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Full Length Research Article

ADSORPTIVE KINETIC MECHANISMS OF BROMOCRESOL GREEN DYE REMOVAL FROM WASTEWATER USING MODIFIED GROUNDNUT SHELL ADSORBENT

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ABSTRACT
This study explores the production and application of modified groundnut shell (MGNS) adsorbent to remove Bromocresol Green Dye (BCGD) from wastewater as an alternative to cost intensive wastewater treatment technologies. The adsorbent was characterized for physicochemical properties, and by employing Scanning Electron Microscope (SEM), Fourier Transform Infrared (FTIR) and Energy dispersive X-ray spectrometers (EDX) as characterization tools respectively. The effects of contact time on the percentage of dye recovery were evaluated. The kinetic data were fitted to kinetic models such as Brouers Weron Sotolongo-Coasta (BWS), Fractal Pseudo-second-order (FPSO), Pseudo-First-Order (PFO), Pseudo-second-order (PSO) models using a non-linear form of the equations. The results revealed that the biomass has a pH (6.60), moisture content (14.20) %, volatile matter, (10.20) %, Ash content (8.10) %, fixed carbon (65.50) %, bulk density (0.440) g/cm^3, surface area (690) m^2/g and particle size (250) µm. The adsorbent possesses high carbon content and a well-developed pore structure. The adsorbent percentage dye removal efficiency (% R) was time-dependent (30 min). The adsorbent has maximum percentage dye removal of 84% at the optimum time. The kinetic data that best described the removal of BCGD from wastewater was BWS (R^2 = 0.9644). Overall, the prepared adsorbent from MGNS was efficient, eco-friendly and economically viable in treating dye polluted wastewater, ensuring regulatory compliance and facilitating water reuse.

Keywords: Bromocresol Green, Compliance, Contact time, Dosage, Technologies, Wastewater

INTRODUCTION

Textile dyes and other industrial dyestuffs constitute one of the largest groups of organic compounds that represent an increasing environmental threat (Jabar *et al*., 2020; 2022; Olafadehan *et al*., 2022). Industries such as paper, textile, plastic, detergents, cosmetics, leather, pharmaceutical and food industries continually discharge into the environment effluents containing dyes and their breakdown products which are toxic to living organisms and ecosystem (Hameed *et al*., 2008; Giwa *et al*., 2015; Carneiro *et al*., 2022).

About 1–20 % of the total world production of dyes is lost during the dyeing process and is released as effluents (Munagapati *et al*., 2018; Mansour *et al*, 2020). The toxicity of some dyes even at very low concentrations may significantly affect aquatic life. Incidences of skin irritation, allergy, and cancer to humans may also result (Giwa *et al*., 2015). Bromocresol green dye (BCGD) ($C_{21}H_{14}Br_4O_5S$), 3,3-Bis(3,5-dibromo-4-hydroxyl-2-methylphenyl)-2,1λ6-benzoxathiole-1,1(3H)-dione, is a triarylmethane dye with three aromatic rings (two brominated) attached to a central carbon atom, while the sulfonic group provides the water-soluble character. The hydroxyl group participates in pH-dependent colour change, and the bromine atoms enhance its molecular reactivity and stability.

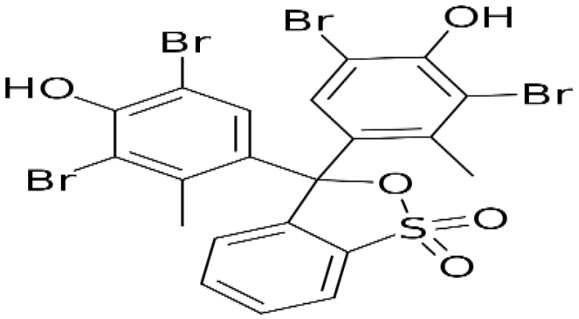

Figure 1: Molecular structure of BCGD

The groundnut shell (GNS) also known as peanut shell, is the protective outer casing of the groundnut (GN). It is a thin, hard, and fibrous material that surrounds the edible groundnut kernel (GNK) (Ajala and Ali, 2020). The GN is typically oval or elongated in shape and varies in colour from light tan to dark brown, depending on the variety of GN. It comprises the outer shell and inner seed coat. (Ajala and Ali, 2020). The GNS have various uses beyond their roles in protecting the groundnuts (GNs). They are commonly used as a good source of biomass fuel; they can be used as animal feed or as a component in livestock bedding material. In some cases, the shells are utilized for industrial purposes such as production of particle boards, mulch, and compost. GNS can also be used as an adsorbent in the adsorption phenomenon in the removal of dyes, heavy metals and other impurities from wastewater, and effluents including remediation of waste cooking oil (Onawumi *et al*., 2021). The present study focuses on the remediation potential of GNS adsorbent on BCGD polluted wastewater.

MATERIALS AND METHODS

Procurement and preparation of samples and reagents

Groundnut shell

GNS was obtained from Tombia market, Yenagoa, Bayelsa State, and was authenticated by Mr. Sunday Okpata (Voucher's number: FUO-081), Department of Biological Sciences, Federal University Otuoke, Bayelsa State, Nigeria. They were washed to get rid of

sand, dirt and other impurities, and then sun-dried to remove the excess moisture. They were further oven-dried at a temperature of 105 ^{o}C until a constant weight was achieved. The oven-dried GNS were subjected to further crushing, after which they were ground to the desired particle size according to our previous works (Onawumi *et al*., 2021; Sangoremi *et al*., 2024). The modification was done according to the method described by Bello *et al*. (2017) with slight modifications, and the resultant product is referred to as modified groundnut shell (MGNS) adsorbent. The modification was achieved by increasing the molarity of phosphoric acid (H_3PO_4) from 0.3 M to 0.5 M. An accurately weighed 14.0 g of crude sample of GNS was placed into Erlenmeyer flask containing 250 cm^3 of 0.5 M phosphoric acid. The substance of the container was entirely blended and warmed on a hot plate until a thick paste was formed. The paste of GNS was moved into a crucible which was set in a furnace and warmed at 500 ^{o}C for 60 minutes. The sample was allowed to cool and afterward washed severally with distilled water to a pH of 6.60, and thereafter, oven dried at 105 ^{o}C for 5 hours and the adsorbents were put an air tight container for usage (Bello *et al*., 2017).

Characterization of Groundnut Shell Adsorbent

The Scanning Electron Microscope (SEM) of GNS adsorbent was taken with a ThermoFisher Scientific (Axia ChemiSEM, 120 x 120 mm^2 5-axis motorized eucentric, USA). The surface of the MGNS was studied with the microscope operated at 10.0 kV. The samples were coated with a 10 nm thick layer of gold.

Fourier Transform Infrared Spectrometer (FTIR) (Thermo Fisher Scientific, Nicolet iS50, USA) technique of analysis was employed to study the functional groups in MGNS adsorbent. The infrared spectra of MGNS were obtained by using MGNS mixed with potassium bromides at ratio 1;100 in a mortar and pestle. The mixture was taken in a disc of specific dimension to form pellet by pressing with a handpress machine, placed on the sample holder of IR spectrometer (Agilent Technologies, 4100 ExoScan, California, USA) operated at spectra range 4000 – 400 cm^{-1}.

The physicochemical properties and proximate composition of MGNS adsorbent that were determined include: pH, moisture content (MC), volatile matter (VM), ash content (AC), fixed carbon (FC), bulk density (BD), surface area (SA), particle size (PS) by employing the methods described by Onawumi *et al*. (2021), ASTMD-3838-80, and Sangoremi *et al*. (2024).

Bromocresol Green Dye

Furthermore, BCGD and orthophosphoric acid (H_3PO_4) were Analytical grade reagents procured from Sigma Aldrich Chemical, Germany. BCGD (1000 mg) was accurately weighed into 250 ml conical flask, and small quantity of distilled water was added and stirred continuously for total dissolution. The dissolved dye solution was transferred into 1000 cm^3 standard volumetric flask and carefully made up to mark with distilled water. The aqueous BCRD solution was standardized on a UV-visible spectrophotometer (Agilent Technologies, Agilent 8453, California, USA). The BCRD wavelength at maximum (λ_{max}) was found to be 624 nm, and was used to determine the absorbance of the serially diluted solutions of dye prepared from the stock solutions (5, 10, 15, 20 25 mg/L). In addition, the absorbance of the BCRD effluent after the adsorption processes was measured to provide means of evaluating the percentage dye removal/uptake (% R) by the adsorbent at a particular time and at equilibrium.

Effect of contact time on the adsorption process

A batch adsorption study was carried out on the influence of contact on BCGD removal using 25 ml dye solution in a 100 ml conical flask placed on a water bath shaker at a shaking speed of 120 rpm. The adsorption process was carried out at temperature range (303 – 343 K) at 10 K interval. The initial dye concentration was 20 mg/l, 0.4 g adsorbent dosage, and the contact time was varied from (10 to 50 min) at 10 min interval. After the adsorption experiments, the adsorbent was separated from dye effluent by centrifugation using centrifuge at room temperature at 4000 rpm for 15 min.

The absorbance of aliquot part of dye effluent was read on UV-visible spectrophotometer and the concentration was interpolated from the working graph. Further, the percentage of BCGD adsorbed on the surface of GNS was determined according to Jabar *et al*. (2020) as shown in equation 1.

$$\% \boldsymbol{R} = \frac{(Co-Ce)100}{C_o} \quad (1)$$

The amount of BCGD adsorbed per unit weight of GNS was calculated as shown in equation 2 and 3 respectively:

$$q_e = \frac{(Co-Ce)V}{W} \quad (2)$$

$$q_t = \frac{(Co-Ct)V}{W} \quad (3)$$

% R = Percentage BCGD removed
C_o = Initial dye concentration (mg/mg)
C_e = Equilibrium dye concentration (mg/g)
C_t = Concentration at time (t)
V = Volume of dye solution (L)
W = Weight of the adsorbent (g)

Adsorption kinetic Models

A detailed design of a wastewater treatment plant using the adsorption method entails precise data on the rate of removal of contaminants from wastewater. Providing this information requires treatment of data from the influence of time on the adsorption process. In the present work, the data obtained from the effect of contact time on the amount of BCGD adsorbed on the surface of GNS were modeled by Brouers Weron Sotolongo-Costa (BWS), Fractal Pseudo-second-order (FPSO), Pseudo-first-order (PFO) and Psuedo-second-order (PSO) kinetic models using a non-linear form of the equations.

Brouers Weron Sotolongo

The equation for the Brouers Sotolongo kinetic model is given as from equation 4.

$$q_t = q_e(1 - ne^{(-(\frac{t}{\tau})^{\alpha})}) \quad (4)$$

τ = time necessary to adsorbed half of the equilibrium quantity (min).
t = time (min)
α = time variation of rate constant
n = fraction order of adsorption
q_e = amount of adsorbent adsorbed at equilibrium (mg/g)
q_t = amount of adsorbent adsorbed at time t (mg/g)

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Fractal pseudo-second order

The general expression for non-linear Fractal pseudo-second-order (FPSO) is given as:

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

Where:

K_F = coefficient of fractal pseudo-second-order (mg/gmin^{-1})

q_t = quantity of adsorbate adsorbed by the adsorbent at time t (mg/g)

q_e= amount of adsorbate adsorbed on adsorbent surface at equilibrium

q_t = amount of adsorbate adsorbed at any time

Pseudo-first order

For Pseudo first order nonlinear equations 6 and 7;

$$\frac{d_{qt}}{dt} = K_1(q_e - q_t) \quad (6)$$

$$q_t = q_e(1-e^{-K_t t}) \quad (7)$$

Pseudo-second order

Pseudo second order nonlinear equation given in equations 8 and 9;

$$\frac{d_{qt}}{dt} = k_2(q_e - q_t)^2 \quad (8)$$

$$q_t = \frac{k_2 q_e^2 t}{1+k_2 q_e t} \quad (9)$$

Software application on adsorption models.

To enhance the plan of an adsorption framework for the removal of adsorbates, it is essential to lay out the most fitting correlation for the adsorption balance curve. KyPlot® version 2.0 programming with non-linear numerical rendition of the kinetic model was utilized. The product utilizes the Semi Newton (least square) enhancement apparatus for fitting data to models. The coefficient of determination (R^2), standardized Chi square error (x^2) and the sum of square of error (SSE) were utilized to decide the model that best portrayed both the equilibrium and kinetic information for the different adsorbents (Unuabonah *et al*., 2017). The numerical conditions for x^2 and SSE (Olafadehan *et al*., 2022) are:

Normalized Chi square error test,

$$x^2 = \sum_{K=1}^{N} \left| \frac{(q_k(\exp) - q_k(pred)^2}{\sum_{k=1}^{Ne}(q_{k,\,pred} - q_e)^2} \right| \quad (10)$$

Coefficient of determination

$$R^2 = 1 - \frac{\sum_{k=1}^{Ne}(q_{k,\exp} - q_{k,pred})^2}{\sum_{k=1}^{Ne}(q_{k,pred} - q_e)^2} \quad (11)$$

RESULTS AND DISCUSSION

Table 1, presented the physicochemical properties of the prepared biosorbent under consideration with the following outcomes: pH (6.60), moisture content (14.20) %, volatile matter, (10.20) %, Ash content (8.10) %, fixed carbon (65.50) %, bulk density (0.440) g/cm^3, surface area (690) m^2/g and particle size (250) µm, they all conform to those reported in the literature (Ajala and Ali, 2020; Abdullahi *et al*., 2022), and as well in agreement with quality threshold value of the adsorbents as recommended by National Industrial Standard of Indonesia (SII) No. 0258-79, and National Standard of Indonesia (SNI) No. 06-3730-1995 (Table 2).

Table 1: Physicochemical properties of modified groundnut shell adsorbent

S/No	Parameters	Mean
1	pH	6.60
2	Moisture content (%)	14.20
3	Volatile matter (%)	10.20
4	Ash content (%)	8.10
5	Fixed Carbon (%)	65.50
6	Bulk density (g/cm^3)	0.440
7	Surface area (m^2/g)	690.00
8	Particle size (µm)	250.00

Table 2: Quality threshold standard for activated carbon; Sangoremi *et al*. (2024)

Parameter	SII No. 02587-79	SNI 06-3730-1995
Moisture content (%)	Maximum 10	Maximum 15
Ash content (%)	Maximum 2.5	Maximum 10
Volatile matter (%)	Maximum 15	Maximum 25
Iodine number (mg/g)	Minimum 200	Minimum 750
Fixed Carbon (%)	Maximum -	Maximum 65

Figure 2 shows the FTIR spectra of MGNS adsorbent. The characteristic functional groups in MGNS adsorbent were identified from FTIR spectra. The peak at 3471 cm^{-1} indicates the presence of an O-H stretching vibration of phenol or alcohols in lignin and cellulose of MGNS adsorbent. Other peaks in the spectrum of MGNS adsorbent are 2773.73, 2000.25, 1637.62, 1172.76 and 935.51 cm^{-1}. These are due to N-H, C≡C, C=C (C=O), C-O and C-H stretching respectively (Nandiyanto *et al*., 2019; Sangoremi *et al*., 2024).

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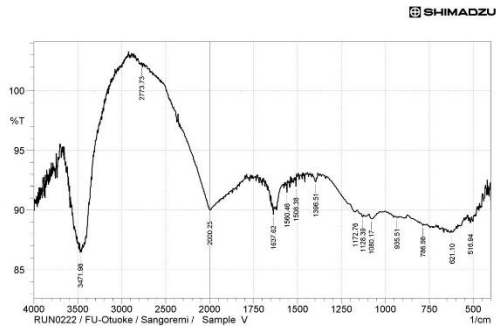

Figure 2: FTIR spectra of modified groundnut shell

Figure 3 shows the Scanning Electron Micrograph of the MGNS adsorbent. The SEM image of MGNS adsorbent reveals the presence of holes on its surface. These cavities are available pores at the surface, where BCGD molecules are captured from aqueous solution. The captured dye molecules traveled to fill the available pores on MGNS by diffusion of BCGD molecules from the aqueous solution to the MGNS adsorbent surface through the boundary layer. This was followed by migration of dye molecules from the adsorbent surface to the inner pores and finally adsorbed at the available vacant active sites on its surface. The adsorption of BCGD on the surface of MGNS might be physical adsorption (physisorption), through mechanical adhesion of adsorbates on adsorbent. This agrees with observations made by other researchers (Unuabonah *et al*., 2017; Jabar *et al*., 2020).

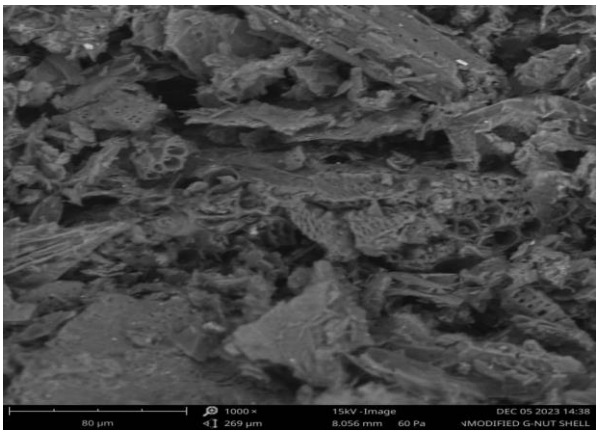

Figure 3: SEM micrograph of Modified Groundnut Shell

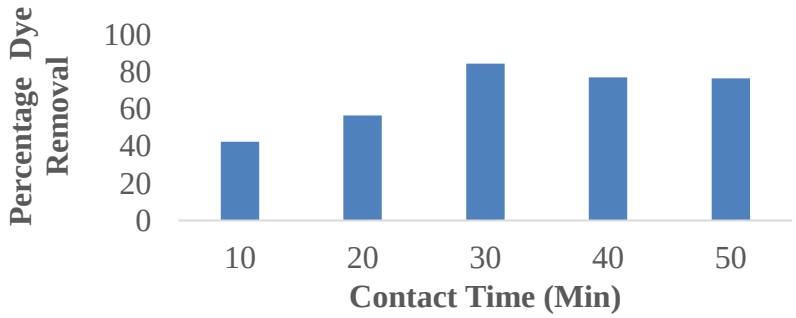

Figure 4: Graph of Relationship between percentage BCGD removal and contact time onto MGNS

Effect of contact time on dye removal and dye-uptake

The quantity of dye removed from the aqueous solution by MSC increased as the time of adsorption increased up to 30 minutes of the adsorption process (Figure 4). After 30 minutes of adsorbate adsorbent interaction, the percentage removal of BCGD declined marginally, this was a sign that the optimum time of adsorption is 30 minutes. This observation agreed with Jabar *et al*. (2020), on the kinetics and mechanisms of congo-red dye removal from aqueous solution using an activated Moringa oleifera seed coat as an adsorbent. The rapid increase in BCGD removal at the initial stage up to 30 min might be because of availability of active adsorption vacant sites on the surface of MGNS adsorbent. After 30 minutes of adsorption process, the active vacant sites got filled up by BCGD molecules. This might have resulted in repulsive force between the dye molecules on MGNS and those in solution (Jabar *et al*., 2020, 2021, 2022). The repulsive force might result into reduction in quantity of dye adsorbed after 30 minutes. This observation is in line with the findings of Jabar *et al*. (2022), when African almond (*Terminalia catappah*) leaves biochar prepared through pyrolysis using H_3PO_4 was used as chemical activation for sequestration of methylene blue dye. The same trend of adsorbate-adsorbent interaction was observed on the effects of contact time on BCGD removal from aqueous solution as seen in (Figure 4).

Adsorption Studies

Kinetic Investigation

Figure 5 reveals the various kinetic models used for the experimental data fittings, which include: Brouers Weron Sotolongo-Coasta, (BWS), Fractal Pseudo-second-order (FPSO), Pseudo-first-order (PFO), Pseudo-second-order (PSO) kinetic models respectively. BWS kinetic model best described the adsorption process with the coefficient of correlation (R^2 = 0.9646). Additionally, BWS described a very complex adsorption system and provided a better fit of experimental data than other kinetic models based on Normalized Chi square Error (0.0177) and Sum Square of Error (0.0059) values obtained from the non-linear regression modeling of experimental data from the kinetic models. The n parameter in the BWS model predicts a first order rate uptake of BCGD on the prepared MGNS adsorbent. The BWS kinetic model via a complex mechanism including electrostatic and weak Van der Waals forces (Unuabonah *et al*., 2017). Judging from BWS kinetic model data, the $t_{1/2}$ (min) for the adsorption of BCGD onto GNS adsorbent is in the increasing order which connotes that the adsorbent has a better rate of BCGD uptake (Unuabonah *et al*., 2017). The result was also established from PFO and PSO kinetic models where the general rate of BCGD uptake improved tremendously. Using the Normalized Chi Square Error (NCSE) and Sum Square of Error (SSE) models for determining the best model for the rate of uptake of BCGD adsorption onto MGNS adsorbents, it is observed that BWS kinetic model best describes the kinetic data (Unuabonah *et al*., 2017; Jabar *et al*., 2020). This implies that the adsorption of BCGD onto the MGNS adsorbent involves a complex adsorption phenomenon which may include simultaneous chemical interaction between the solute and chemical interaction between the dye molecules and chemical functional groups on the surface of the adsorbent via electrostatic interactions, Van der Waals, hydrogen bonding, complexation and ligand exchange (Unuabonah *et al*., 2017). This might also suggest that the surface nature of the adsorbent is heterogeneous.

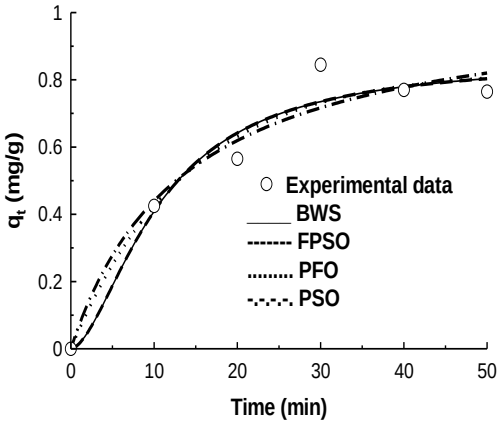

Figure 5: Kinetic model of BCGD adsorption onto MGNS adsorbent

Conclusion

The prepared adsorbent possesses high carbon contents, low inorganic contents, high surface area, and heterogenous pore structures that make a viable precursor for the removal of BCGD from aqueous solution. The dye removal from the wastewater was time-dependent with maximum removal efficiency of 84 % within 30 minutes. The kinetic model that best described the removal of BCGD from the wastewater was Brouers Weron Sotolongo-Coasta kinetic model with a coefficient of determination of (R^2 = 0.9644). The prepared adsorbent was efficient in remediating the dye polluted wastewater to useable status.

Acknowledgements

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Conflict of interest

The authors declare that there is no known conflict of interest as regard this work.

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