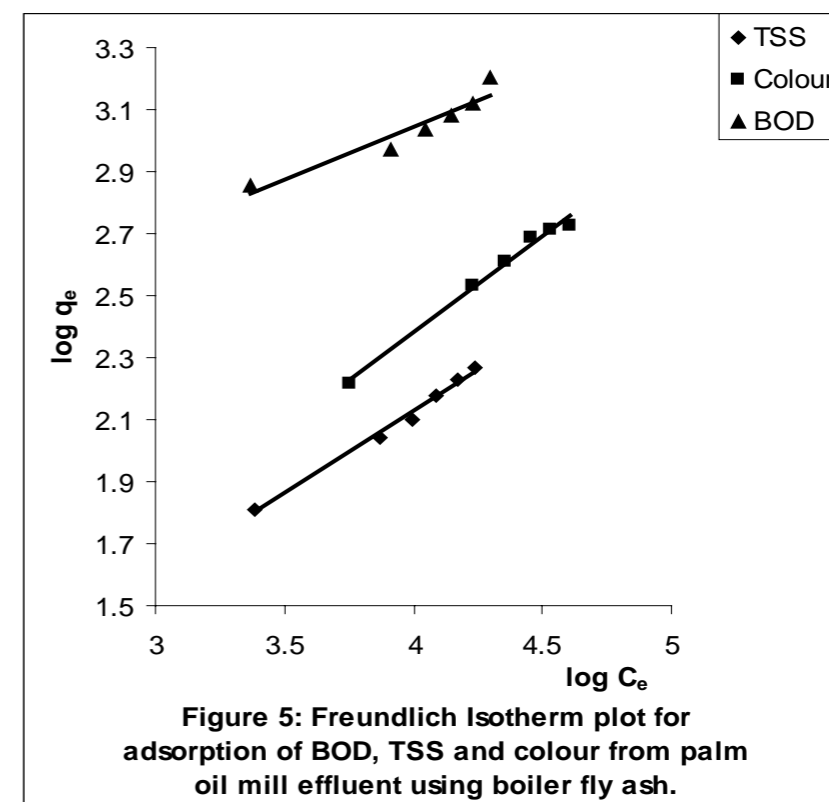


Figure 5: Freundlich Isotherm plot for adsorption of BOD, TSS and colour from palm oil mill effluent using boiler fly ash.

$$y = 0.3381x + 1.6912 \quad R^2 = 0.8974 \quad (8)$$

$$y = 0.6195x - 0.0964 \quad R^2 = 0.9898 \quad (9)$$

$$y = 0.5315x + 0.0002 \quad R^2 = 0.9893 \quad (10)$$

intensity. Hence the higher value of K_F for BOD adsorption followed by colour and then TSS.

From these equations and R^2 values, it could be seen that the Freundlich equation gave a very good fit to the adsorption process. With R^2 value of 0.9898 for colour reduction, which is the highest, it means that the Freundlich isotherm fits the adsorption of colour more, followed by TSS and then BOD. The values of the Freundlich constants K_F and n were generated and are shown on Table 1. The Freundlich exponents ($1/n$) are all less than one showing that significant adsorption takes place at low concentration but the increase in the amount adsorbed with concentration becomes less significant at higher concentration and vice versa [22]. Also, K_F and n determines the curvature and steepness of the isotherm [23]. Thus, the higher the K_F value, the greater the adsorption

TABLE 1. Adsorption isotherm constants for Freundlich, Langmuir and Dubinin-Radushkevich isotherm for adsorption of BOD, Colour and TSS using boiler fly ash.

Freundlich isotherm				
Parameter	$1/n$	$K_F(L/g)$	R^2	
TSS	0.53	1.00	0.9893	
Colour	0.62	0.80	0.9898	
BOD	0.34	49.11	0.8975	
Langmuir isotherm				
Parameter	As(mg/g)	K(L/g)	R^2	
TSS	277.78	1.0×10^{-4}	0.9118	
Colour	909.09	4.29×10^{-5}	0.9824	
BOD	2000.00	1.47×10^{-4}	0.8851	
Dubinin-Radushkevich isotherm				
Parameter	$\beta(KJ/mol)$	$q_D(mg/g)$	E(KJ/mol)	R^2
TSS	0.82	152.81	0.06	0.8102
Colour	5.44	478.81	0.03	0.9101
BOD	0.47	1233.24	0.02	0.6235

The Langmuir equation was chosen for the estimation of maximum adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface. The Langmuir isotherm is represented by the following equation [24];

$$q_e = K_L C_e / (1 + a_L C_e) \quad (11)$$

The linearized form of equation (11) could be written as;

$$C_e/q_e = 1/K_L + (a_L / K_L) C_e \quad (12)$$

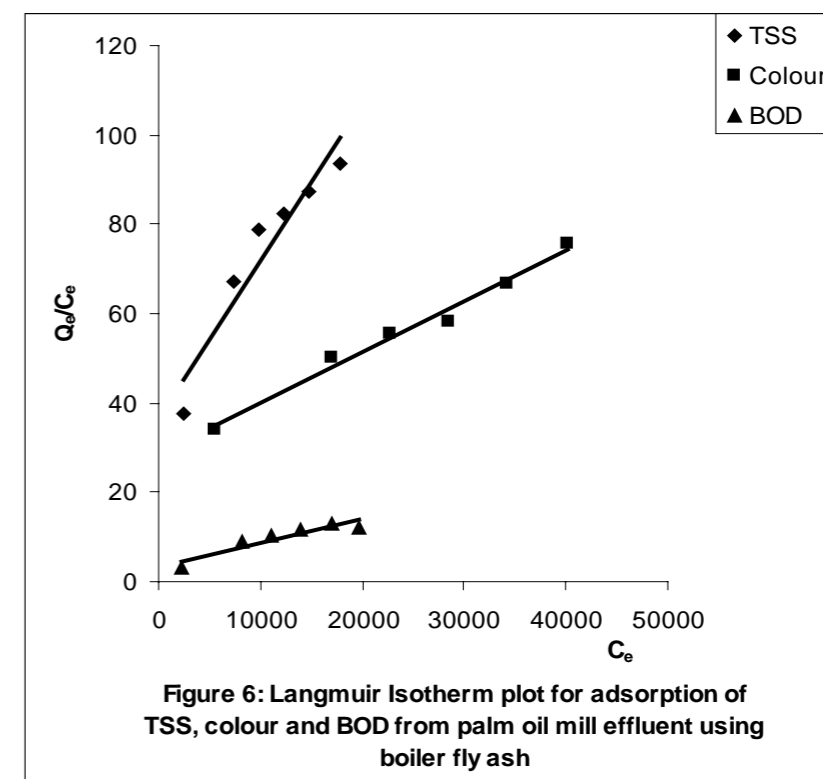
Where;

C_e is the equilibrium concentration of the parameter; q_e is the amount of parameter adsorbed. The constants K_L and a_L are the Langmuir constant (L/g) and Langmuir isotherm constant (L/mg), respectively. The ratio of a_L/K_L represents the maximum adsorption capacity, Q_{max} . Another form of the Langmuir equation [25], shown below was used to model the adsorption data.

$$C_e/Q_e = 1/K_A s + C_e/As \quad (13)$$

Where As is the sorbent binding capacity and K is the binding constant. The plots of C_e/Q_e against C_e gave straight lines and are shown on Figure 6 for TSS, BOD and colour adsorption

on fly ash. The regression equations and the linear coefficient of regression (R^2) are shown on the plots. The plots and the R^2 values show that the Langmuir model gave did not give a very good fit to the sorption process for BOD and TSS. The values of the Langmuir constants are show on Table 1. It is well known that the Langmuir equation is intended for a homogeneous surface. A good fit of this equation reflects monolayer adsorption [22]. Therefore, the higher the adsorption, the greater the value of the sorbent binding capacity (As).



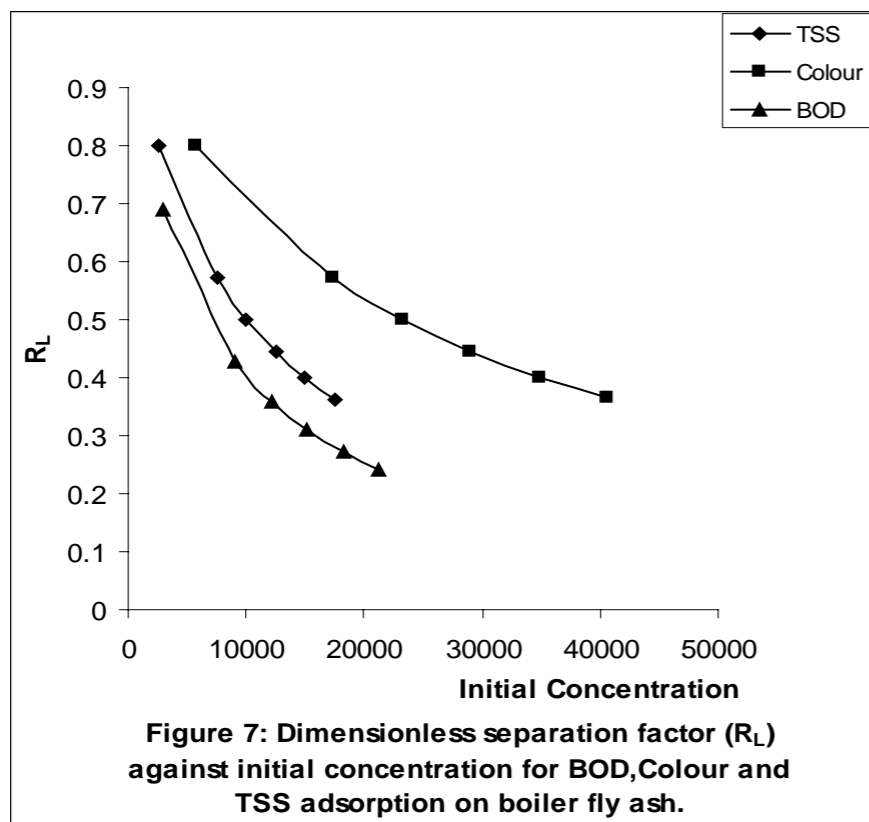
The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter, R_L , which describes the type of isotherm [26] and is defined by the equation (11);

$$R_L = 1 / (1 + K_L C_o) \quad (14)$$

Where, C_o is the initial concentration of the parameter. The parameter R_L shows the shape of the isotherm according to Table 2. The R_L values were calculated and plotted as shown on Figure 7. The values of R_L range from 0.2422 to 0.6911 of BOD; 0.3647 to 0.8009 for colour and 0.3636 to 0.8000 for TSS at various initial concentrations. From Figure 7, it means that the sorption process is favourable because the values lie between zero and one.

TABLE 2. R_L values and isotherms

R_L values	Type of isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible



The Dubinin-Radushkevich isotherm was chosen to estimate the characteristic porosity of the fly ash and the model is represented by equation (15) [27];

$$q_e = q_D \exp(-\beta [RT \ln(1+1/C_e)]^2) \quad (15)$$

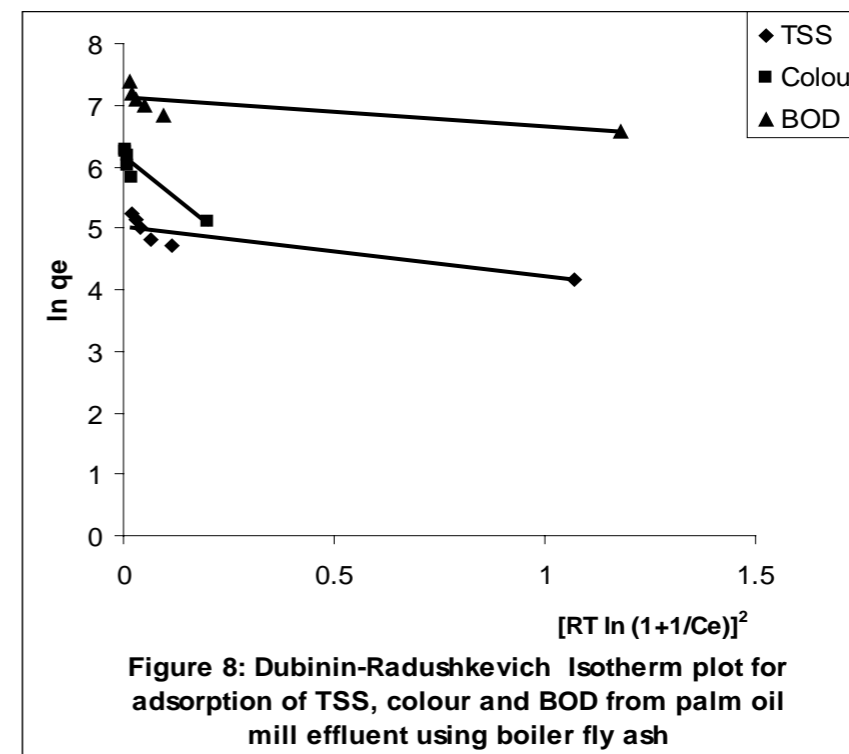
Where β is related to the free energy of sorption per mole of the sorbate as it migrates to the surface of the adsorbent from infinite distance in the solution and q_D is the Dubinin – Radushkevich isotherm constant related to the degree of sorbate sorption by the sorbent surface. The linear form of equation (15) is given as;

$$\ln q_e = \ln q_D - \beta \varepsilon^2 \quad (16)$$

Where, ε is the Polanyi potential [28,29] which is related to the equilibrium concentration as;

$$\varepsilon = RT \ln(1+1/C_e) \quad (17)$$

The plots of $\ln q_e$ against $[RT \ln(1+1/C_e)]^2$ gave fairly straight lines and are shown on Figure 8 for TSS, BOD and colour adsorption on fly ash. The Linear regression equations and R^2 values show that the isotherm model does not describe BOD and TSS removal process very well (Table 1). The apparent energy of adsorption from the Dubinin–Radushkevich isotherm model can be computed using the relationship [27],



$$E = 1 / (2 \beta)^{1/2} \quad (18)$$

CONCLUSION

From the Dubinin–Radushkevich isotherm, the values of q_D , which is related to the degree of sorbate sorption, shows that BOD has a higher value (1233.24), followed by colour (478.81) and then TSS (152.81). This also shows that the trend of sorption is BOD > colour > TSS. Also, from the value of β , the apparent energy (E) of adsorption was calculated. The values of the energy 0.06, 0.03 and 0.02 (KJ/mol) for TSS, colour and BOD respectively showed that the sorption of BOD, colour and TSS are physisorption process. This is because the values obtained are within the range for physisorption. Thus, what takes place is physical sorption without chemisorptions. The values of the Dubinin–Radushkevich isotherm constants are also shown on Table 1, with the R^2 values and the apparent energy of adsorption.

Boiler fly ash was utilized as an adsorbent for the reduction of BOD, colour and TSS from palm oil mill effluent (POME). Adsorption studies were carried out to investigate the effect of weight (g) of fly ash and initial concentration of the effluent. The boiler fly ash was capable of removing BOD, colour and TSS with the efficiency of BOD removal being higher than colour and TSS. The amount adsorbed increased as weight (g) of boiler fly ash was increased. The sorption process was modeled using three sorption isotherms namely; Langmuir, Freundlich and Dubinin-Radushkevich isotherms. Dubinin-Radushkevich isotherm cannot be used to describe the removal of TSS Colour and BOD by this sorbent. Langmuir isotherm can be used to describe Colour and TSS removal. BOD removal cannot be described by the three isotherm models. Colour removal is more easily described, a good correlation is found when applying the Freundlich and Langmuir models. Generally, from the coefficient of determina-

tion (R^2) for the three isotherms, we can compare their fitness to the experimental data as Freundlich > Langmuir > Dubinin-Radushkevich. From the results of this study, it means that boiler fly ash can be used as an adsorbent to further reduce the concentration of BOD, colour and TSS from POME. Therefore, because boiler fly ash is a waste in the palm oil industry, this is a good step in the right direction to industrial waste management.

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