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Adsorption study of Bisphenol A (BPA) by Base Activated coconut shaft as a Low-Cost Adsorbent (Isotherm, Kinetics and thermodynamic studies)

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Abstract

This study investigated the effect of base-modified coconut shaft (BAC) on the elimination of Bisphenol A (BPA) from aqueous solution. An alkali (NaOH) was employed to activate the coconut shaft. The characterization of the activated adsorbent before and after adsorption was done using Fourier Transform Infrared (FTIR) spectroscopy. Various operational parameters, such as pH, adsorbent dosage, temperature, contact time, and initial BPA concentration, were examined in batch experiments. The functional groups such as hydroxyl, carboxyl, and aromatic groups which are responsible for the adsorption of the contaminant were revealed by FTIR characterization. It was determined that the optimal factors for BPA removal were pH 2, adsorbent dosages of 0–5 g/L, contact time 80 min, 100ppm initial concentration and 45°C. Kinetic studies indicated that the adsorption process followed pseudo-second-order kinetics, suggesting that chemisorption was the rate-limiting step. The Langmuir model provided a maximum monolayer adsorption capacity of 35 mg/g, while the Freundlich model more accurately described the adsorption isotherms, indicating a heterogeneous adsorption process. Thermodynamic analysis showed that adsorption was impulsive and endothermic, with positive values for enthalpy (ΔH° = 5. 76 kJ/mol) and entropy (ΔS° = 8. 27 kJ/mol·K), and negative Gibbs free energy (ΔG°) values ranging from -1. 08 to -2. 13 kJ/mol across the temperature range of 25–65°C. The study demonstrates that base-modified coconut shaft (BMC) is a promising, eco-friendly, and cost-effective biosorbent for BPA removal from solutions. Its performance, characterized by favorable thermodynamics, kinetics along with excellent adsorption capacity qualify it as a viable alternative adsorbent to be considered for adsorption purpose.

Keywords: Adsorption, Bisphenol A (BPA), Thermodynamics, Kinetics, Adsorbent

INTRODUCTION

Significant environmental problems could result from the release of various organic and inorganic pollutants into the environment. It has been recognized for a long time that numerous types of chemicals are employed in manufacturing and other industrial processes. However, most of these compounds are inherently hazardous and present an escalating environmental threat that necessitates urgent action. A trace organic contaminant such as bisphenol A, also identified as 4,4´-(propane-2,2-diyl) diphenol, or BPA. In the chemical industry, it is a widely used organic chemical molecule in the production of food packaging, polyester, flame retardants, thermal paper, brake fluid, epoxy resins, and polycarbonate polymers (PC) (Kang *et al* 2014; Keykavoos, 2012).

BPA is a white, solid material with a slight phenolic odour, low vapor pressure, and minimal solubility in water at room temperature. According to Wu *et al.* (2018) and Jun *et al.* (2022), BPA is classified as an endocrine-disrupting chemical (EDC).

EDCs can disrupt the natural hormone systems and functions of the body by acting either agonistically or antagonistically on key hormones receptors, such as those for estrogen and androgen (Fowler *et al.* 2012). They are harmful, causing metabolic and functional disorders in

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both humans and animals, including obesity, thyroid issues, infertility and abnormal reproductive functions (Diamanti-Kandarakis *et al* 2010). The primary sources of BPA are thought to be wastewater from the plastic industry that is discharged into surface waters. As noted by Bohdziewicz and Liszczyk (2013), sewage sludge and landfill leachate are also considered point sources of BPA in surface water. Humans mainly encounter this contaminant through food, water supplies, and drinking water bottles (Vandenberg *et al*, 2012). The reality underscores and reinforces the growing body of research on effective methods and materials for removing bisphenol A from water (Wang *et al*. 2022). Currently, oxidation, adsorption, catalysis, and biodegradation are the techniques employed to eliminate BPA. Among these, adsorption is one of the most advantageous due to its efficiency, simplicity, and cost-effectiveness (Wang *et al*. 2022).

The adsorption method was proposed for this study to eliminate BPA from aqueous solutions based on its cost-effectiveness, efficiency, robustness, and ease of design. A common adsorbent employed in the adsorption process for removing emerging contaminants is activated charcoal (AC) (Lazim *et al*, 2015; Jeirani *et al*, 2017; Zhou *et al*, 2019). When compared to commercially activated charcoal, agricultural waste adsorbents are currently the most preferred and widely employed. This is because agricultural wastes have been recognized to be abundant in nature and contain different functional groups and components that possess the potential sorption capabilities for numerous pollutants. They are also reliable, abundant and eco-friendly and have been thoroughly researched and recognized to be low-cost adsorbents (De Disi et al., 2016; Inyinbor et al., 2017; Omo-Okoro et al., 2018). In the present study, the adsorption capacity of coconut shaft as an alternative adsorbent was investigated.

MATERIALS AND METHODS

Equipment

The laboratory equipment used in this study included a magnetic stirrer, analytical balance, oven, vacuum filter, mortar, and pestle while the analytical instruments used include Fourier Transform Infrared (HITACH), UV-Vis spectrophotometer (U-2010 Spectrophotometer), shaker (SCILOGEX), glassware and plastic apparatus.

Collection of Biosorbent

Coconut shaft was used as a low-cost biosorbent for the removal of Bisphenol A from aqueous solution in this study. The coconut shaft was collected from local coconut vendors in Ijagun, Odogbolu Local Government Area of Ogun State, southwest Nigeria. The collected shafts were rinsed with distilled water to remove all dirt and soluble contaminants, oven dried for 24 h and then cut into small pieces, ground then stored in airtight containers until further use.

Preparation and Modification of Biosorbent

The activation of the surface of the adsorbent involved treating 50g of the dried coconut shaft with 1M NaOH for 18 h. Soaking allows the adsorbent to capture and retain specific molecules or ions from the activating reagent. After 18 h, the coconut shaft was sieved and thoroughly rinsed with distilled water. This rinsing step is crucial for removing excess NaOH from the surface of the adsorbent. After a thorough wash, the adsorbent was dried for 24 h in a laboratory oven. The activated adsorbent was ground after drying in order to increase the surface area of the adsorbent, it was later stored in an airtight container.

Characterization of the Biosorbent

Fourier Transform Infrared Spectroscopy (FTIR)

The base (NaOH) treated coconut shaft was analyzed using FTIR before and after the experiment in order to identify potential functional groups on the surface of the biosorbent and determine the change that may have occurred upon adsorption of the pollutant. The samples were studied in the 4000-400 cm^{-1} region

Preparation of Bisphenol A Solutions

In this experiment, 20 mg of bisphenol A (BPA) granules were dissolved in 500 mL of distilled water in a volumetric flask to create a stock solution. A magnetic bar was used to stir the heated solution until it was completely dissolved. To achieve the desired concentrations, four standards with varying BPA concentrations (25 mg/L, 50 mg/L, 100 mg/L, and 150 mg/L) were prepared by diluting the stock solution. After placing the blank and standards into the UV-Vis Spectrophotometer, analysis was carried out at a wavelength of 214 nm. The absorbance values for both the blank and each standard were recorded. Following this, absorbance as a function of concentration was

plotted on a standard calibration graph to form a calibration curve. The concentration of the BPA samples was determined using this curve after they reacted with base activated coconut shaft powder.

Biosorption Experiments

Adsorption of the pollutant (BPA) was performed by batch experiment by varying different parameters. A certain amount of the activated coconut shaft was put in contact with 25 mL of BPA solution for 2 h at different initial concentration of the pollutant (20-150 mg/L). By altering the pH range between 2 – 6, the impact of the starting pH on the adsorption process was examined. Experimental parameters such as biosorbent dose (0.5 – 5 g), initial BPA, contact time (20 – 120min), and temperature (25 – 65 °C), were investigated so as to determine the efficiency of the NaOH-treated coconut shaft towards Bisphenol A removal.

Calculations

Efficiency and adsorption capacity of the adsorbent (BCS) towards the adsorption of BPA from their aqueous solution were calculated using equations 1 and 2 (Bansal et al., 2020; Ayuba and Idoko 2021).

$$\% \text{ BPA removal} = \frac{Ci - Ce}{Ci} \times 100 \quad (1)$$

$$q_e = (C_i - C_e)\frac{V}{m} \quad (2)$$

V = volume of solution (L), m = mass (g) of adsorbent, qe = adsorption capacity (mg/g) and Ci and Ce = initial and final concentrations (mg/l) of the contaminant in the solution.

Biosorption Kinetics

To understand the adsorption kinetics of BPA onto BCS, two kinetic model were used to test the data from batch adsorption studies. Both pseudo-first-order and pseudo-second-order models were employed in their linear form as shown in the equations below (Kamarehie *et al.*, 2020; Ayuba and Sani 2022) :

$$\text{Log}\,(q_e - q_t) = Log\ q_e - \frac{k_1}{2.303}t \quad \text{(pseudo first order)} \quad (3)$$

where q_t (mg/ g) is the amount of BPA adsorbed per time, t is the contact time (min), and the pseudo-first-order constant is K (min).

By plotting the log ($q_e - q_t$) versus t, K_1 and qe were calculated from the slope and intercept, respectively.

Pseudo-second-order was calculated using equation (4):

$$\frac{t}{q_t} = \frac{1}{q_e^2 K_2} + \frac{1}{q_e}t \quad \text{pseudo-second order (4)}$$

The pseudo second order rate constant, K_2 (mg/g), is obtained from the intercept of the plot of t/q_t vs t, while qe is the obtained from the slope.

2.7 Biosorption Isotherms

The adsorption isotherms are useful for determining how the adsorbate is distributed on the adsorbent's surface under equilibrium conditions (Sharma et al., 2018). To identify the mechanistic characteristics of interactions between the adsorbent and the adsorbate at equilibrium, Langmuir and Freundlich were tested. The linearized form of these models is presented in equations (5-6) as reported by Sadani et al. (2020).

The Freundlich adsorption isotherms, which assume that adsorption takes place on heterogeneous surfaces, can be expressed as:

$$lnq_e = lnK_F + \frac{1}{n}lnC_e \quad (5)$$

Langmuir equation

$$\frac{C_e}{q_e}\text{“} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (6)$$

Where Ce stands for the equilibrium concentration (mg/L); qe and $q_m$for the equilibrium and maximum adsorption capacities (mg/g) respectively; KL denotes the Langmuir rate constant related to the affinity between adsorbate and adsorbent; *KF* and n are the rate constants of the Freundlich model that indicate the affinity of BPA towards the adsorbent (Nourmoradi et al., 2015). Table 2 displays the calculated values of the isotherm parameters for BPA removal by BCS.

The Langmuir constants, qm (maximum adsorption capacity) (mg/g) and KL (values for the Langmuir constant related to the energy of adsorption) (L/mg), are predicted from the plot of qe/Ce versus Ce. The Freundlich isotherm graph plots log q_e against log Ce to provide the value of KF and n. Plotting a linear curve of the Langmuir and Freundlich graphs allows one to calculate the correlation factor (R^2).

2.8 Thermodynamic Studies

The thermodynamic of the adsorption can be determined by calculation of enthalpy (ΔH^o), entropy (ΔS^o) and Gibbs free energy. Equations 7 and 8 were used to calculate the thermodynamic parameters. The slope and intercept of the van't Hoff's plot of lnKc vs 1/T as used to compute ΔH^o and ΔS^o (Min et al., 2015; Umar et al., 2022).

$$\Delta G^o = -RT\ln K_c \quad (7)$$

$$\ln K_c = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \quad (8)$$

RESULTS AND DISCUSSION

FTIR Characterization of Base-Modified Coconut Shaft

The main functional groups found on the surface of the base-modified coconut shaft prior to and after BPA uptake is shown in figure 1. A broad band characteristic of O-H stretching and indicating the presence of cellulose, hemicellulose and lignin (Kumar et al., 2020) in the coconut shaft was observed between 3600 and 3200 cm^{-1}. A vibrational frequency characteristic of aliphatic C-H stretching were found around 2920 and 2850 cm^{-1}. At approximately 1730, a band representing the C=O stretch of caboxyl and ester groups was found and this may have been enhanced as a result of the base modification process. (Li et al., 2018). As for the prominent peak observed around 1610 cm^{-1}, this is indicative of an aromatic C=C bond. The band between 1500 and 1400 cm^{-1} can be attributed to the C-H bending in methyl and methylene groups. Upon BPA adsorption, slight shifts were observed in some characteristic peaks for example, a shift from 1730 to 1725 cm^{-1} or 1610 to 1615 cm^{-1}. These shifts suggest the involvement of carbonyl and aromatic groups in the adsorption process. The broad band (3600 – 3000 cm^{-1}) representing the O-H, Post-adsorption, the broadening of the O-H stretching band (3600 – 3200 cm^{-1}) may indicate hydrogen bonding between BPA and the adsorbent surface (Tran et al., 2020). The appearance of an intense peak between 1200 – 1000 cm^{-1} after BPA absorption could be attributed to C–O stretching vibrations, suggesting interactions between BPA and oxygen-containing functional groups on the surface of the modified coconut shaft (Zbair et al., 2018).

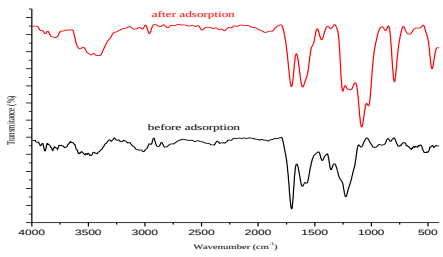


Figure 1. FTIR Spectrum of Base-Modified Coconut Shaft Before and After Adsorption of Bisphenol A.

Effect of pH

The effect of pH on the adsorption of BPA onto the BCS surface was analyzed by adjusting the pH of the solution between 2 and 6. The result of this experiment revealed that the capacity of the BCS to adsorb decreased as the pH of the BPA solution rose (Zubair et al., 2017).

Thus, at a pH of 2, the highest adsorption efficiency for BPA reached 91.25%. The reduction in BPA adsorption at higher pH is attributed to electrostatic repulsion and competition between hydroxide ions and BPA oxyanions for active adsorption sites and changes in the surface charge of the adsorbent (Dehghani *et al*, 2020). Conversely, reducing the pH of the solution enhances the quantity of the positive charges on the surface of the adsorbent, which in turn improves the adsorption of BPA onto BCS surface (Rashidi *et al.*, 2021)

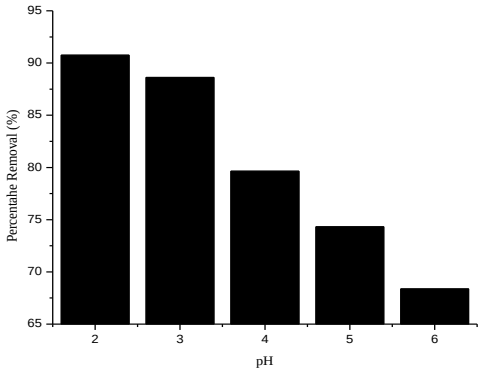


Figure 2. Effect of pH

Effect of Adsorbent Dosage

Figure 3 illustrates the relationship between adsorbent dose and the adsorption efficacy of BPA. The percentage of BPA uptake increased as the BCS dosage increased. The maximum removal at 83%was achieved at the dosage of 2 g. The increased surface area and available adsorption sites are responsible for the initial sharp rise in the removal efficiency (Li et al., 2019).

A saturation point is reached after which increase in the amount of the adsorbent does not appreciably enhance the contaminant removal from the solution. As saturation of the adsorbent surface is reached at higher dosage and adsorption efficiency become low.

This could be because of particles aggregation reduces the effectiveness of the available surface area for adsorption of the contaminant. This finding is in agreement with study of Ogundiran et al. (2022).

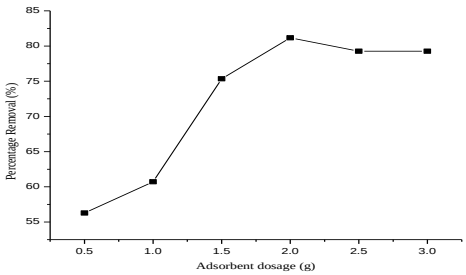


Figure 3. Effect of Adsorbent Dosage.

Effect of Temperature

The effect of temperature on the adsorption of BPA onto BCS is presented in Figure 4. The result indicates that the process of adsorption of BPA onto BCS is endothermic in nature as the adsorption capacity increased with increase in temperature.

From the plot, it is noticed that increasing the temperature from 25°C to 45°C led to a gradual increase in BPA uptake (). This increase is attributed to increased molecular motion at higher temperatures, which facilitated the BPA-adsorbent interaction and expansion of pores within the adsorbent structure thereby improving uptake efficiency of the contaminant.

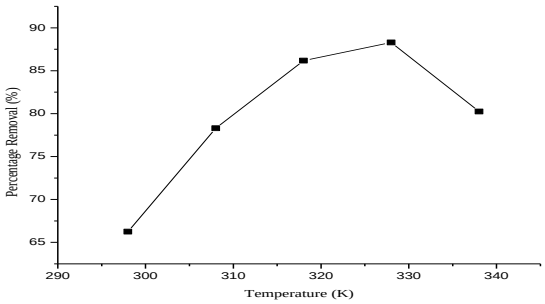


Figure 4. Effect of Temperature.

Effect of Contact Time and Initial Concentration

There was a rapid uptake of BPA initially when the adsorption started, which was later characterized by slower removal as equilibrium was approached. This is due to the presence of numerous sites available at the of the adsorption process, the sites on the adsorbent were gradually occupied by the BPA molecules over time. Figure 5 shows the plot of contact time alongside the initial BPA concentration. The plots showed an increase in the adsorption capacity at various initial concentrations versus the change in time.

It is noteworthy to state that the greater equilibrium adsorption capacities are the outcome of higher initial BPA concentrations and this can be explained by the increased mass transfer driving force at higher concentration gradients, resulting in more effective use of the surface of the coconut shaft's available adsorption sites Wang et al. (2021).

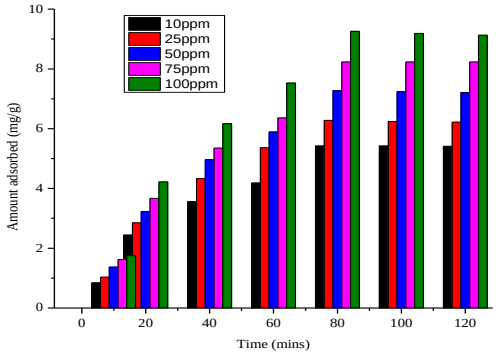


Figure 5. Effect of Contact Time and Initial Concentration.

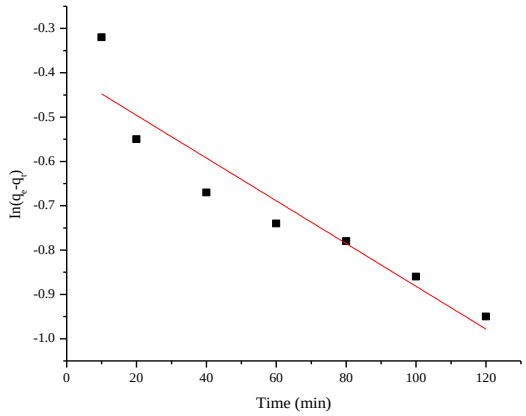
Adsorption Kinetics

Figures 6 and 7 show the pseudo-first-order and pseudo-second-order kinetic models for the elimination of BPA onto base-modified coconut shaft, respectively. These plots are important in determining the rate-limiting step and the general adsorption mechanism.

The values of the parameters and the correlation coefficients (R^2) of both kinetic models used for this study are presented in Table 1 below. The results are obtained for various initial BPA concentrations. Pseudo second order kinetic has higher values of correlation coefficient at all concentrations when compared to the pseudo-first-order model ($\leq$ 0.965). The pseudo-second-order model's applicability is further supported by the fact that the computed qe values from it agree more closely with the experimental qe values (Li *et al.*, 2019; Yao *et al.*, 2020). It appears that higher concentrations result in faster adsorption rates because the pseudo-second-order rate constant (k2) increases as the initial BPA concentration rises.

These kinetic results suggest that chemisorption may be significant in the adsorption of BPA onto base-modified coconut shafts, especially given the pseudo-second-order model's good fit.

Figure 6. Pseudo-First Order Kinetic Model of Adsorption of Methylene Blue on Base-Modified



Coconut Shaft.

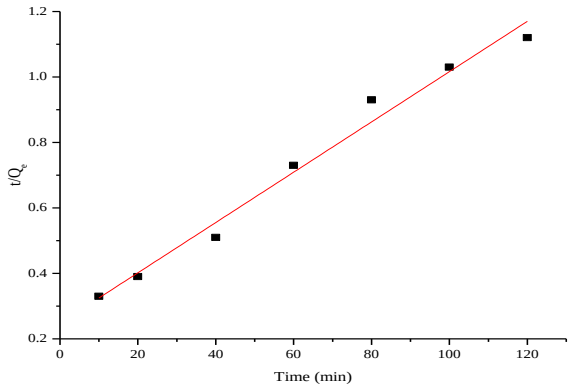


Figure 7. Pseudo-Second Order Kinetic Model of Adsorption of BPA on Base-Modified Coconut Shaft.

Table 1. kinetic model parameters of Base-Modified Coconut Shaft for the removal of BPA.

				PSEUDO FIRST-ORDER			PSUEDO SECOND-ORDER			
	C_e	Q_e(exp) (mg/g)	Q_e(cal) (mg/g)	K_1 (min^{-1})	R^2	% SSE	Qe(cal)	K_2	R^2	% SSE
10	1.68	5.41	4.25	0.22	0.965	0.088	5.39	0.37	0.996	0.002
25	2.76	6.22	5.82	0.45	0.930	0.026	6.21	0.44	0.990	0.001
50	3.46	7.21	6.11	0.65	0.950	0.062	7.19	0.53	0.980	0.001
76	7.33	8.23	6.82	0.76	0.950	0.070	8.25	0.73	0.980	0.001
100	8.57	9.13	7.06	0.82	0.940	0.093	9.15	0.880	0.990	0.001

Isotherms Studies

Figures 8 and 9 show the Langmuir and Freundlich adsorption isotherm models, respectively, for the biosorption of BPA onto base-modified coconut shaft. These isotherms are crucial for understanding the interaction between BPA molecules and the adsorbent surface, as well as for determining the maximum adsorption capacity of the system. Figure 8 illustrates the Langmuir isotherm model. The Langmuir isotherm assumes monolayer adsorption on a homogeneous surface with a finite number of identical sites. The calculated Qmax value of 8.35 mg/g indicates the maximum quantity of BPA that can be adsorbed per unit mass of the modified coconut shaft. The Langmuir separation factor (RL) of 0.63, falling between 0 and 1, suggests that the adsorption process is favorable under the conditions studied. Figure 9 presents the Freundlich isotherm model. The Freundlich isotherm describes heterogeneous systems and assumes a non-uniform distribution of adsorption heat and affinities over the adsorbent surface. The calculated 1/n value of 0.280, falling between 0 and 1, confirms the favorability of the adsorption process and suggests that the surface of the base-modified coconut shaft is heterogeneous (Ahmed et al, 2021). Both models' isotherm parameters are given in Table 2. By comparing the correlation coefficients (R^2), the Freundlich isotherm (R^2 = 0.996) fits better than the Langmuir isotherm (R^2 = 0.952). The adsorption of the BPA onto the base-modifies coconut shaft may hence be suitably explained by a heterogeneous adsorption process, according to this. R2 > 0.95, the good fit of both models, suggest that the homogeneous and the heterogeneous surface binding mechanisms are probably involved in the adsorption process. Following the Langmuir model's comparatively good fit, selected locations on the coconut shaft surface exhibit some levels of monolayer coverage. These isotherm results therefore provide valuable insights into the adsorption capacity and the nature of BPA interactions with the base-modified coconut shaft surface. Both models fit well, indicating that the adsorption process is intricate and involves a variety of binding sites or interactions.

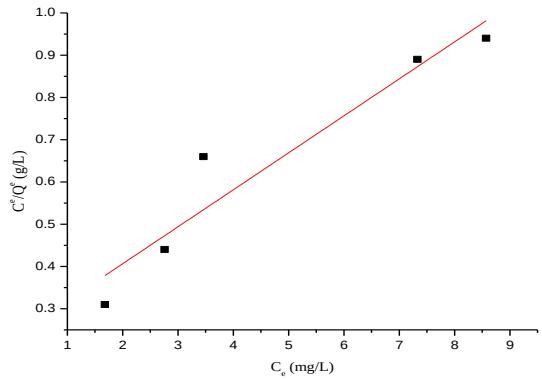


Figure 8. Langmuir Adsorption Isotherm Model on Base-Modified Coconut Shaft Uptake of BPA.

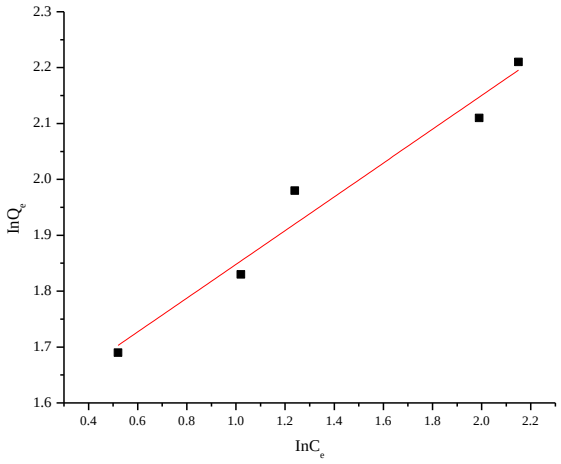


Figure 9. Freundlich Adsorption Isotherm Model for Base-Modified Coconut.

Table 2. Isotherm constant for the adsorption of BPA using Base-Modified Coconut Shaft.

Langmuir		Freudlich	
Qmax	8.35	Kf	10.34
KL	0.63	1/n	0.280
R2	0.952	R2	0.996

Thermodynamic Studies

The result of thermodynamic analysis is displayed in Table 3. As indicated by the negative ΔG^o values, the adsorption system was found to be thermodynamically viable and spontaneous. At values less than -20 kJ/mol, adsorption process was determined to be physical (Nourmoradi et al., 2015; Ayuba and Idoko, 2020). Due to increase in temperature from 298 to 338 K, a rise in adsorption rate was observed. Additionally, a positive enthalpy changes ΔH^o confirmed the thermodynamic nature of the adsorption of BPA by BCS. It was further revealed that ΔH^o values were positive and below 80 kJ/mol, suggesting that the interaction between the adsorbate and the surface of the adsorbent was physical during the adsorption. Furthermore, the positive entropy (ΔS) change value suggested that BCS can readily release BPA, hence eindicating that the adsorption process is primarily driven by entropy rather than enthalpy (Asadullah et al., 2019).

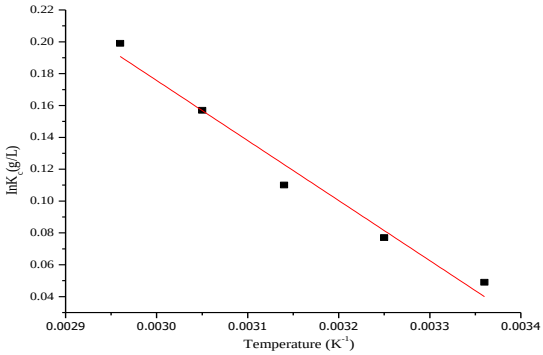


Figure 10. Thermodynamic parameters for the sorption of Bisphenol A onto Base-Modified Coconut Shaft.

Table 3. Thermodynamic parameters for the sorption of Bisphenol A onto Base-Modified Coconut Shaft.

T (^{O}K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (kJ/mol)
298	-1.08		
308	-1.13	5.76	8.27
318	-1.22		
328	-1.84		
338	-2.13		

CONCLUSION

The adsorbent derived from the coconut husk was treated with sodium hydroxide solution. It was evaluated for its ability to remove BPA from effluents. The most efficient conditions for the coconut husk to remove BPA maximally were at a temperature of 330K, a pH of 2, a dosage of 2 g, a contact time of 80 min, and an initial solution concentration of 100 ppm. The alkaline treatment of the adsorbent enhanced the electrostatic repulsion between the BPA and the adsorbent; while modifying the surface characteristic of the adsorbent it also led to a higher negative charge that aids its ability to bind. The activated coconut husk showed the highest R2 value for BPA

adsorption based on the Freundlich model, which was 0.99. The equilibrium adsorption capacity for BPA (qe) for the banana shaft was recorded as 9.13 mg/g, closely aligning with the figure obtained from the pseudo-second-order model at 9.15 mg/g. At the same time, the R2 value for the pseudo-second-order model was 0.99, indicating a better match compared to the pseudo-first-order model. Thus, it is implied that the overall rate of BPA adsorption takes place through chemisorption with the adsorbent proposed.

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