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Fixed-bed adsorptive removal of metanil yellow from simulated wastewater in a fixed-bed column by nitric acid-treated- H_3PO_4 -activated carbon (NATPAAC) from oil palm fruit mesocarp fibre

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ABSTRACT

We carried out fixed-bed column adsorption of metanil yellow from simulated wastewater on NATPAAC derived from oil palm fruit mesocarp fibre so as to determine the adsorption capacity, q_e , of the carbon under the effects of inlet concentration, C_o , carbon bed height, H , and the dye solution flow rate, Q . Our results indicate that the optimum q_e was 15.982 mg/g by C_o 25 mg/L, H 4.1cm and Q 8 mL/min. In the study, q_e was observed to decrease with the increase in C_o and Q . The optimum bed height was 4.1 cm. Our experimental data were modelled by applying Thomas and Yoon-Nelson kinetic models. Correlation coefficients, R^2 values (generally above 0.85) show that the two kinetic approaches provide an effective model of the experimental data. We conclude that oil palm fruit mesocarp fibre has potential as a precursor for production of carbon for acid-dye removal from wastewater.

Keywords: Adsorption capacity, activated carbon, fixed-bed column, kinetic models, metanil yellow

1. INTRODUCTION

Increasing awareness of water pollution and its far-reaching effects in recent times have prompted concerted efforts toward pollution abatement. A lot of wastewater treatments that are carried out in industries are focused toward treating water polluted with organic pollutants due

to the fact that organics are common waste constituents (Bello *et al.*, 2012). A large fraction of these industrial effluents contain dyes (Aksu, 2005). Various aquatic lives are jeopardized since the discharge of the dyes effluents, which are highly coloured wastewater, to aquatic systems can block the penetration of sunlight and oxygen, thereby arresting photosynthesis of aquatic plants. Many of the dyes are carcinogenic (Crini, 2006). Metanil yellow ([3-(4-anilinophnylazo)] benzene sulphonic acid sodium salt, also called Acid Yellow 36, is an organic compound which constitutes one or more azo bonds (-N=N-) used in a number of industries, such as dyeing, food, cosmetics, paper printing, and textiles industries with the textiles industry being a very large consumer. Metanil yellow is toxic if absorbed through the skin, respiratory, and intestinal tract, and may act as a skin, eye, or respiratory irritant. It is harmful when swallowed or inhaled, and may be carcinogenic under a long time exposure (Jain *et al.*, 2009; Sivashankar *et al.*, 2013). Biological or physicochemical techniques, which include adsorption, oxidation-reduction, chemical coagulation, ozone treatment, and membrane filtration, are employed in removing waste dyes from the polluted water (Pramanpol and Nitayapat, 2006)

Adsorption is the concentration of a substance at the surface. The adsorption at a surface or interface is largely as a result of binding forces between atoms, molecules, and ions of the adsorbate on the surface, (Isiuku *et al.*, 2014). Adsorption onto activated carbon has been found to be superior to other techniques of wastewater treatment because of its capability to adsorb a broad range of different types of adsorbates efficiently, and its simplicity of design, (Ahmad and Hameed, 2006). Adsorption is a well-established technique for the removal of low concentrations of organic pollutants from large volumes of potable water, process effluents, wastewater, and aqueous solutions (Patel and Vashi, 2012)

Batch adsorption experiments are used easily in the laboratory for the treatment of small volumes of effluents, but less convenient to use on industrial scale, where large volumes of wastewater are continuously generated. Batch adsorption provides a certain preliminary information which includes pH for maximum adsorption, maximum initial adsorbate concentration, particle size for optimum adsorption of adsorbate, approximate time for adsorption of adsorbate, and the maximum adsorption capacity of the adsorbent. All these informations are useful in packed-bed studies (Unuabonah *et al.*, 2010).

For the removal of toxic organic compounds from wastewaters, packed-bed columns containing activated carbon are used successfully. In the fixed-bed adsorption, the adsorbate is continuously in contact with a given quantity of fresh adsorbent, thus providing the required concentration gradients between adsorbent and adsorbate for adsorption (Unuabonah *et al.*, 2010; Markovska *et al.*, 2001). Fixed-bed adsorption of pollutants involves percolation of wastewater through a percolating material. In course of the flow of the wastewater through the percolator, the wastewater is purified by physicochemical processes. The design and theory of fixed-bed adsorption systems focuses on establishing the shape of the breakthrough curve and its velocity through the bed. Breakthrough and bed volumes are employed in the determination of the performance of a fixed-bed described using the concept of breakthrough curve (Unuabonah *et al.*, 2010; Cheng and Wang, 2000). Activated carbons are usually obtained from materials with high carbon content and possess a great adsorption capacity, which is mainly determined by their porous structure (Otero *et al.*, 2003). The inherent nature of the starting material and the method and conditions employed for synthesizing carbon strongly affect the final pore size distribution and the adsorption properties of the activated carbons.

Oil palm fruit mesocarp fibre is the fibre obtained after expressing oil from the fruit mesocarp. For every tonne of fresh fruit bunches processed, 120 kg of the fibre is produced. Oil

palm fruit mesocarp fibre contains on a dry weight basis, approximately 40% cellulose, 21% lignin, 24% pentosan, and 5% ash (Nwabanne and Igbokwe, 2012). Researches had been carried out in removing metanil yellow from aqueous solution by adsorption, but work on removal of the dye by fixed-bed adsorption on oil palm fruit mesocarp fibre carbon is rare. The packed-bed adsorption of metanil yellow on oil palm fruit mesocarp fibre carbon was carried out to determine the adsorption capacity of the carbon under the influences of C_0 , H , and Q . Experimental data were modelled with Thomas and Yoon-Nelson kinetic models.

2. MATERIALS AND METHODS

2. 1. Adsorbate

Metanil yellow (Merck, Switzerland), also named 3-(4-anilinophenylazo) benzene sulphonic acid sodium salt, C.I. Acid Yellow 36, Tropaeoline G, and Acid Leather Yellow R (**Fig. 1**) used in this work, was purchased at Onitsha, Nigeria. The adsorbate was used without further treatment. The stock solution was prepared by dissolving 1 g per litre solution with a distilled water.

2. 2. Preparation of activated carbon

The method of (Isiuku *et al.*, 2013; Ahmad and Hameed, 2010) was employed. The oil palm mesocarp fibre biomass collected at an oil mill at Umuapu, Ohaji, Imo State, Nigeria, was washed with distilled water to remove dirt and dust. After drying under sunlight for three days, the fibre was ground. The biomass was soaked in 20% H_3PO_4 at a ratio of 1 biomass : 3 H_3PO_4 by mass overnight. Excess acid was drained off and the biomass dried. The dry acid-treated fibre was carbonized at 500 °C for 5 h, cooled and washed with distilled water to pH 6. The washed carbon was dried in a hot air oven at 110 °C for 2 h. After drying and cooling the carbon was treated with 1 M HNO_3 solution overnight. Excess acid was decanted and the carbon was washed with distilled water to pH 6, dried at 110 °C, for 2 h, cooled and sieved to get 0.420-0.841 mm particle sizes. The carbon was packed in an airtight plastic container.

2. 3. Adsorption Process

The adsorption column was a cylindrical Pyrex glass tube of measurement 30 cm in length and 1.8 cm internal diameter. The adsorption tube was plugged at the base with glass wool. A known mass of the carbon (equivalent to 2.1, 4.1, or 6.3 cm height) was introduced. The adsorbent was sealed with glass wool and the remaining space in the tube packed with glass beads. The glass beads ensured uniformity in the flow of the adsorbate. Different working concentrations of the dye obtained by diluting the stock solution with distilled water (25, 50, or 100 mg/L) were made to flow upward through the column at the specific flow rates (8, 16, or 26 mL/min) for a given time at 29 °C with the help of a metering pump (Chem – Tech Pal No. 0-111.808). Samples of the effluent were collected at specific intervals for analysis using a UV-Visible spectrophotometer (Shimadzu UV-752, Japan), at $\lambda_{max} = 440$ nm.

2. 4. Analysis of fixed-bed column data

The major characteristic of fixed-bed column adsorption is the history of effluent concentration (Nwabunne and Igbokwe, 2012). These concentrations – time curves are

commonly called the breakthrough curves, and the time at which the effluent concentration reaches the threshold value is called the breakthrough time. The breakthrough curves show the loading behaviour of the adsorbate to be removed from solution in a fixed-bed column and are usually expressed in terms of adsorbed adsorbate concentration C_{ad} , influent adsorbate concentration C_o , effluent adsorbate concentration, C_t , or normalized concentration (C_t/C_o), as a function of time or volume of effluent for a given bed height or adsorbent mass (Patel and Vashi, 2012; Ahmad and Hameed, 2006; Taty-Custodes *et al.*, 2005; Aksu and Gönen, 2004). The effluent volume (V_{eff}) can be calculated from eqn. 1:

$$V_{eff} = Qt_{tot} \dots \dots \dots (1)$$

where: t_{tot} is the total flow time (min).

The area under the breakthrough curve (A) obtained by integrating the adsorbed concentration [C_{ad} (mg/L)] versus time (min) plot can be used to find the total adsorbed adsorbate quantity (maximum column capacity) (q_{tot}). For a given influent concentration and flow rate, q_{tot} is determined from eqn. 2:

$$q_{tot} = \frac{QA}{1000} = \frac{Q}{1000} \int_{t=0}^{t=t_{tot}} C_{ad} dt \dots \dots \dots (2)$$

The total amount of dye sent to the column (m_{tot}) is determined using eqn. 3:

$$m_{tot} = \frac{C_o Qt_{tot}}{1000} \dots \dots \dots (3)$$

Total removal (%) of adsorbate (column performance) with respect to flow volume can also be found from the ratio of total adsorbed quantity of adsorbate (q_{tot}) to the total amount of adsorbate sent to the column (m_{tot}), eqn. 4:

$$Total\ removal\ (\%) = \frac{100q_{tot}}{m_{tot}} \dots \dots \dots (4)$$

Equilibrium uptake of the adsorbate q_e or column adsorption capacity at the end of total flow time is defined by eqn. 5 as the maximum column capacity per the amount of adsorbent used x (g)

$$q_e = \frac{q_{tot}}{x} \dots \dots \dots (5)$$

Un-adsorbed adsorbate concentration at equilibrium in the column, C_e (mg/L), can be calculated from eqn. 6:

$$C_e = \frac{(m_{tot} - q_{tot})1000}{V_{eff}} = \frac{(m_{tot} - q_{tot})100}{Q_{tot}} \dots \dots \dots (6)$$

2. 5. Column adsorption modeling

A number of mathematical models have been developed for the evaluation of efficiency and applicability of the column models for large scale operations. The Thomas and Yoon-Nelson models were used to analyze the behaviour of adsorbent-adsorbate system in this work.

2. 5. 1. Thomas kinetic model

The Thomas model is one of the most general and widely used models in column performance simulation. The expression by Thomas for an adsorption column (Unuabonah *et al.*, 2010; Fu and Viraraghavan, 2003) is given by eqn. 7:

$$\frac{C_t}{C_o} = \frac{1}{1 + \exp[K_{Th} (q_o x - C_o V_{eff})/Q]} \dots \dots \dots (7)$$

where: C_t (mg/L) is the effluent adsorbate concentration at time t , q_o (mg/g) the maximum adsorption capacity of the adsorbent, x (g) mass of adsorbent used, and K_{Th} (mL/mg min) the Thomas rate constant.

The value t (min) is expressed as eqn. 8:

$$t = \frac{V_{eff}}{Q} \dots \dots \dots (8)$$

The linearized form of Thomas model is expressed as eqn. 9:

$$\ln \{ [C_t/C_o] - 1 \} = \frac{K_{Th} q_o 1000 x}{Q} - K_{Th} C_o t \dots \dots \dots (9)$$

The Thomas rate constant (or kinetic coefficient) K_{Th} and the maximum adsorbate adsorption capacity of the adsorbent q_o can be determined from the plot $\ln [(C_o/C_t) - 1]$ versus t .

2. 5. 2. Yoon-Nelson model

The Yoon-Nelson model (Kundu and Gupta, 2007) is based on the assumption that the rate of decrease in the probability of adsorbate and the probability for a single component system is expressed as eqn. 10:

$$\ln \left[\frac{C_t}{(C_o - C_t)} \right] = K_{YN} t - \tau K_{YN} \dots \dots \dots (10)$$

where: K_{YN} (min^{-1}) is the Yoon-Nelson rate constant, t (min) sampling time and τ (min) the time required for 50% adsorbate breakthrough.

The calculation of theoretical breakthrough curves for a single-component system requires the determination of the parameters K_{YN} and τ for the adsorption from the slope and intercept, respectively of a straight-line plot of $\ln [C_t/(C_o - C_t)]$ versus sampling time t .

The slope yields K_{YN} and the intercept $-\tau K_{YN}$. Based on the obtained value of τ the adsorption capacity, q_{oYN} can be calculated (Patel and Vashi, 2012) applying eqn. 11:

$$q_{oYN} = \frac{q_{tot}}{x} = \frac{C_o Q \tau}{1000x} \dots \dots \dots (11)$$

3. RESULTS AND DISCUSSION

3. 1. Effect of inlet dye solution concentration

The effect of C_o on the fixed-bed adsorption of metanil yellow on NATPAAC from oil palm fruit mesocarp fibre at fixed H and Q are shown in **Fig. 2** where, it is seen that the higher the C_o , the quicker the adsorbent reached saturation. **Table 1** shows that q_e decreased with the increase in C_o . Total dye removal R (%) followed the same trend. The decrease in q_e and total dye removal from 15.982 mg/g for 25 mg/L to 7.14 mg/g for 50 mg/L was far less than from 7.14 mg/g for 50 mg/L to 1.57 mg/g for 100 mg/L. The reason for the trend was due to the fact that the active sites on the adsorbent were saturated at higher adsorbate concentrations (Njoku *et al.*, 2012).

3. 2. Effects of carbon bed height

The impact of removing metanil yellow from aqueous solution by carbon in a fixed-bed column by activated carbon from oil palm fruit mesocarp fibre at fixed C_o 25 mg/L and Q 8 mL/min at various H , is shown in **Fig. 3**. The breakthrough curves for H 4.1 and 6.3 cm are virtually the same. The two beds could not reach saturation in 360 min. Table 1 shows that the equilibrium adsorption capacity and total dye removal were maximum for H 4.1 cm. q_e and total removal increased from 9.407 to 15.982 mg/g and 8.04 – 27.32%, respectively for H 2.1-4.1 cm, but decreased from 15.982 to 5.97 mg/g and 27.32 – 15.39%, respectively (H 4.1-6.3 cm) as H increased. The decrease may be due to the splitting effect of flux concentration gradient between the adsorbate and adsorbent with the increase in H (or carbon mass) causing a decrease in the amount of dye adsorbed onto a unit mass of carbon (Pokordi and Kumar, 2006).

3. 3. Effect of flow rate

The effect of adsorbing metanil yellow on NATPAAC from oil palm fruit mesocarp fibre in a fixed-bed column at various Q 8, 16, and 26 mL/min, C_o 25 mg/L, and H 4.1cm, is shown in **Fig. 4**. The Figure shows that the higher the Q , the quicker the adsorbent reached the saturation. Table 1 shows that q_e decreased with the increase in Q ; similarly, total dye removal decreased with the increase in Q . This trend is attributed to the insufficient residence time of the solute in the column to allow for diffusion of the solute into the pores of the carbon (Salman and Hameed, 2011).

3. 4. Fixed-bed adsorption modeling

The time for breakthrough appearance and the shape of the breakthrough curve are important characteristics for determining the operation and dynamic response of an adsorption column. Again, successful design of an adsorption column requires prediction of the

concentration-time profile from the breakthrough curve for the effluent from the column. The Thomas and Yoon-Nelson models were applied in this work.

3. 5. Thomas kinetic model

Experimental data at fixed C_o , H and Q were analysed with the Thomas model. **Figs. 5-7** show the $\ln [(C_o/C_t) - 1]$ versus time plots. **Table 2** shows the Thomas parameters and correlation coefficient values. The Table shows that q_o increased with the increase in C_o , decrease in H , and decrease in Q . The correlation coefficient values ($R^2 > 0.85$) show that the experimental data fitted well to the Thomas model.

3. 6. Yoon-Nelson kinetic model

Yoon-Nelson model was applied to the experimental data with respect to C_o , H , and Q . A linear regression analysis was used on each set of experimental data to evaluate the Yoon-Nelson model parameters (see **Figs. 8 – 10**). **Table 3** shows these parameters and the respective R^2 values. The Table shows that q_o increased with the increase in C_o , decrease in H , and decrease in Q ; K_{YN} decreased with the increase in C_o , increase in H , and τ showed the same trend with q_o . The R^2 values, which are generally above 0.85, show that the experimental data fitted well to the Yoon-Nelson model.

4. CONCLUSION

Fixed-bed column adsorption on NATPAAC prepared from oil palm fruit mesocarp fibre was applied to remove metanil yellow from wastewater. The adsorption capacity of the carbon was evaluated at various C_o , H and Q . Experimental data showed optimum q_e as 15.982 mg/g. Thomas and Yoon-Nelson models simulated experimental data well based on R^2 values. Results show that the optimum C_o , H and Q were 25 mg/L, 4.1cm and 8mL/min respectively. The results also show that oil palm fruit mesocarp fibre activated carbon has potential as an adsorbent for acid dyes.

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Table 1. Experimental data for fixed-bed column adsorption of metanil yellow from aqueous solution on NATPAAC from oil palm fruit mesocarp fibre at various process parameters.

Q (mg/L)	H (cm)	Q (mL/min)	q _{tot} mg/g	q _e mg/g	Total removal (%)
25	4.1	8	63.928	15.982	27.32
50	4.1	8	28.56	7.14	6.11
100	4.1	8	6.28	1.57	0.67
25	2.1	8	18.814	9.407	8.04
25	6.3	8	35.82	5.97	15.39
*25	4.1	8	44.18	11.045	61.36
*25	4.1	16	39.34	9.835	27.32
*25	4.1	26	38.284	9.571	16.39

*Adsorption was for 240 min

Table 2. Thomas model parameters and correlation coefficient R² for fixed-bed column adsorption of metanil yellow on NATPAAC from oil palm fruit mesocarp fibres at various process conditions

C _o (mg/L)	H (cm)	Q (mL/min)	K _{Th} × 10 ⁻⁴ (mL/min mg)	q _o (mg/g)	R ²
25	4.1	8	1.88	0.944	0.9017
50	4.1	8	0.88	29.711	0.9012
100	4.1	8	0.25	324.06	0.8791
25	2.1	8	1.52	29.991	0.9121
25	4.1	8	2.12	7.029	0.8726
25	6.3	8	2.24	4.598	0.8549
*25	4.1	8	1.96	20.088	0.8782
*25	4.1	16	2.12	14.058	0.8549
*25	4.1	26	1.88	3.049	0.9019

*Adsorption was run for 240 min

Table 3. Yoon-Nelson model parameters for fixed-bed column adsorption of metanil yellow on NATPAAC from oil palm fruit mesocarp fibre at various adsorption conditions

C_o (mg/L)	H (cm)	Q (mL/min)	K_{YN} (min ⁻¹)	T (min)	q_{OYN} (mg/g)	R^2
25	4.1	8	0.0053	18.06	0.903	0.8495
50	4.1	8	0.0044	297.25	29.725	0.9109
100	4.1	8	0.0027	1490.78	298.156	0.903
25	2.1	8	0.0038	299.61	29.961	0.9114
25	4.1	8	0.0053	140.51	7.026	0.8548
25	6.3	8	0.0056	138.07	4.602	0.8728
*25	4.1	8	0.0049	403.63	20.182	0.8844
*25	4.1	16	0.0053	140.509	14.051	0.8548
*25	4.1	26	0.0047	18.638	3.029	0.9025

*Adsorption was run for 240 min

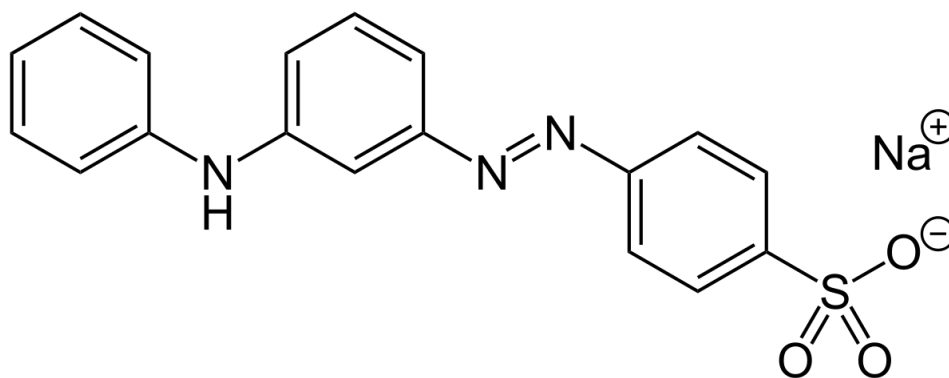


Fig. 1. Structure of metanil yellow

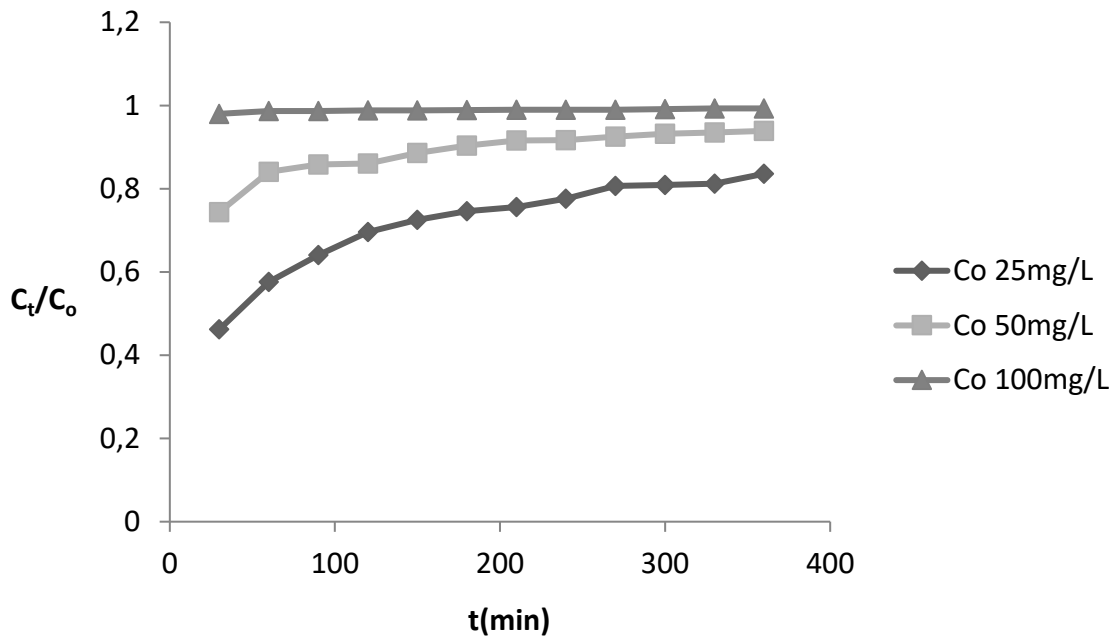


Fig. 2. Effect of dye concentration on the adsorption of metanil yellow on activated carbon from palm fibre

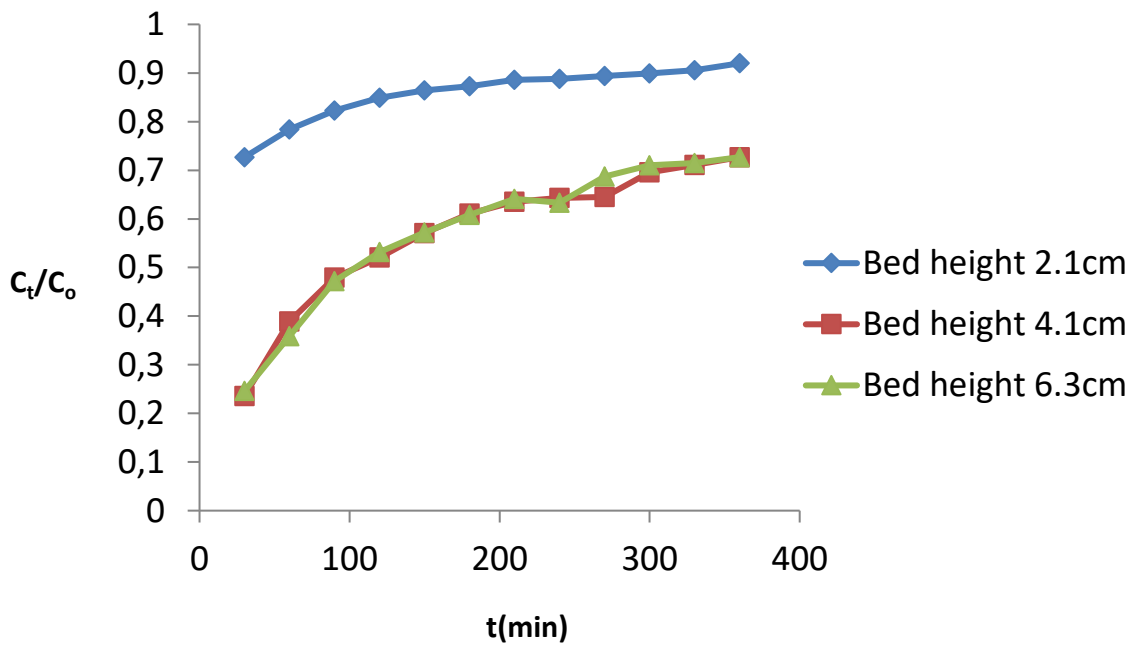


Fig. 3. Effect of bed height and the adsorption of metanil yellow on activated carbon from palm fibre

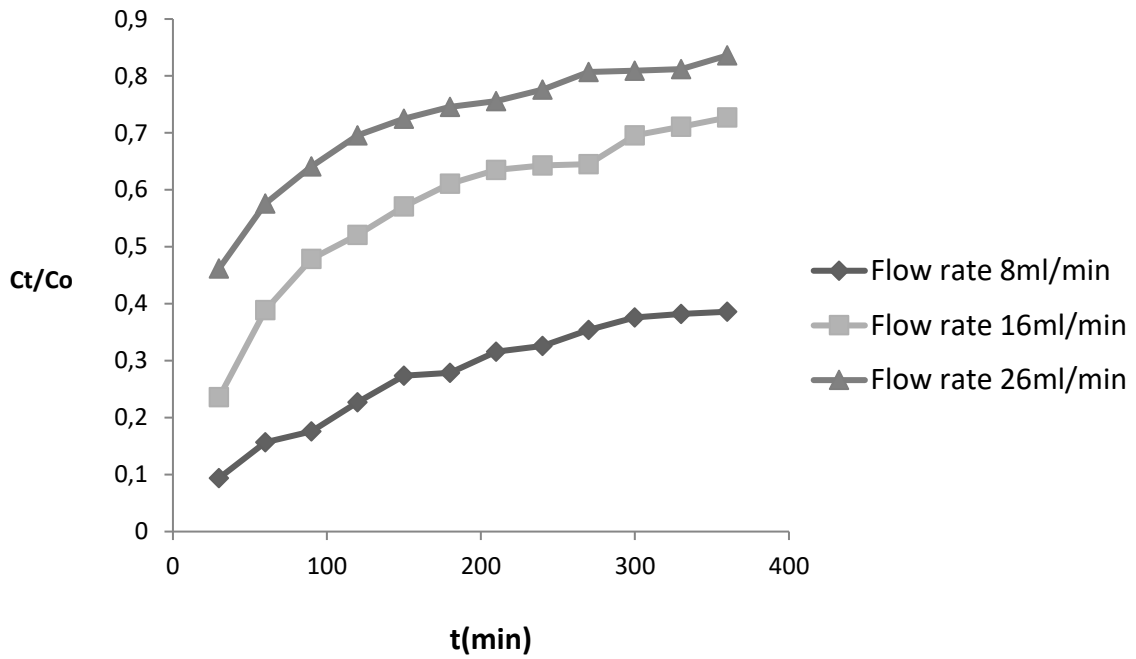


Fig. 4. Effect of flow rate on the adsorption of metanil yellow on activated carbon from palm fibre

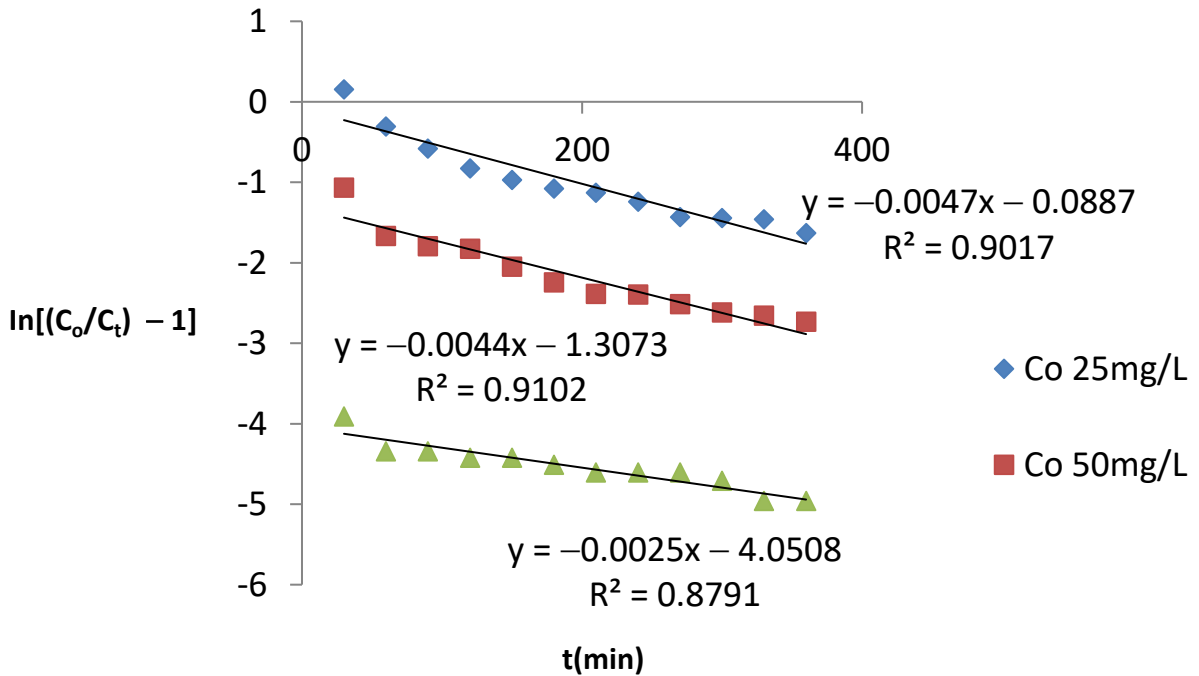


Fig. 5. Thomas model for the adsorption of metanil yellow on activated carbon from palm fibre at various initial concentrations.

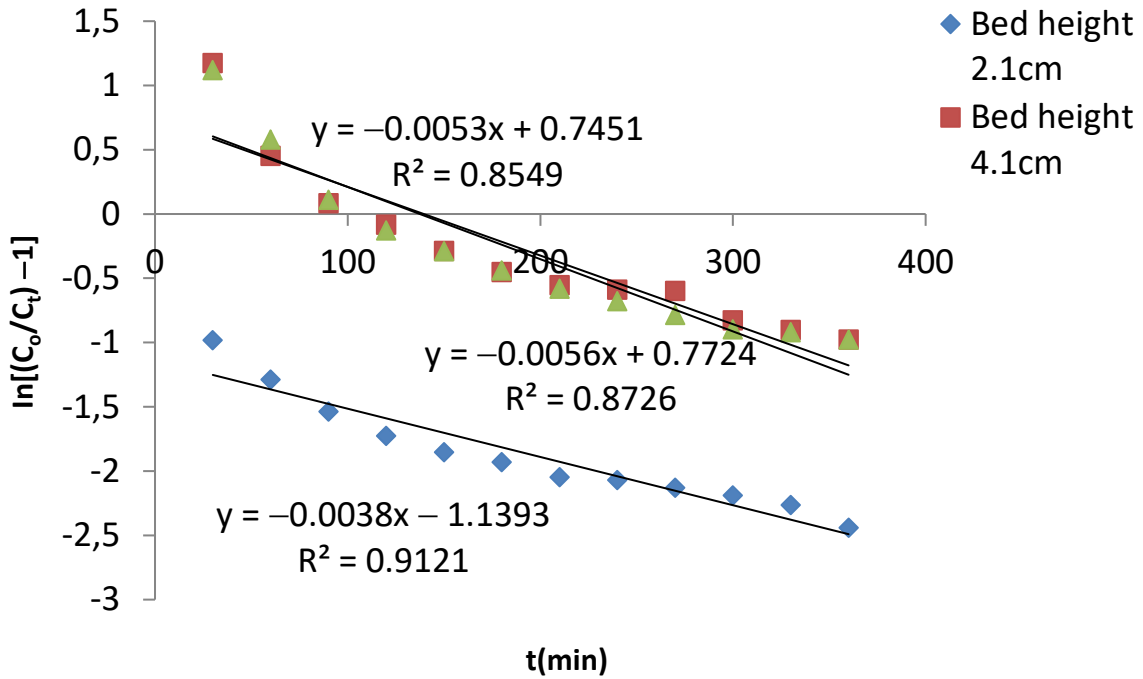


Fig. 6. Thomas model for the adsorption of metanil yellow on activated carbon from palm fibre at various bed height

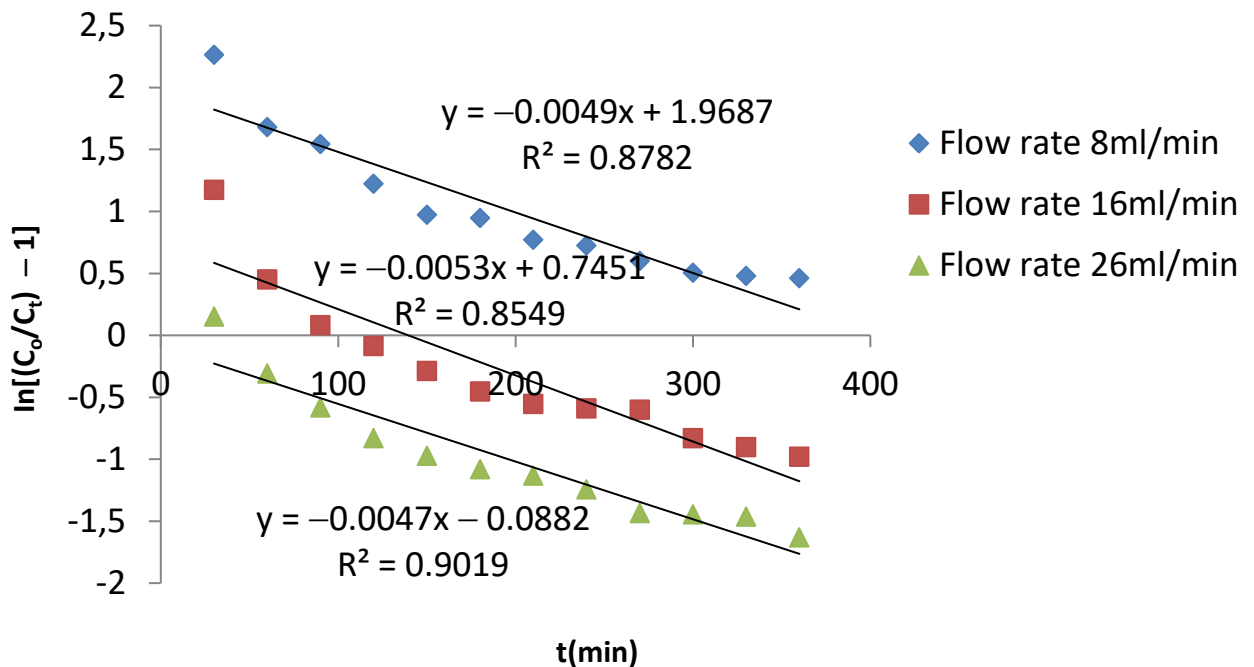


Fig. 7. Thomas model for the adsorption of metanil yellow on activated carbon from palm fibre at various flow rates.

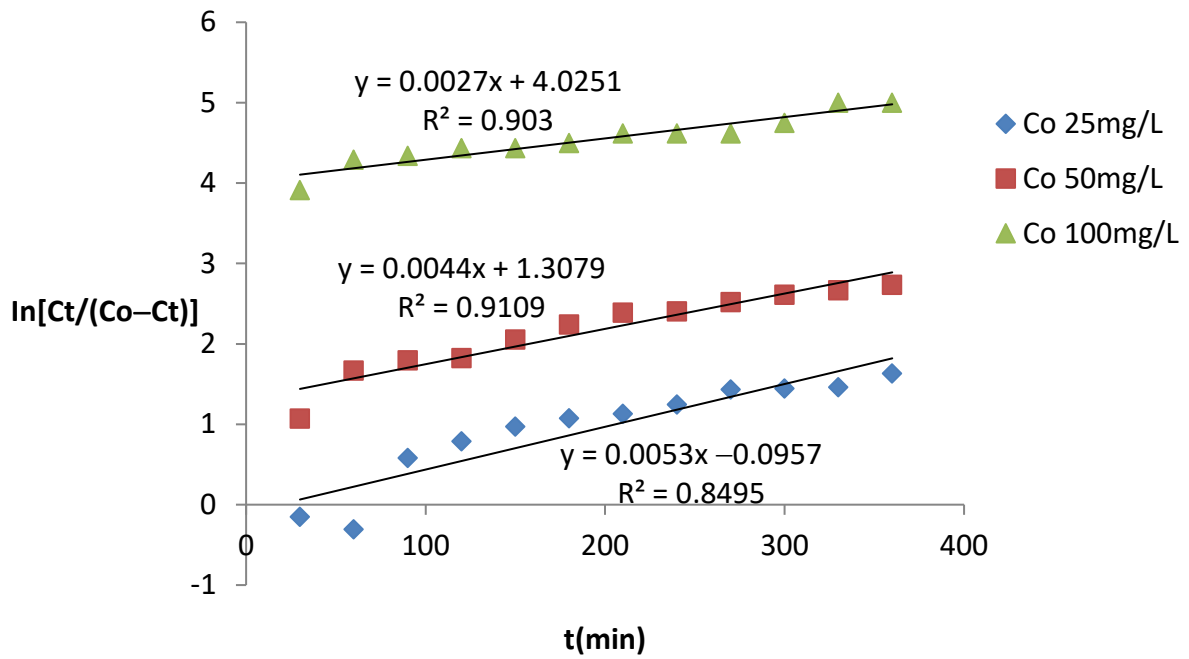


Fig. 8. Yoon-Nelson model plot for the adsorption of metanil yellow on palm fibre activated carbon at various initial concentrations.

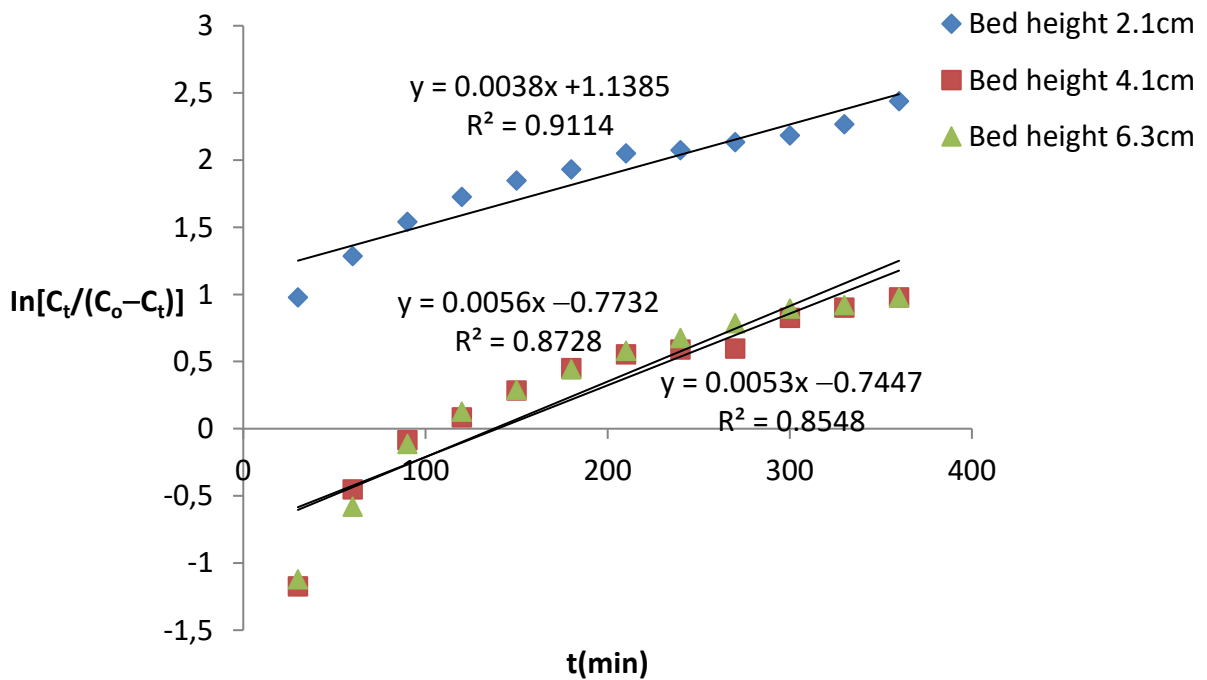


Fig. 9. Yoon-Nelson model plot for the adsorption of metanil yellow on activated palm fibre carbon at various bed heights.

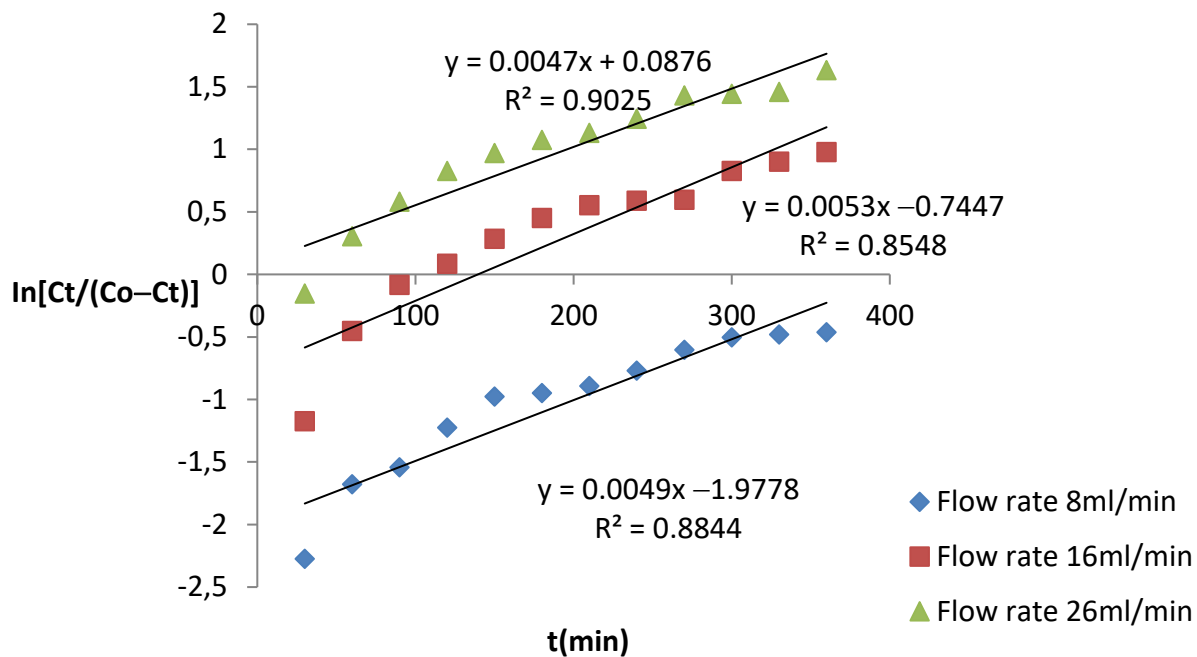


Fig. 10. Yoon-Nelson model plot for the adsorption of metanil yellow on activated palm fibre carbon at various flow rates.