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Equilibrium Isotherm Studies of Bromocresol Green Dye Removal from Wastewater Using Modified Coconut Shell Adsorbent

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Abstract: ***This study examines the synthesis and application of modified coconut shell (MCNS) adsorbent for the removal of Bromocresol Green Dye (BCGD) from wastewater as an alternative to cost intensive wastewater treatment techniques. The adsorbent was characterized for physicochemical properties, and also by utilizing Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectrometer (FTIR) and Energy Dispersive X-ray spectrometer (EDX). The effects of adsorbent dosage on percentage dye recovery were assessed. Isotherm data were fitted to isotherm models such as Brouers Weron Sotolongo-Coasta (BWS), Freundlich, Jovanovich, Langmuir and Sips by employing non-linear model equations. The outcomes uncovered that the biomass has a pH (7.10 ± 0.101), moisture content (3.50 ± 0.110) %, volatile matter, (9.00 ± 0.012) %, ash content (15.70 ± 0.111) %, fixed carbon (71.80 ± 0.001) %, bulk density (0.769 ± 0.000) g/cm^3, surface area (1120.00 ± 0.000) m^2/g and particle size (300.00 ± 0.000) µm. The adsorbent has high carbon content and a well-developed pore structure. The adsorbent possesses high carbon content and a well-developed pore structure. The adsorbent percentage dye removal efficiency (% R) was dosage-dependent. The adsorbent has maximum percentage dye removal of 95.5 % at the optimum dosage (0.4 g). The equilibrium isotherm data that fairly described the removal of BCGD from wastewater was Freundlich isotherm model with coefficient of determination (R^2 = 0.8999), which suggests that the dye removal from the wastewater was multilayer adsorption on the heterogeneous surface which is more of physiosorption rather than chemisorption. By and large, the preparedadsorbent from MCNS was proficient, eco-friendly and economical in the treatment ofdye contaminated wastewater, ensuring legislative complianceand ease water reuse.***

Keywords: Wastewater, Isotherm, contaminated, eco-friendly, physisorption, chemisorption

I. INTRODUCTION

Textile dyes and other industrial dyestuffs constitute one of the largest groups of organic compounds that embody 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 release into the environment effluents encompassing dyes and their breakdown products which are lethal 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 implicitly affect aquatic life. Occurrences 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 increase its molecular reactivity and stability.

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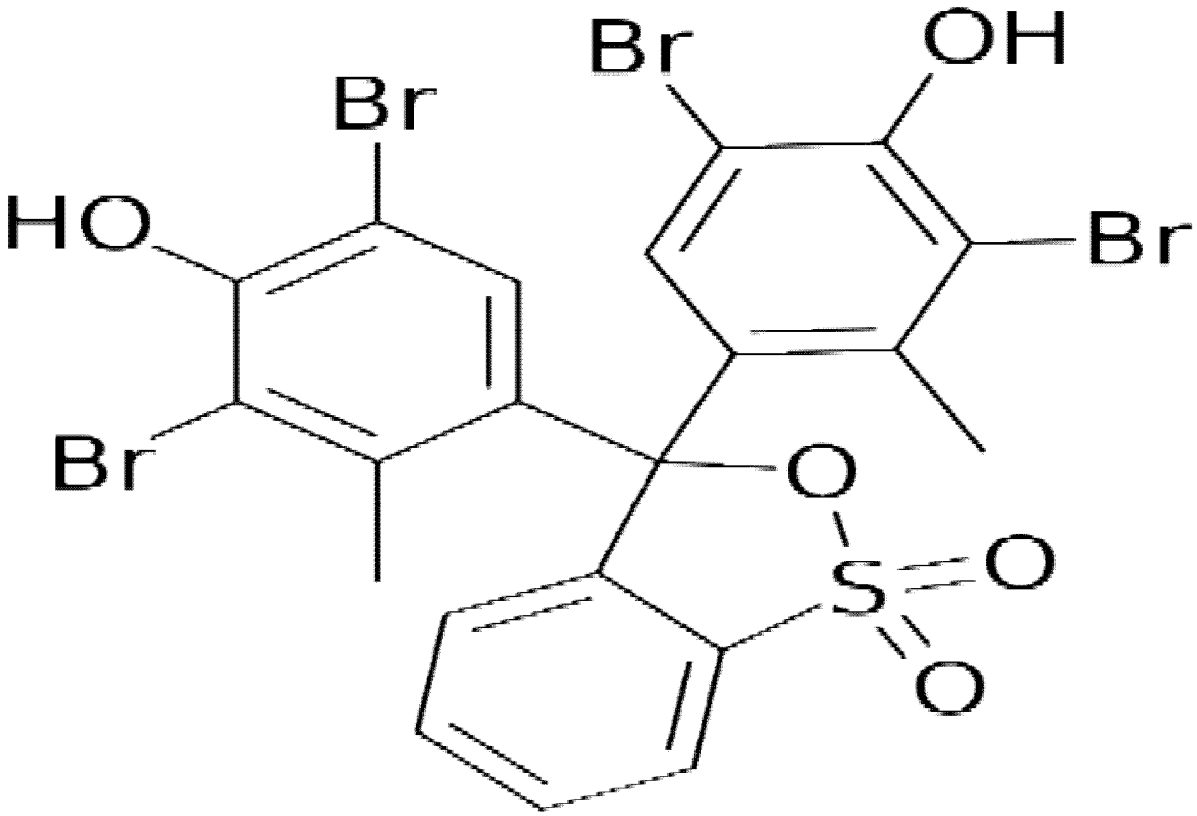


Figure 1: Molecular structure of BCGD

The coconut shell (CNS) is the hard, outermost layer that surrounds the inner flesh of a coconut. It is a natural protective covering that serves to safeguard the inner contents of the coconut, including the water (known as coconut water) (Usman *et al*., 2020).

The CNS is exceptionally hard and durable. It is one of the hardest natural materials and has a high compressive strength, enabling it to withstand external forces and protect the inner contents (Usman *et al*., 2020; Adewuyi and Olabanji, 2022). Coconut shells (CNSs) are generally oval or round in shape, with a slightly elongated form. They can vary in size and dimensions but typically have a diameter of around 15-20 cm (6 – 8 inches) (Usman *et al*., 2020). The outer surface of CNS is typically brown or tan in colour. It has a rough, fibrous texture with natural irregularities and grooves. The inner surface, in contrast, is smoothen and often exhibits a lighter colour (Sujiono*et al*., 2022). The CNS consists of several distinct layers. The outermost layer is the exocarp, which is a thick, fibrous layer that provides strength and rigidity. The innermost layer is the endocarp, which directly encases the coconut meat (Yasdi*et al*., 2021).

The CNS contains small pores and openings which allows for gas exchange and facilitate the transportation of water and nutrients. These pores also contribute to the unique acoustic properties of the coconut, making it to resonate (Sujiono*et al*., 2022). The CNS structure is well-suited for the environment in which coconut grow. It helps protect the inner contents from physical damage, moisture loss, and external threats such as insects or microbial attacks. The shell also acts as a thermal insulator, helping to regulate the temperature of the coconut meat (Usman *et al*., 2020; Adewuyi and Olabanji, 2022). Its constituents include lignin (36.15 %), cellulose (33.61 %), pentosane (29.27 %), ash (0.61 %), dry basis. The other constituents are hemicellulose and extractives. These extractives contain tannins, pectins, with polyphenolic, carboxyl and hydroxyl groups (Okafor *et al*., 2012). The lignin and cellulose in the coir afford its adsorptive properties (Okafor *et al*., 2012). Overall, CNS is a remarkable natural structure that combines strength, durability, and protection. Its unique properties make it useful for various applications such as crafts, utensils, activated carbon production, and as a raw material for creating sustainable products (Oribayo *et al*., 2020; Ambarsari*et al*., 2023; Bai *et al*., 2023). The CNS adsorbent exhibits a significant adsorption capacity for a wide range of substances, including organic compounds, heavy metals, dyes, and other pollutants.

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The adsorption process occurs through various mechanisms, such as physical adsorption, chemical adsorption, and ion exchange (Song *et al*., 2014; Oribayo *et al*., 2020; Usman *et al*., 2020; Yasdi*et al*., 2021; Adewuyi and Olabanji, 2022; Ambarsari*et al*., 2023; Bai *et al*., 2023).

II. MATERIALS AND METHODS

A. Procurement and preparation of samples and reagents

1) Coconut shell

CNSwas obtained from Otuoke market, Ogbia Local Government, Bayelsa State, and was authenticated by Mr. Sunday Okpata (Voucher's number: FUO-083), 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-driedCNS were subjected to further crushing, after which they were groundto the desired particle size according to our previous works (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 coconut shell (MCNS) adsorbent. The modification was achieved byincreasing 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 CNS 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 CNS was moved into a crucible which was set in a furnace and warmed at 500 ^{o}C for 60 minutes. The sample wasallowed 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).

2) Characterization of Coconut Shell Adsorbent

The Scanning Electron Microscope (SEM) of MCNS adsorbent was taken with a ThermoFisher Scientific (Axia ChemiSEM, 120 x 120 mm^2 5-axis motorized eucentric, USA). The surface of the MCNS was considered with the microscope run 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 MCNS adsorbent. The infrared spectra of MCNS were obtained by using MCNS 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 MCNS 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).

3) Bromocresol Green Dye

Additionally, BCGD and orthophosphoric acid (H_3PO_4) were Analytical grade reagents procured from Sigma Aldrich Chemical, Germany. BCGD (1000 mg) was correctly weighed into 250 ml conical flask, and small quantity of distilled water was added and stirred uninterruptedly 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.

4) Effect of adsorbent dosage 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 130 rpm. The optimum initial dye concentration was 20 mg/L, 30 minutes contact time, 50 ^{o}C, and the adsorbent dosage was varied from (0.1 to 0.5) g at 0.1 g interval. After the adsorption experiments, the adsorbent was separated from dye effluent by centrifugation using centrifuge at room temperature at 2000 rpm for 20min.

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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 MCNS was determined according to Jabar *et al*. (2020) as shown in equation 1.

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

The amount of BCGD adsorbed per unit weight of MCNS 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 (MCNS) (g)

B. Adsorption Isotherm models

1) Brouers Weron Sotolongo isotherm

For BWS isotherm model from equation 2.36 which states as follows:

$$q_e = q_{\max}(1(\exp(-K_W C_e^{\alpha}) \quad 4$$

Where:

q_e = maximum adsorption capacity (mg/g)

$q_{\max}$ = saturation value

$$K_W = K_F / q_{\max} \quad 5$$

Where K_F is the low C_e Freundlich constant, for a given temperature

C_e = equilibrium concentration of adsorbate (mg/L)

α = is a measure of the width of the sorption energy distribution and that of the energy heterogeneity of the adsorbent surface (Unuabonah*et al*., 2017).

The plot of qe against Ce gives isotherm model.

2) Freundlich isotherm model

Freundlich nonlinear equation is given as;

$$q_e = K_F C_e^{\frac{1}{n}} \quad 6$$

The plot of q_e against C_e gives the model parameters,
The linear form of Freundlich is given thus;

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \quad 7$$

Using the logarithmic form of Freundlich isotherm model given equation 7, a plot of
log q_e against log C_e gives a straight line with 1/n as the slope and log K_F as intercept, from which $K_F$is calculated.

3) Jovanovich isotherm model

For Jovanovich isotherm nonlinear form is given as:

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$$q_e = q_m(1 - e^{KJ} C_e) \quad 8$$

Where:

q_e = maximum adsorption capacity (mg/g)

q_m = saturation value

C_e = equilibrium concentration of adsorbate (mg/L)

K_J = Jovanovich isotherm constant

4) Langmuir isotherm

From Langmuir isotherm model, the nonlinear equation is given as;

$$q_e = \frac{K_L Q_o C_e}{1 + K_L C_e} \quad 9$$

The linear form is given as;

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_o} + \frac{C_e}{Q_o} \quad 10$$

A plot of C_e/q_e against C_e gives a straight line with $1/Q_0$ as slope and $1/K_LQ$

5) Sips isotherm

From the Sips isotherm, the nonlinear form is given thus;

$$q_e = \frac{K_s C_e^{\beta s}}{1 + a_s C_e^{\beta s}} \quad 11$$

Where:

K_s and α_s the Sips isotherm constants

β_s = The exponent which lies between 1 and 0

The linear form of Sips isotherm is given as;

$$\beta_s \ln C_e = \ln(\frac{k_s}{q_e}) + \ln a_s \quad 12$$

6) 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 (χ^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 χ^2 and SSE (Olafadehan*et al.*, 2022) are:

Normalized Chi square error test, $x^2 = \sum_{K=1}^{N} \left| \frac{(q_k(\exp) - q_k(pred)^2}{\sum_{k=1}^{Ne}(q_{k,\,pred} - q_e)^2} \right|$ 13

Coefficient of determination $R^2 = 1 - \frac{\sum_{k=1}^{Ne}(q_{k,\exp} - q_{k,pred})^2}{\sum_{k=1}^{Ne}(q_{k,pred} - q_e)^2}$ 14

III. RESULTS AND DISCUSSION

Table 1, presented the physicochemical and proximate properties of the prepared adsorbent under study with the following outcomes: pH (7.10 ± 0.101), moisture content (3.50 ± 0.110) %, volatile matter, (9.00 ± 0.012) %, ash content (15.70 ± 0.111) %, fixed carbon (71.80 ± 0.001) %, bulk density (0.769 ± 0.000) g/cm^3, surface area (1120.00 ± 0.000) m^2/g and particle size (300.00 ± 0.000) µm. They all conform to those reported in the literature (Ajala and Ali, 2020; Abdullahi *et al.*, 2022; Sangoremi *et al.*, 2024), 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 1: Physicochemical Properties and Proximate Compositions of Modified Coconut Shell Adsorbent (CSAC)

S/no	Parameters	Mean ± SE
1	pH	7.10 ± 0.101
2	Moisture content (%)	3.50 ± 0.110
3	Volatile matter (%)	9.00 ± 0.012
4	Ash content (%)	15.70 ± 0.111
5	Fixed carbons (%)	71.80 ± 0.001
6	Bulk density (g/cm^3)	0.769 ± 0.000
7	Surface area (m^2/g)	1120.00 ± 0.000
8	Particle size (µm)	300.00 ± 0.000

Figure 2 shows the FTIR spectra of MCSAC adsorbent. The characteristic functional groups in MCNS biosorbent were identified from FTIR spectra. The peak at 3429 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 2926.11, 2004.11, 1633.76, 1464.02, 1246.06 and 1033.88 cm^{-1}. These are due to C-H, C=C, C=O, C=O, CH_2, C-N and C-O stretch respectively (Nandiyanto*et al.*, 2019; Sangoremi *et al.*, 2024).

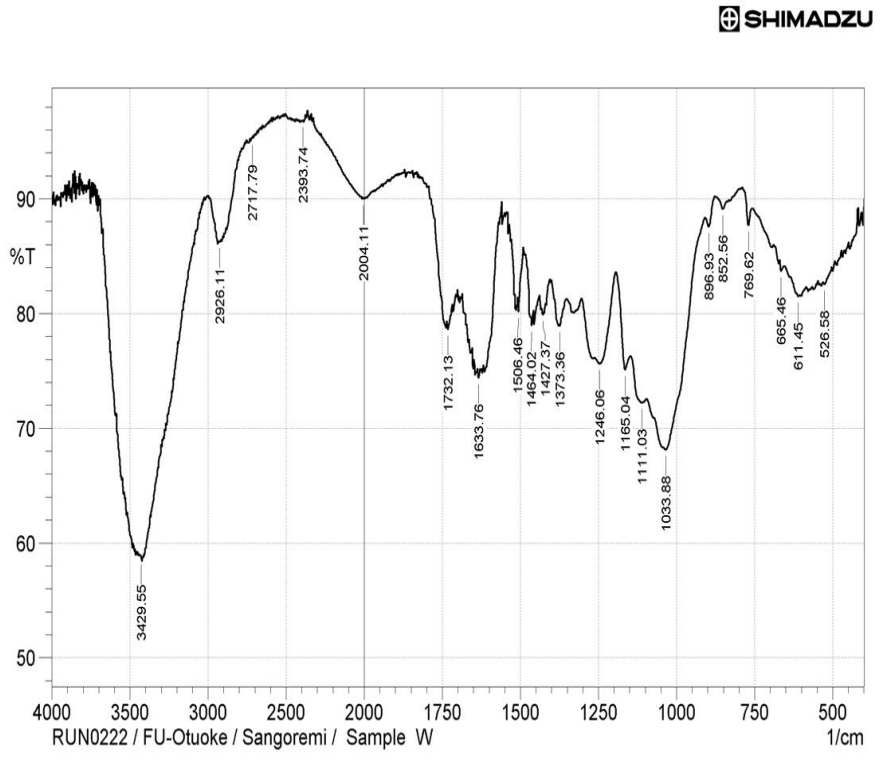


Figure 2: FTIR of modified coconut shell activated carbon (MCNS)

Table 2: FTIR Spectra Analysis of Modified Coconut Shell Adsorbent

S.No	Wave number (cm^{-1})	Frequency range (cm^{-1})	Functional group
1	3429.55	3500-3200 (m)	O-H stretch
2	2926.11	3000-2850 (s)	C-H stretch
3	2004.11	2100-1800	C≡C
4	1732.13	1750-1730 (s)	C=O, esters –COOR, saturated aldehydes -CHO
5	1633.76	1680-1620	C=O stretch
6	1464.02		$-CH_2-$
7	1246.06	1320-1000 (s)	C-N stretch
8	1033.88	1120-1030	C-O stretch

A. Scanning Electron Micrographs (SEM)

Figure 3 shows the Scanning Electron Micrograph of the MCNS adsorbent. The SEM image of MCNS 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 the adsorbent by diffusion of BCGD molecules from the aqueous solution to the MCNS 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 MCNS 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).

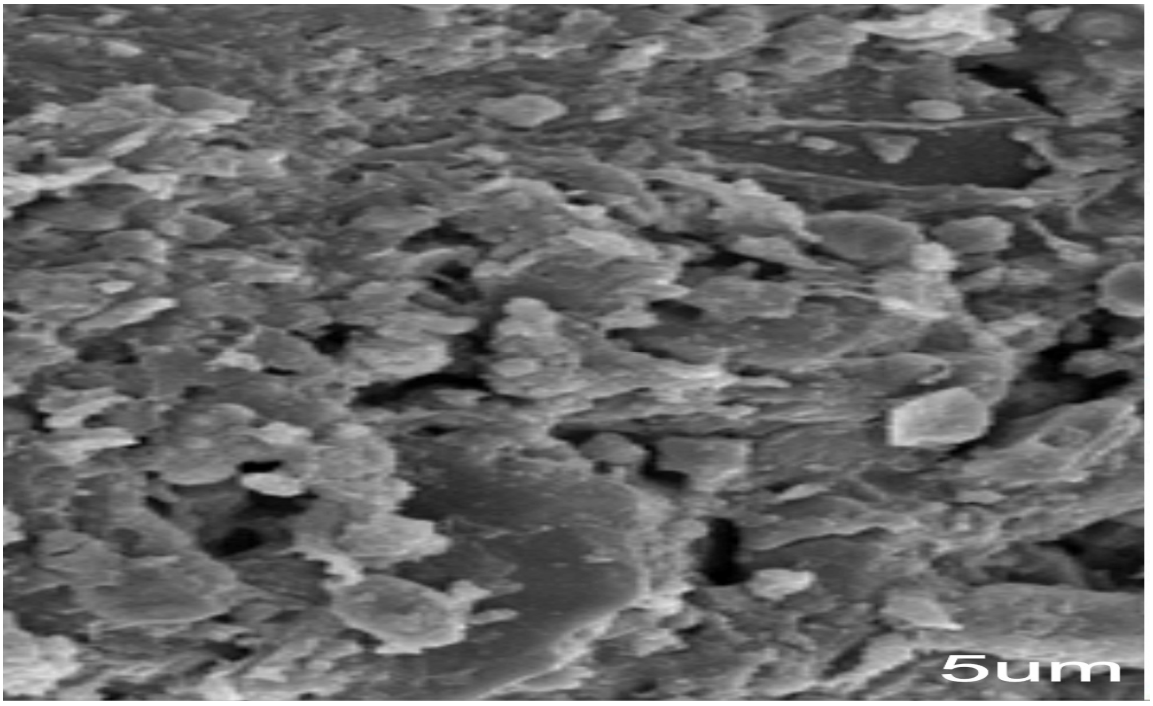


Figure 3: SEM Micrograph of Modified Coconut Shell Adsorbent

B. Electron Dispersive X-ray Spectroscopy Analysis (EDX)

The EDX analysis is a method of elemental analysis associated with electron microscopy based on the generation of characteristic x-rays that divulges the presence of element present in a sample (Scimeca *et al.*, 2018). Figures 4 revealed the EDX spectra of MCNS biosorbent, while Tables 2 revealed the elemental composition of the adsorbents respectively. The carbon content was56.11 % which demonstrates that the process of activation has enriched the carbon content in theadsorbents. Other elements present in percentage atomic weight include: oxygen (43.77 %), and titanium (0.21%) (Ushedo*et al.*, 2022; Sangoremi *et al.*, 2024).

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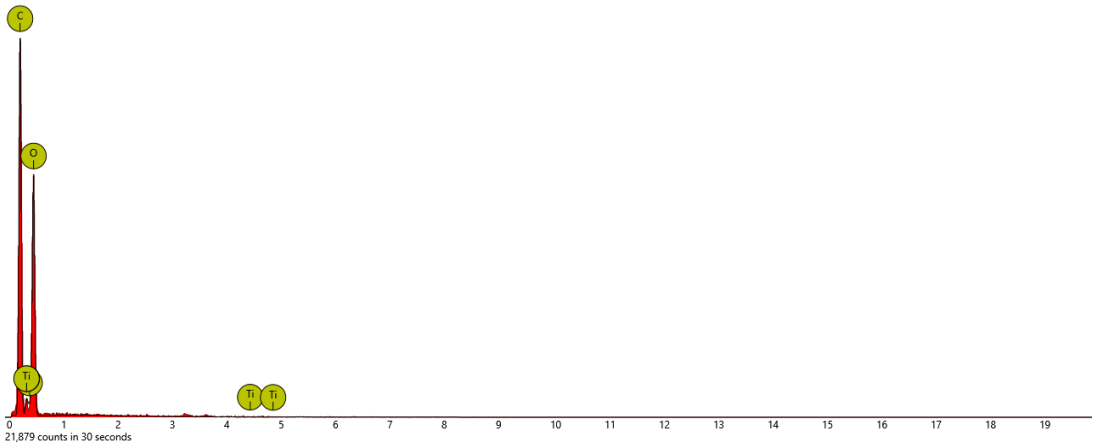


Figure 4: EDX Adsorption Spectrum of Modified Coconut Shell Adsorbent

Table 3: Elemental Composition of MCNS Adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	65.00	56.11
8	O	Oxygen	34.80	43.77
22	Ti	Titanium	0.20	0.12

C. Adsorption Isotherm Model

Experimental isotherm data obtained in this study were analysed with five theoretical models: Langmuir, Freundlich, Jovanovich (two-parameters isotherm models), Brouers Weron Sotolongo Coasta and Sips isotherm models (three parameters isotherm models). The plots of the fittings of these theoretical models compared to experimental data are shown in Figure 5. The heterogeneity factor (n) in the Freundlich model can be used to indicate the degree of favourability of adsorption (Chung *et al*., 2015). The Freundlich constant, n should have values lying in the range 1 to 10 for classification as favourable adsorption. The value of n for the adsorbent used in the current study is greater than 1 but less than 10 indicating that the adsorption of BCGD onto MCNS is favourable. The same observation was made by Unuabonah et al (2017) onFacile synthesis of new amino-functionalized agrogenic hybrid Composite Clay adsorbents for phosphate capture and recovery from water. From the analysis of all the isotherms used in this study with respect to coefficient of determination (R^2), that the equilibrium data for the adsorption of BCGD onto MCNS was best described by Freundlich isotherm model which predicts non-ideal and reversible adsorption on heterogeneous adsorption sites with no formation of monolayer on the adsorbent's surface (Olu-Owolabi *et al*., 2010).

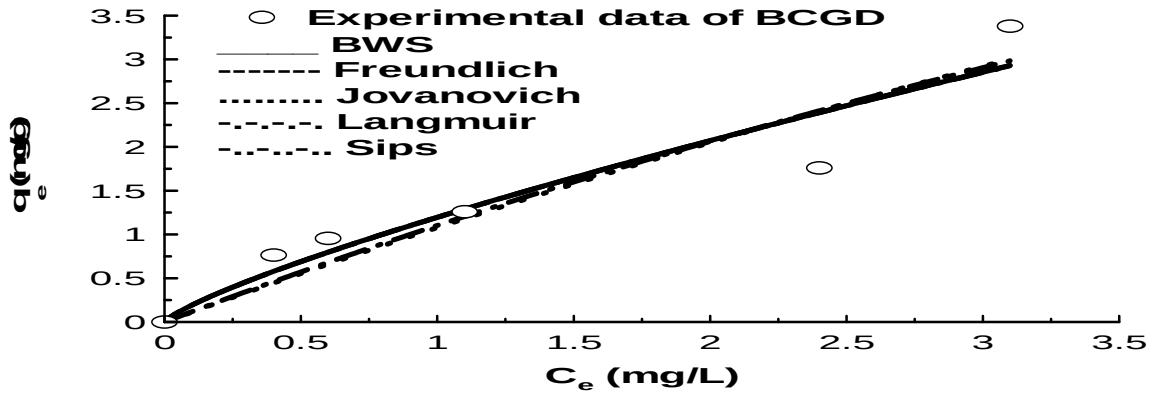


Figure 5: Adsorption IsothermModel of BCGD onto MCSAC

D. Effects of Operational Parameter

Effect of the Adsorbent Dosage

Figure 6 shows the effects of the adsorbent dosage on the percentage dye removal. As dosage of MCNS adsorbent increases from 0.1 g, the percentage BCGD removal also increases, this could be as a result of availability of large adsorption pore surface areas which connotes the availability of morevacant adsorption sites for dye removal till it reaches the optimum dosage of 0.4 g. After which the percentage dye removal was marginal. This is a stage where the vacant adsorption sites where fully saturated that they could no longer hold more molecules of dye molecules from wastewater. It is a state of dynamic equilibrium, where the rate of adsorption is equal to the rate of desorption. This is in tandem with the reports of other researchers (Sangoremi *et al*., 2024).

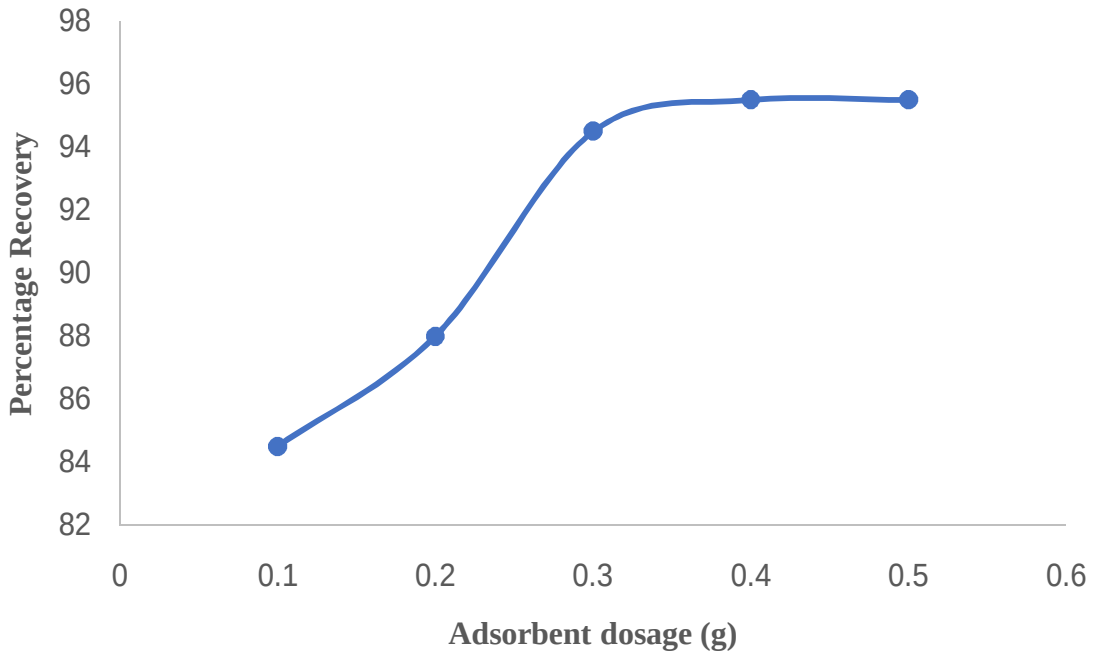


Figure 6: Relationship between Adsorbent Dosage and Percentage BCGD Bound onto MCNS Adsorbent

IV. CONCLUSION

The prepared adsorbent possesses high carbon contents, low inorganic contents, high surface area, and heterogenous pore structures that make it a viable precursor for the removal of BCGD from aqueous solution. The dye removal from the wastewater was dosage-dependent with maximum removal efficiency of 95.5 % at optimum dosage (0.4). The isotherm model that fairly described the removal of BCGD from the wastewater was Freundlich isotherm model with a coefficient of determination of (R^2 = 0.8999). The prepared adsorbent stands efficient in remediating the dye polluted wastewater to serviceable status.

V. ACKNOWLEDGEMENTS

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Conflict of interest

The author declares that there is no known conflict of interest as regard this work.

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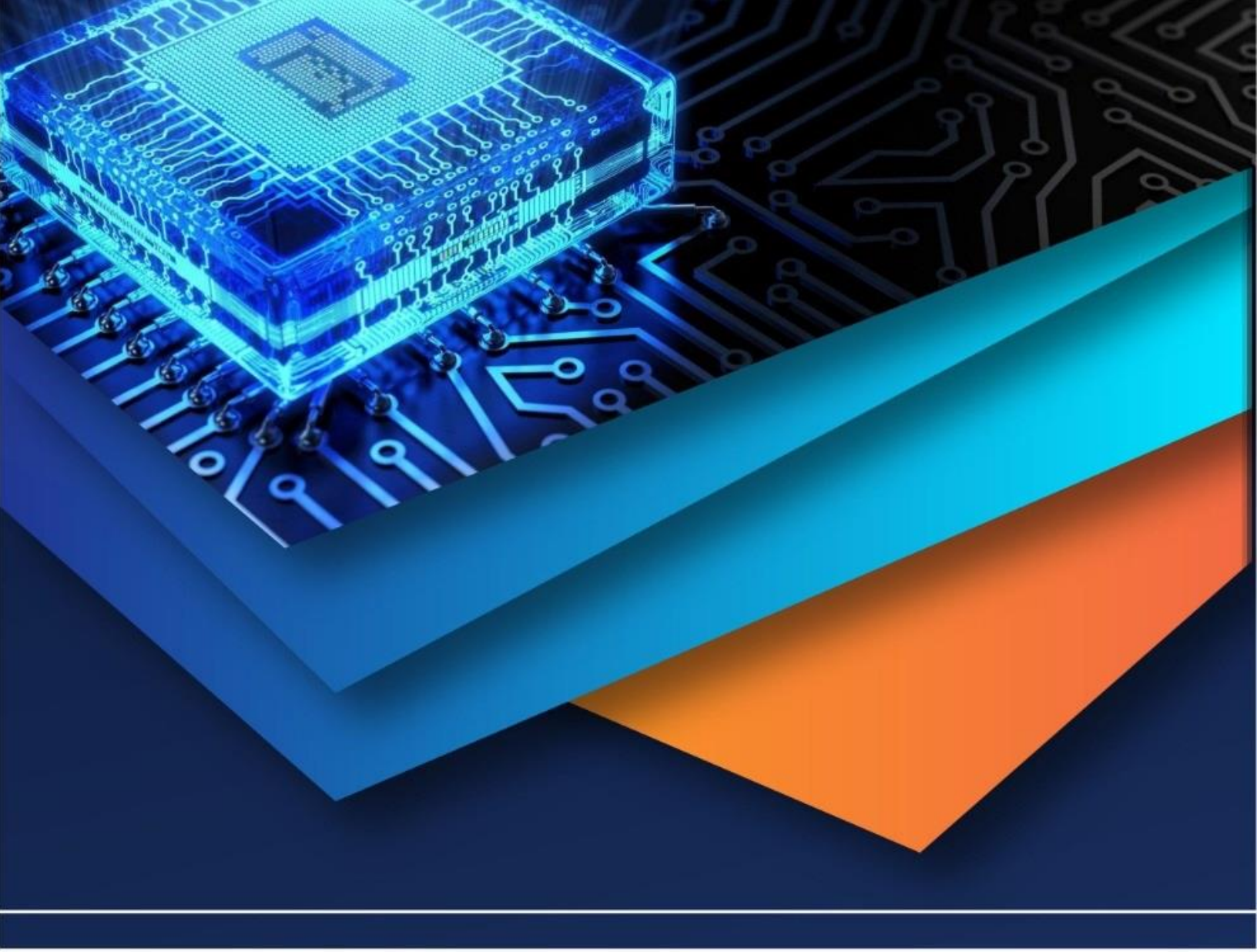
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