

PRODUCTION AND CHARACTERIZATION OF ACTIVATED CHARCOAL FROM *BORASSUS AETHIOPIUM* SEED AND ITS APPLICATION ON DYE EFFLUENT

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ABSTRACT

Activated carbon was produced from *Borassus aethiopum* seeds through chemical activation using H_3PO_4 and alternative agents at 400–600°C for 20–60 minutes. Characterization showed carbonized samples (0.439 g/cm³ density, 73% carbon content) developed effective porosity (0.470 cm³/g pore volume) compared to raw seeds (0.531 g/cm³, 85.4% carbon). The material demonstrated excellent dye removal (84.4–99% efficiency) at 4–26 g dosage. Effluent analysis revealed significant improvements post-treatment: turbidity decreased from 33.26 to 1.02 NTU, conductivity from 1340 to 78.50 μ S/cm, and pH normalized from 12.50 to 6.00. COD and BOD were reduced from 139.60 to 38.30 mg/L and 78.30 to 11.64 mg/L, respectively, meeting WHO standards. FTIR analysis identified key functional groups (O-H at 3441 cm⁻¹, C=O at 1799.72 cm⁻¹) contributing to adsorption, which followed pseudo-second-order kinetics ($R^2 > 0.99$), indicating chemisorption dominance. SEM confirmed the development of porosity in activated samples versus non-porous raw seeds. *Borassus aethiopum* is an effective precursor for wastewater treatment with comprehensive characterization supporting its adsorption mechanism.

Keywords: Activated Carbon, *Borassus aethiopum*, Wastewater, Dye Removal, Adsorption, SEM.

INTRODUCTION

The textile industry is a major consumer of water, generating approximately 40–65 liters of effluent per kilogram of fabric produced (Manu and Chaudhari, 2002). A significant environmental challenge associated with this wastewater is the presence of synthetic dyes, which are highly visible, resistant to degradation, and often toxic or mutagenic (Weisburger, 2002). To address dye pollution, various physicochemical and biological treatment methods have been explored, including coagulation, oxidation, and microbial degradation (Slokar and Le Marechal, 1998; Robinson et al., 2001; Forgacs et al., 2004). While these techniques vary in efficacy, stricter environmental regulations have shifted focus toward greener and cleaner technologies (Hai et al., 2007). However, such methods often entail higher operational costs. Hybrid systems—combining multiple approaches—offer a promising solution by balancing efficiency and affordability.

Among hybrid strategies, activated carbon stands out due to its exceptional adsorption capacity, tunable porosity, and versatile surface chemistry (Marsh and Rodríguez-Reinoso, 2006). Traditionally, activated carbons are used as a standalone adsorbent for dye removal (Kannan and Sundaram, 2001); they also function as a catalyst or support material in advanced

treatment processes. Its surface oxygen groups, for instance, enhance catalytic activity (Rodríguez-Reinoso, 1998; Figueiredo and Pereira, 2009). The ability to engineer its physical and chemical properties further amplifies its potential for optimizing textile wastewater treatment.

In this work, the production and characterization of activated carbon from *Borassus aethiopum* for textile dye removal are reported. The activation process was optimized for maximum adsorption capacity. Furthermore, the evaluation of the material's performance in treating real textile effluent and adsorption mechanisms through kinetic modeling was elucidated.

MATERIALS AND METHODS

Sample Collection and Preparation

Mature seeds of *Borassus aethiopum* (African fan palm) were collected from multiple locations within Alkaleri Local Government Area (10°15'N, 10°20'E), Bauchi State, Nigeria. The collected seeds were immediately placed in high-density polyethylene (HDPE) bags (0.5 mm thickness) to prevent moisture absorption and contamination during transport. The washed sample was exposed to sunlight and dried for 3 days. The sun-dried samples were crushed into fine particles with a mechanical crusher to determine the size before carbonization. Dye wastewater was collected from the local pits at Maiduguri Bye-pass, Bauchi state. All chemicals and reagents used for this work were of analytical grades, and the major equipment used is: turbidity meter, UV-Visible Spectrophotometer (model 752N by Searchtech instruments), Muffle furnace, Scanning Electron Microscope (SEM) (Jeol Ltd with model JSM-17600F, USA) for characterization.

Preparation of carbonized adsorbent

The Carbonized adsorbent was produced according method described elsewhere (Sumrit et al., 2015). Briefly, 200 g of the sieved *Borassus aethiopum* seed was carbonized by placing it in an electric furnace at 500 °C and heating it for 30 minutes. After this, it was allowed to cool and labeled in an air-tight polyethylene bag.

Chemical activation of the carbonized adsorbent

Fifty grams (50 g) of carbonized carbon was impregnated with 150 mL of 0.5 M hydrochloric acid, placed in a furnace set at 500 °C, and heated for 60 minutes. Cold water was used to wash the activated carbon produced and further dried for 60 minutes at 110 °C in an oven till a constant weight of activated carbon was obtained (Sumrit et al., 2015).

The percentage yield was calculated as follows:

$$\text{Percentage Yield} = \frac{W_1 - W_2}{W_1} \times 100$$

Where,

W_1 = Weight of raw material

W_2 = Weight of Carbonized raw material

Characterization of activated carbonized adsorbent

Determination of the bulk density of carbonized adsorbent

A small quantity of charcoal carbonized at 400, 500, and 600 °C was taken and ground to powder; sieved and separately put into the density bottle and weighed. The weight and the volume of the density bottle were initially determined (Andi and Paulus, 2018). The bulk density was calculated using the following expression:

$$\text{Bulk density} = \frac{\text{mass of carbon sample}}{\text{volume}}$$

Determination of ash content of carbonized adsorbent

Ash content of the carbonized was determined as reported in literature (Rafie et al., 2013). Briefly, 3 g of the carbonized sample was weighed and heated in open crucibles at 750 °C for 1.5 hours. The crucible was then cooled and reweighed. This was calculated as:

$$\text{Ash percentage} = \frac{\text{Ash weight}}{\text{Oven dry weight}} \times 100$$

Determination of moisture content

One gram (1g) of the sample was weighed and heated in a watch glass at 105 °C for 2 hours. The heated crucible was cooled and re-weighed (Rafie et al., 2013)

$$\text{Moisture percentage} = \frac{\text{Loss in weight on drying (g)}}{\text{Initial weight (g)}} \times 100$$

Determination of the percentage of carbon

The percentage of carbon in the raw material was determined by removing the Ash percentage and Moisture percentage from 100 % (Anon, 2011).

$$\begin{aligned} \text{Percentage Carbon} \\ = 100 - \text{Percentage ash} \\ - \text{percentage moisture} \end{aligned}$$

Determination of Physicochemical Parameters of the Untreated and Treated Dye Effluent.

The physicochemical analysis of the dye effluent was carried out on the untreated and treated samples with the activated carbon (*Borassus aethiopum*). Some of the parameters, such as temperature, turbidity, total dissolved solids (TDS), conductivity, BOD, COD, and pH, were carried out immediately after sampling, while others were carried out at the laboratory (Coughlin, 2015).

Determination of pH of the dye effluent

After calibrating the pH meter, the measurement was carried out by rinsing the electrode with distilled water before it was dipped into a

50 ml beaker containing the test samples (treated and untreated) (Ramesh, 2017).

Temperature of dye effluent

Temperature was determined by dipping a mercury-in-glass thermometer calibrated in degrees centigrade (°C) into the three (3) samples, it was allowed to stand for some minutes before the readings were recorded (Das, 2011).

Turbidity of dye effluent

Turbidity was measured using a DR 2000 (HACH) spectrometer. Freshly sampled dye effluent was filled into a 25 ml sample cell, and another containing sampled water was analyzed. The knob of the spectrophotometer was turned to a programmed number of turbidity, which is 750, and wavelength 450mm. The distilled water was inserted first into the cell holder, and the lid (cover) was closed. The knob for zero was pressed to standardize the instrument. After that, the sample was inserted into the cell holder, and the red button was pressed, and the result was displayed on the display screen in formazine turbidity unit (FTU=NTU).

Total dissolved solids (TDS) of dye effluent

In determining TDS, 200ml of distilled water and 100 ml of dye effluent were poured into two separate beakers. The TDS/conductivity meter was switched on, and its sensor rod was dipped into the beaker containing distilled water, which gave a reading of 0.00 mg/litre TDS. After that, the sensor rod was dipped into the second beaker containing dye effluent, and the TDS/Conductivity meter then displayed the TDS value in mg/litre for both treated and untreated samples (Neumann and Fatula, 2016).

Electrical conductivity of dye effluent

The electrical conductivity of the treated and untreated dye effluent samples was determined by dipping the electrode inside a measured volume in the beakers containing the solutions. The conductivity of the untreated dye effluent was read (Neumann and Fatula, 2016). The procedure was repeated for the treated dye effluent.

Biological oxygen demand (BOD) of dye effluent

The biochemical oxygen demand was determined using standard methods (WHO, 2010). The treated dye effluent in the BOD bottle was thoroughly aerated, after which 10 ml of dye effluent was diluted with considerable distilled water. DO determination was carried out as described above. The bottle was sealed and incubated in the dark for 5 days at 20 °C (Deng et al., 2010). DO was determined again for the second time. The BOD is the difference between the two determined DO levels as indicated below:

DO = Dissolved oxygen found in the sample on the initial day (initial day may be termed day Zero).

1. Untreated dye effluent.

$$\text{BOD}_5, \text{ mg } 1^{-1} = \frac{D_0 - D_T}{P}$$

2. Treated dye effluent with activated carbon

$$\text{BOD}_5, \text{ mg } 1^{-1} = \frac{(D_0 - D_T) - f \times (B_0 - B_T)}{P}$$

Where;

D_0 = dissolved oxygen of diluted sample initial, mg/L at 20 °C.

D_T = dissolve oxygen of diluted sample after 5 days' incubation
 P = decimal volumetric fraction of sample used (volume of sample used/total volume of the diluted sample).

Bo = dissolve oxygen of dye effluent control initially, mg/L
 B_T = dissolve oxygen of dye effluent control after incubation, mg/L
 F = ratio of % dye effluent in diluted sample to % dye effluent in dye effluent control.

Chemical oxygen demand (COD) of dye effluent

COD is another measure of organic material contamination in dye effluent specified in mg/L. COD is the amount of dissolved oxygen required to cause chemical oxidation of the organic material in water. COD parameter was measured using COD Vials (COD 251-1500 mg/L, Merck Germany) (Agarwal *et al.*, 2006).

Adsorption Kinetic Investigation

Activated carbon (1000 mg) from *Borassus aethiopum* was mixed with 50 mL of dye effluent (50–125 mg/L) at 30°C with shaking. After 30 min, samples were filtered (Whatman paper), and dye concentration was measured via UV spectrophotometry (590 nm). The process was repeated at 50 mg/L with sampling at 20–100 min intervals.

Adsorption kinetic models

The pseudo-first order and second order models need to be tested to determine which model is in good agreement with the experimental adsorption capacity (q_e) value, thus suggesting which model the adsorption system follows.

Pseudo-first-order kinetic model

The Pseudo-first-order kinetic model was determined using the equation below:

$$\ln(q_e - q_t) = \ln q_e - k_1 t$$

Where q_e is the amount of adsorbate adsorbed at equilibrium, (mg/g), q_t is the amount of solute adsorbed per unit weight of adsorbent at time, (mg/g), k₁ is the rate constant of pseud-first order sorption (1/h). A plot of ln(q_e - q_t) versus t gives a straight line with slope of k₁ and intercept of ln q_e.

Pseudo-second-order kinetic model

The Pseudo-second-order equation was expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_{2e}} + \frac{1}{q_e} t$$

a plot of t/q_e versus t gives 1/q_e was determined.

RESULTS AND DISCUSSION

Effect of Carbonization on the Physical Properties of Uncarbonized and Carbonized *Borassu aethiopum*

The physical properties of uncarbonized and carbonized *Borassu aethiopum* are presented in Tables 1 and 2.

Table 1: Physical properties of the uncarbonized *Borassus aethiopum*

Physical properties	Uncarbonized <i>Borassus aethiopum</i>
Bulk density (g/cm ³)	0.531
Moisture content (wt%)	4.7
Ash content (wt%)	2.3
Percentage of carbon (%)	85.48
Percentage of yield (%)	73.00

Table 2: Physical properties of the carbonized *Borassus aethiopum*

Physical properties	Temperature (°C)	Carbonized <i>Borassus aethiopum</i>
Bulk density (g/cm ³)	500	0.439
Moisture content (wt%)	600	11.5
Ash content (wt %)	500	2
Percentage of carbon (%)	500	73.00
Percentage of yield (%)	600	72.87

Effect of Carbonization on Bulk Density

The bulk density measurements in Tables 1 and 2 showed predictable variations. The uncarbonized *Barrisu aethiopicum* sample had a bulk density of 0.531 g/cm³, while carbonization at higher temperatures reduced it to 0.439 g/cm³. This decrease aligns with previous studies by Putshaka and Adamu (2009), who observed a similar trend in carbonized leather waste, sawdust, and lignite. The reduction is likely due to the material's high carbon content. Other studies, such as those by Ekebafe *et al.* (2017) and Efeobokhan *et al.* (2019), also reported lower bulk densities in activated carbons from rubber seed shells and plantain peels, suggesting that high temperatures enhance micropore formation within the carbon structure. Differences in feedstock properties may explain variations across studies.

Effect of Carbonization on Moisture Content

The raw material initially had a moisture content of 4.7%, but after carbonization at 600°C (20–60 min), it increased to 11.5% in *Barassu aethiopum*-derived activated carbon (Maina and Anuka, 2014). At 650°C, moisture levels dropped to 0.09–4.20%. Anisuzzaman *et al.* (2015) found a similar range (2.20–2.91%) in activated carbon from *Typha orientalis* leaves, attributing the post-activation moisture gain to atmospheric adsorption.

Effect of Carbonization on Ash Content

The raw materials' ash content was 2.3%, decreasing to 2.0% after carbonization at 500°C (Tables 1 and 2). Extended carbonization further reduced ash content. In contrast, Putshaka and Adamu (2009) reported higher ash levels (4.34–4.44%) in leather waste-derived activated carbon. Maina and Anuka (2014) observed a decline from 2.01% to 0.75% in *Canarium schweinfurthii*-based carbon when processed at 650°C for 60 min. Generally, charcoal ash content ranges between 0.5% and 5%, depending on feedstock.

Effect of Carbonization on Fixed Carbon Content

Fixed carbon was calculated by subtracting ash and moisture percentages from 100%. The uncarbonized sample had 85.48% fixed carbon, which dropped to 73.00% after carbonization (Tables 1 and 2). Despite this decrease, the material remained suitable for activated carbon production. Maina and Anuka (2014) reported an increase from 80% to 99% in *Canarium schweinfurthii* seeds carbonized at 350–650°C for 60 minutes, as higher temperatures reduce ash, moisture, and volatile matter.

Effect of Carbonization on Percentage Yield

The yield reduced slightly from 73.00% at 500°C to 72.87% at 600°C (Table 2). This is mainly due to volatile matter release and minor moisture loss (Eric *et al.*, 2014). Anisuzzaman *et al.* (2015) found yields between 39.22% and 73.10% for *Typha orientalis* carbon, with higher yields linked to increased H₃PO₄ concentration. Conversely, Ariany *et al.* (2018) noted yield reductions from 50% to 35% (800–900°C, 120 min) in sugarcane bagasse and rambutan twig-derived carbon, indicating that higher temperatures and longer processing times decrease yield. Similarly, Maina and Anuka (2014) observed a drop from 86% to 65% when activation temperature rose from 350°C to 650°C after 60 minutes, reinforcing that volatile matter loss at elevated temperatures reduces yield.

Physicochemical parameters of a dye effluent

Table 3: Physicochemical parameters of a dye effluent before and after treatment with the synthesized activated carbon

Properties	Effluent before treatment	After treatment with activated carbon	Permissible limit for effluent discarded (WHO)
Colour	Black	Colourless	Black
Odour	Pungent	Odourless	Odourless
Electrical conductivity (μs/cm)	1340	78.50	100.0
Density (g/cm ³)	1.023	0.977	1.00
TDS (mgL ⁻¹)	802	47.00	2100
TSS (mgL ⁻¹)	0.235	0.0041	-
Turbidity (NTU)	33.26	1.02	5
COD	139.60	38.30	50mg/L
BOD	78.30	11.64	30mg/L
Temperature °C	30.40	30.10	Below 40
pH	12.50	6.00	6 - 9
TDS	-	Total Dissolved Solids	
TSS	-	Total Suspended Solids	
BOD	-	Biological Oxygen Demand	
COD	-	Chemical Oxygen Demand	

Electrical Conductivity (EC):

EC reflects ion concentration, with higher values indicating salinity. The untreated sample recorded 1340 μS/cm, exceeding WHO (2010) limits (100 μS/cm), while the treated effluent measured 78.50 μS/cm, within permissible levels. EC serves as a proxy for mineral content and pollution extent (Neumann and Fatula, 2016).

Total Dissolved Solids (TDS):

TDS measures inorganic salts and dissolved substances. High TDS levels (802 mg/L in untreated vs. 47 mg/L in treated) pose salinity risks in irrigation. The untreated sample surpassed WHO (2010) limits, necessitating treatment.

Total Suspended Solids (TSS):

TSS levels were 0.235 mg/L (untreated) and 0.004 mg/L (treated). Comparatively, Suriyapratha and Fulekar (2018) reported extreme TSS values (9140–21,220 mg/L), far exceeding WHO thresholds.

Turbidity:

Elevated solids increased turbidity (33.26 NTU untreated vs. 1.02 NTU treated), impairing water clarity and photosynthesis. The treated sample met the WHO's 5 NTU limit.

Chemical Oxygen Demand (COD):

COD indicates organic pollutant levels. The untreated effluent (136.60 mg/L) exceeded the WHO's 50 mg/L limit, while the treated effluent (38.30 mg/L) complied. High COD suggests toxicity and reduced dissolved oxygen (DO), endangering aquatic life (Yasar, 2012).

Biological Oxygen Demand (BOD):

BOD measures microbial oxygen consumption during organic matter decomposition. Untreated effluent (78.30 mg/L) surpassed permissible levels (<100–300 mg/L), whereas treated effluent (11.64 mg/L) was compliant. Elevated BOD correlates with lower DO, stressing aquatic ecosystems (Rahman, 2011).

Morphological and Compositional Modifications

The micrograph of the untreated and activated carbon from *Borassus aethiopum* are presented in Figures 4 and 5, respectively.



Figure 1: SEM Micrograph of uncarbonized Barassu aethopium seed (magnification = 1000)



Figure 2: SEM Micrograph of carbonized Barassu aethopium seed (magnification = 1000)

SEM micrographs revealed that raw *Borassus aethiopum* lacked porosity (Figure 1), whereas carbonized samples (Figure 2) exhibited a rough, porous morphology with crystalline structures.

These structural changes are attributed to acid activation and high-temperature carbonization, which create micropores and mesopores, as reported by Prakash *et al.* (2012). EDX analysis confirmed the dominance of carbon (**88.04 wt%**), with minor traces of nitrogen and silicon, corroborating findings by Isil (2016) for ACs used in sulfide removal. The high carbon content and developed porosity explain the material's superior adsorption capacity (Yagmur *et al.*, 2008).

Functional Group Dynamics

FTIR spectra identified critical functional groups influencing adsorption. The presence of **O-H (3441 cm⁻¹)** and **C=O (1799 cm⁻¹)** in acid-treated AC (Table 4) indicates oxygenated surface groups, which facilitate dye binding via hydrogen bonding and electrostatic interactions (Baccar *et al.*, 2012). The absence of these groups in uncarbonized samples (Table 6) underscores the role of chemical activation in introducing active sites, consistent with Solum *et al.* (1995).

Table 4: FTIR Analysis of carbonized sample of carbonized Barassu Aethiopium seed

Activate d Carbon	FTIR Band	Standard FTIR absorption band range (cm ⁻¹)	Interpretation of functional group
Hydroge n chloride acid- activate d carbon	3441. 00	00-3000	35 OH stretching of the hydrogen group
	2969.	30	Sp ³ C-H

96	00-2840	30	Sp ³ C-H
20	00-2840	33	OH stretch
24	00-2500	18	acid C=O stretch
38	00-1770		

Adsorption Kinetics

The adsorption process followed **pseudo-second-order kinetics** ($R^2 = 0.9694$), indicative of chemisorption (Ho and McKay, 1999). The poor fit of the pseudo-first-order model ($R^2 = 0.04887$) further supports this mechanism, as noted by Kumar *et al.* (2020) for methylene blue adsorption. The high regression coefficient of the second-order model suggests that dye removal is governed by surface complexation rather than diffusion (Khaled *et al.*, 2008).

Pseudo-first-order kinetic model

Figure 3 shows the linearized plot of pseudo pseudo-first-order model with the regression coefficient R^2 from the data obtained being 0.04887. Kumar *et al.* (2013) observed a regression coefficient value of 0.4998 adsorption of methylene blue onto activated carbon, and Liu *et al.* (2019) also observed a regression coefficient of 0.3984 adsorption of lead ions onto graphene oxide. The curve fitting plot of $\ln(q_e - q_t)$ versus t does not show good results for the entire adsorption period. Kumar *et al.* (2013) also observed linear plot of the adsorption of methylene blue does not fit well in the first order. The result is in agreement with the work of Bello *et al.* (2008), adsorption of methylene blue onto activated carbon derived from periwinkle shells, and also observed by Bello *et al.* (2012) whose adsorption is not well fitted, making a pseudo-first order using imperata cylindrical leaf powder activated carbon.

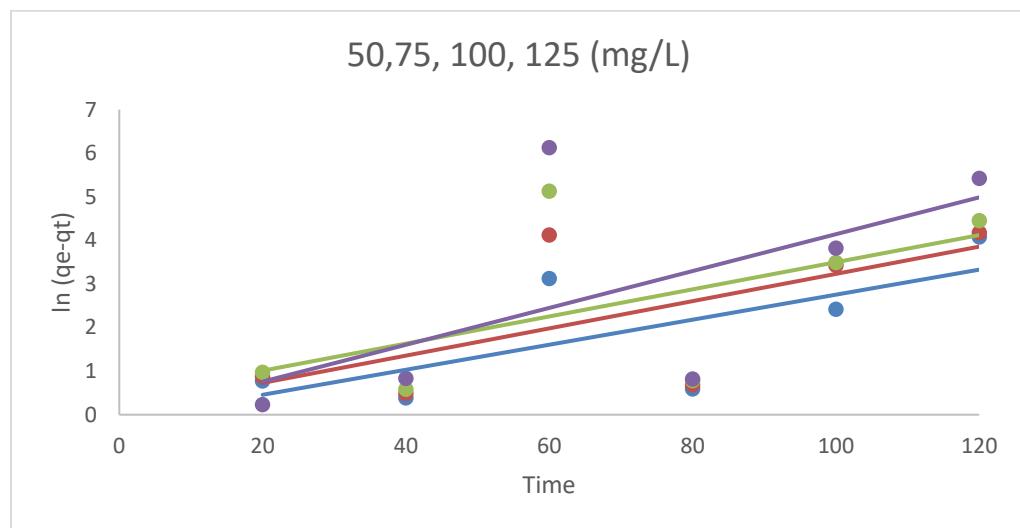


Figure 3: First-order plot of dye effluents adsorption

Pseudo-second order kinetic model

Figure 4 showed the linearized plot of pseudo pseudo-second order model with the regression coefficient R^2 from the data obtained, 0.9694. The models with the highest regression coefficient R^2 have been considered the best model to explain the kinetic model from the data obtained. The value of the regression

coefficient R^2 of the pseudo-second order model is higher than the pseudo-first order model, which shows that the pseudo-second order model is well fitted which indicating the absorption is limited by the chemisorption process. This is supported by Picking *et al.* (2011), who confirmed that pseudo-second order models as the most appropriate to fit the equilibrium experimental data due to a

higher value of coefficient of determination through error analysis compared to other adsorption isotherms, and also the plot gave a strong line for the dye concentration studied as shown. This confirmed the applicability of the pseudo-second order equation. This is in agreement with the work of Khaled *et al.* (2008), whose plot of t/q_t versus t gave a straight line for all the initial dye concentration removal of direct N blue using activated carbon from

orange peel.

Ahmad *et al.* (2014) discovered the regression coefficient R^2 value for the pseudo-second order kinetic model as 0.9842. The adsorption of RBBR dye onto PPAC followed the pseudo-second order kinetic model. This is due to greater competition for the adsorbent site at higher dye concentration (Ahmad *et al.* 2014).

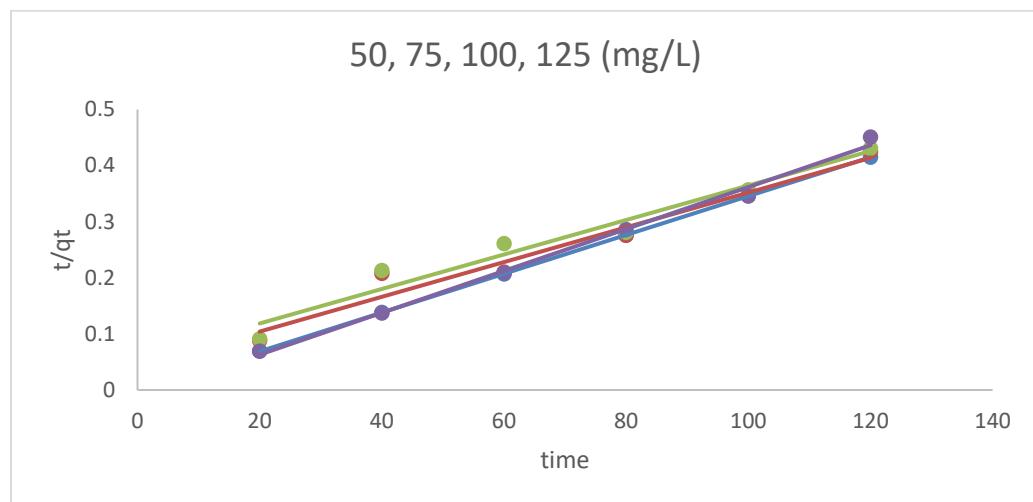


Figure 4: Second-order plot of dye effluents adsorption

Conclusion

This research work was able to produce activated carbon that is cheap and eco-friendly using organic reagents on *Borassus aethiopium*. Various physical parameters like moisture content, bulk density, ash content, carbon content, and percentage yield of the charcoal were determined.

In addition, percentage decolouration of both the treated and untreated samples, various physical and chemical analyses of dye effluent, such as electrical conductivity, density, TDS, TSS, temperature, colour, and odor, were also determined.

The surface morphology of the activated carbon was reported using a scanning electron microscope (SEM), and the functional groups of the activated carbon were determined using Fourier transform infrared spectroscopy FTIR. The kinetic model shows that the experimental data can be well fitted by the pseudo-second order rate.

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