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Adsorption Efficiency of Flamboyant Pods for Indigo Dye Removal from Textile Industrial Wastewater

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Abstract

This study investigates flamboyant pods (FP) and chitosan [extracted from periwinkle shells (PS)] modified flamboyant pods (CMFP) adsorbents for dye removal from textile industrial wastewater, and were compared with commercial activated carbon (CAC). Physicochemical properties with dye concentrations of wastewater were investigated before and after adsorption using standard methods and Ultraviolet-visible Spectrophotometer respectively. Batch adsorption were performed and pH (3.0, 4.0, 6.0, 9.0, 11.5), adsorbent dosage (0.1, 0.2, 0.3, 0.4, 0.5 g), contact time (10, 20, 30, 40, 50, 60 minutes) and initial concentration (25, 50, 100, 125, 250 mg/L) were optimized for Indigo dye using the adsorbents. Initial concentration data was used to test conformity with Langmuir and Freundlich adsorption isotherms. Adsorption efficiencies for simulation ranged from 11.33±0.70 to 83.8±0.00. Optimum adsorption conditions of indigo dye were pH 6, 0.1g sorbent dosage, 60 minutes contact time and 250 mg/L dye concentration; gave efficiencies of 83.8%, 79.6% and 89.8% for FP, CMFP, CAC respectively with wastewater. Physicochemical parameters of wastewater decreased except nitrate which increased from 11.53±0.00 to 34.65±1.41mg/L. Data best fit Langmuir than Freundlich adsorption isotherm. The study inferred that FP and PS could be processed as less expensive, environment friendly alternative adsorbent to the costly CAC for treating textile wastewater.

Keywords: adsorption, flamboyant, indigo dye, textile, UV- Visible Spectrophotometer, Nigeria

1. Introduction

Industrial developments in recent years have left their mark on the different environmental matrixes. Many industries like the textile industry used dyes to colour their products and thus produce wastewater containing organics with a strong colour. Discharge of these dyes into water bodies affects the people who may use the water for domestic purposes such as washing, bathing and drinking [1]. Therefore, it is very important to assess the water quality for a particular use, especially since 1.0 mg/L of dye concentration in drinking water could impart a significant colour, making it unfit for human consumption [2]. Furthermore dyes can affect aquatic plants because they reduce sunlight transmission through water to the plants. Also dyes may become toxic to aquatic life and may be mutagenic, carcinogenic and may cause severe damage to human beings, such as dysfunction of the kidneys, reproductive system, liver, brain and central nervous system [3, 4, 5].

Activated carbon is one of the adsorbents most commonly used to treat industrial effluents, because it is capable of adsorbing a great amount and variety of contaminants on its surface, concentrating and removing them from such effluents. The raw materials traditionally used to prepare activated carbon at an industrial level are wood, mineral carbon, coconut shell, and animal bones [6]. However, the associated environmental problems with the use of these raw materials peculiar to each country have motivated researchers to propose alternative materials to prepare both carbonaceous and lignocellulosic adsorbents from other residues especially “waste”, and thus decrease the consumption of wood or mineral carbon. For example, biochar have been accounted to be produced from various agricultural waste and they find application for example, in water and wastewater treatment thereby turning into another financial asset for agriculturists [7] and environmentalists, scientists and the general populace. In the case of Mexico, it has been proposed to use avocado [8], mango, orange, and guava seeds [9], both in natural and carbonized forms to adsorb dyes. China has recommended the use of powdered peanut hull [10], and Mexico has suggested the use of Opuntia ficus-indica fruit waste [11] as cellulosic adsorbents for dyes removal. Brazil, Egypt, and Poland have proposed the use of Flamboyant (Delonix regia) pods [12], Loofa egyptiaca [13], and corncob [14] respectively, to prepare activated carbons. The effluents from the textile industry pose a serious problem concerning environmental pollution [9] because of the high volume of highly coloured discharges containing a large variety of chemical products, dyes included [11]. Wastewater from textile industries requires proper treatment before being released into the environment. A large amount of dyes, particularly those containing azo-chromophore groups, and their by-products of degradation have been proved to be toxic or carcinogenic [15].

Agricultural waste products are suitable for adsorption. The main advantage of adsorption using the low-cost materials as adsorbent is lowering of the procedure cost [16]. The agricultural waste materials have been used in their natural form or after some physical or chemical modification or by conversion into activated carbon [17]. Hence, this study seeks to determine and optimise the conditions for the effectiveness of flamboyant pods as adsorbent for the adsorption of dye from wastewater which, being waste materials, are cost effective and are more environment friendly.

2. Materials and Methodology

2.1 Collection, Preparation and Carbonization of Sample

The flamboyant Pods (FP) were collected within Obafemi Awolowo University campus, Ile-Ife, Osun State, Nigeria. The flamboyant Pods were washed to remove foreign debris, sun-dried and pulverized to increase the surface area for activated carbon production [18, 19,]. The pulverized known weight (100 g) of the flamboyant pods were prepared by placing the raw material in a crucible, and carbonized in a furnace (Carbolite RHF 1600) at 500 ºC for 1 hour and sieved to <300 µm particle size[18, 20]. Periwinkle shell (PS) was obtained from backyard dumpsite in Uyo, Akwa-Ibom State, Nigeria and commercial activated carbon (GAC F-300) was obtained from a chemical store.

2.2 Chemical Activation of the Carbon

The method is as outlined by [19, 20, 3]. The charred FP (100 g) were soaked in 2% H_2SO_4 (v/v) and placed in an oven at a temperature of 110 ºC for 24 hours. After cooling, the samples were then soaked with distilled water and in 2% $NaOH_3$ (w/v) in turns to remove the residual acid and then washed with distilled water again till neutral pH was attained. The sample was finally dried in an oven at 110 ºC, cooled at room temperature and used as an adsorbent.

2.3 Preparation of Chitosan

2.3.1 Preparation of Chitin

The collected PS was washed to remove foreign debris, sun-dried, pulverized into powered form and sieved <2mm size fraction. The powdered PS (50 g of less than 2 mm particle size) was weighed into a 500 ml beaker and deproteinized by adding 200 mL of 4 % (w/v) KOH, stirred on a magnetic stirrer for 6 hours at 80 ºC and filtered. The residue obtained was washed with distilled water until it is free of base and then dried at 100 ºC for 2 hours. [21]. The residue was then demineralised and decolourised [21, 20] to obtain chitin.

2.3.2 Deacetylation of Chitin

Chitin was deacetylated with 50 % (w/v) NaOH solution in a 250 ml conical flask. The flask with its content was placed on a magnetic stirrer at 30 ºC for 4 hrs. After filtration, the residue, which is Chitosan (2-acetamido-2-deoxy-β-D-glucose-N-(acetylglucosamine), was then washed and dried at 90 ºC for 1 hour [21, 12] and processed into chitosan gel (a white viscous gel) [21,8].

2.4 Modification of the Adsorbent

The adsorbent was modified by slowly adding 100 ml of chitosan gel to 50 g of flamboyant pods (FP) char. After dilution and agitation using a mechanical shaker operated at 200 osc/min for 24 h, the chitosan coated adsorbent was washed, dried and then soaked in 0.5 % (w/v) NaOH solution for 3 h. It was then extensively rinsed with distilled water and dried in an oven at 102 ºC for 2 h, cooled at room temperature and stored in desiccator [22, 21].

2.5 Characterization of the Adsorbents

The elemental composition and the surface morphological characteristics of the FP activated carbon and chitosan modified flamboyant pods (CMFP) were determined based on dry combustion method using Scanning Electron Microscope coupled with Energy Dispersive X-ray Fluorescene Analyzer (High resolution SEM/EDX-RF Carl Zeiss). Structural chemical functional groups of chitosan were determined using the Fourier Transform Infrared Technique (FTIR, Nicolet IS5) [19].

2.6 Batch Adsorption Experiment of Simulated Indigo dye Wastewater

The batch adsorption studies for the removal of indigo dye were carried out according to the method of [23]. Indigo dye polluted wastewater were simulated in the laboratory by preparing standard solutions of the dye with (25, 50, 100, 125, and 250 mg/L) concentrations. Batch adsorption studies were carried out using 0.1, 0.2, 0.3, 0.4 and 0.5 g of the adsorbent made from flamboyant pods and chitosan modified flamboyant pods activated carbon and 10 ml each of the simulated solution of indigo dye wastewater in different conical flask with constant shaking using a mechanical shaker (Stuart Scientific) operated at a speed of 500 rpm. In addition to the concentration and adsorbent dosage, contact time and pH were also optimized for efficient adsorption. The indigo dye content of the filtrates collected after adsorption were determined using Ultraviolet-visible Spectrophotometer. The data obtained were subjected to one way analysis of variance.

2.7 Characterisation of the Textile Wastewater

The physicochemical properties of the textile wastewater samples were determined using standard methods and its dye concentration was determined using Ultraviolet-visible Spectrophotometer.

2.8 Adsorption Efficiency

The percentage of the dye adsorbed (adsorption efficiency %) was determined for each of adsorption process carried out using the following formula: [24, 22, 25].

$$Adsorption\ Efficeincy\ = \frac{(C_0 - C_1)}{C_0}\ \times 100\%$$

Where:

C_0 is the initial concentration before adsorption

C_1 is the final concentration of dye solution (mg/L) in the filtrate after adsorption.

3. Results and Discussion

3.1 Carbonisation of Flamboyant Pods

In the result of carbonisation, 100 g of flamboyant pod (FP) has a percentage yield of 9.657±0.299 % carbon with 90.343g of volatile organic compound. This eventually gave 10.35 g of activated carbon. Also, 50 g of periwinkle shell (PS) yielded 34.25±3.934% amount of chitosan with no volatile organic compound. These results shows that FP that litters ground around is a rich source of carbon that can be harnessed for different uses; PS, which is non-biodegradable in nature, is a good, cheap and available raw material for the production of chitosan.

3.2 Characterization of Activated Carbon

3.2.1 Elemental Composition of Flamboyant Pods Char before Adsorption Experiment

Flamboyant pods char was analyzed using Rutherford backscattering Spectrometry (Energy Dispersive X-ray fluorescence) to determine its elemental composition of the adsorbent before adsorption experiment. The results shows that carbon (44.27%) and oxygen (34.2%) were found in higher percentages compared to other elements (sodium (0.38%), Magnesium (0.82%), Aluminium (1.5%), silicon (0.43%), phosphorus (0.81%), sulphur (0.43%), chlorine (1.16%), potassium (10.22%), calcium (1.7%) and iron (1.61%) present, and this makes the flamboyant pods activated carbon suitable for adsorption [26,19]. The presence of other elements in the adsorbent enhances adsorption through adsorption mechanism such as ion exchange, chelation, co-ordination and complexation reactions [26, 21].

3.2.2 Characterization of the Chitosan by FTIR

Fourier transform infrared spectroscopy (FTIR) was used to examine the functional groups of the chitosan produced and to ascertain the presence of some characteristic functional groups [28]. Figure 1 shows the peaks that indicate the functional groups present in the chitosan extracted from periwinkle shell. The peak around 1082 cm^{-1} represents C─O, The 1492 cm^{-1} peak was due to the CH_2 bonding stretching frequency and the band at 1467.54 cm^{-1} should be attributed to amine (─NH_2) of chitosan [29]. All peaks detected conform with the functional groups of chitosan which confirms complete deacetylation of chitin to chitosan [30, 31, 32].

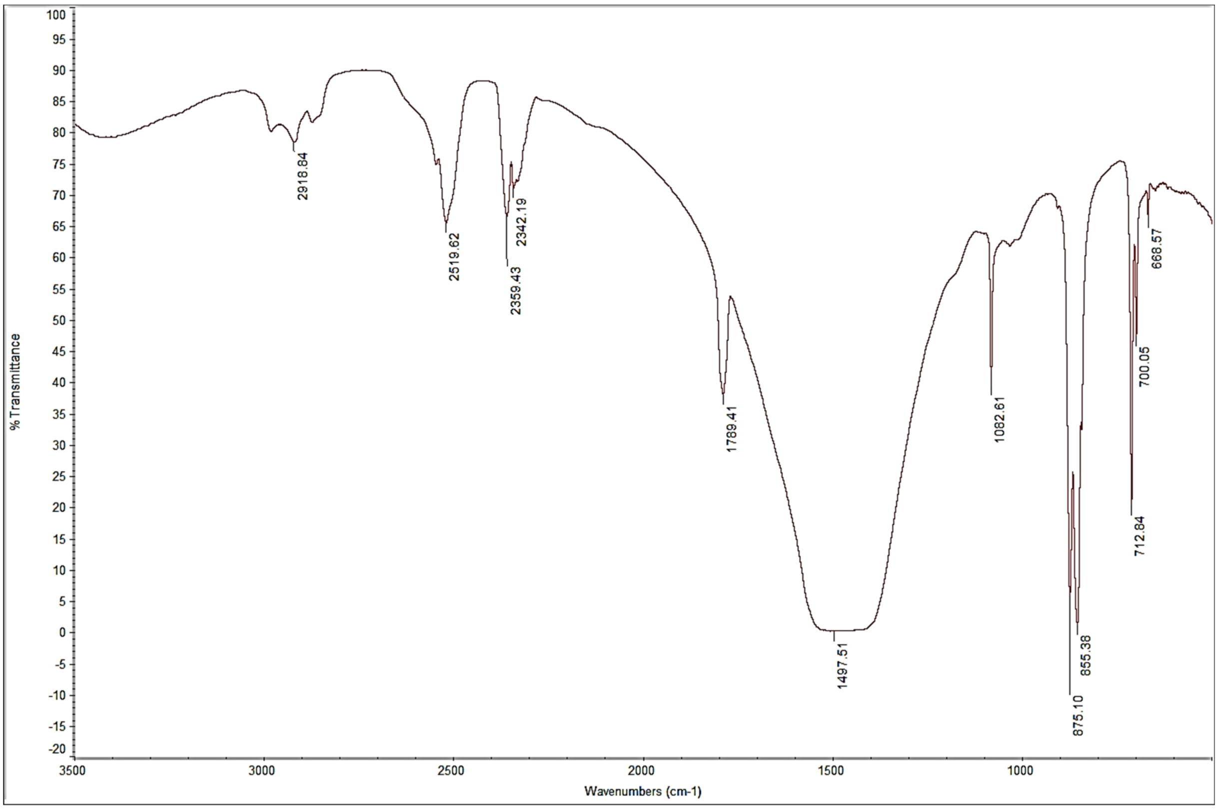

Figure 1. Fourier Transform- Infra Red sprectrum (FTIR) of chitosan extracted from periwinkle shells

3.2.3 Surface Morphology of Flamboyant pod (FP) and Chitosan Modified Flamboyant Pod (CMFP) Activated Carbon

The physical morphologies and surface properties of the FP and CNFP were examined using scanning electron microscopy technique (SEM). SEM is a primary tool for characterizing the surface morphology and fundamental physical properties of the adsorbent surface [27]. Figures 2 and 3 compared the surface morphology of the FP activated carbon before and after modification with chitosan. It was observed that the chitosan used to modify the flamboyant pod activated carbon could be seen as the white patches on the surface of the carbon. The surface of modified flamboyant pod activated carbon contained more pores than the surface of the unmodified adsorbents and this results in higher surface area.

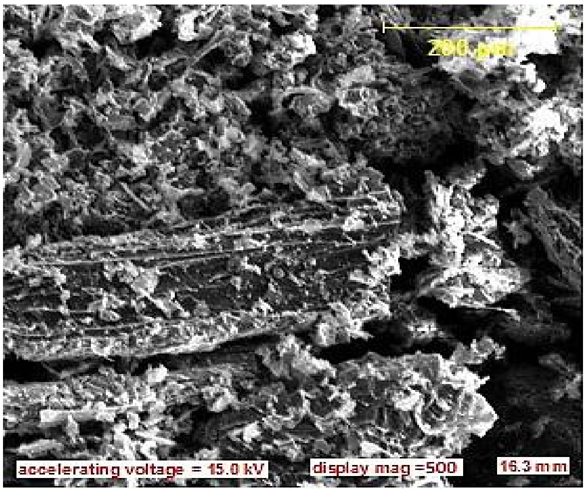

Figure 2. SEM micrograph of flamboyant pod char

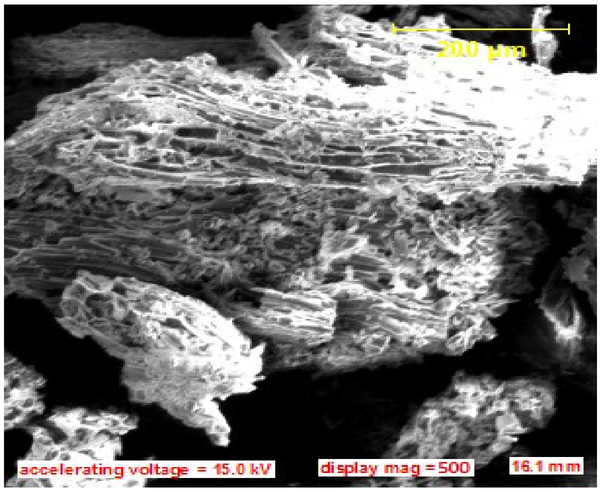

Figure 3. SEM micrograph of chitosan modified flamboyant pod char

The results of the physicochemical parameters of textile industry wastewater samples before and after adsorption are presented in Table 1. This becomes necessary to know the effect of the adsorption of dye on the physicochemical properties.

Table 1. Physicochemical Analysis of Textile Industry Wastewater before and after adsorption

Parameters	Units	Wastewater before Adsorption	Wastewater after Adsorption with Modified Flamboyant Pod	WHO Standards Guideline Values
pH	-	6.06	9.24	6.5
Conductivity	µs/cm	441.75	135.00	2500
Nitrate	mg/L	11.53	34.65	0
Sulphate	mg/L	96.76	2.33	400
Calcium	mg/L	20.58	9.7	75
Magnesium	mg/L	0.050	0.041	20 – 50
Total hardness	mg/L	51.594	22.39	150
Total dissolved solids	mg/L	265.00	81.00	500
Lead	mg/L	0.472	0.025	0.01

The absorbance of the industrial wastewater was determined using the UV spectrophotometer and the concentration was then determined using the Regression equation from the Calibration curve of the standards of the dye solution. The concentration obtained was then used to vary the concentrations of the dye in the textile wastewater (10, 20 and 30 mg/L) used.

3.3 Batch Adsorption studies on Simulated Wastewater of Indigo Dye using Flamboyant Pods, chitosan modified flamboyant pods and Commercial Activated Carbon Adsorbents

The effect of FP and CMFP activated carbon as adsorbents for the adsorption of Indigo dye was explored. The parameters (for example, initial dye concentration, adsorbent dosage, contact time and pH) influencing adsorption studies were examined using the flamboyant pod based adsorbents in comparison with the commercial activated carbon. The discussion of the effects of each of the parameters follows:

3.3.1 Effect of Initial Dye Concentration

The effect of the various initial concentration (25, 50, 100, 125 and 250 mg/l) of indigo dye on the adsorption process using flamboyant pods, chitosan modified flamboyant pods and commercial activated carbon are presented in Figure 4. The adsorption of dye by each of the adsorbent are significantly different for different initial concentrations, with the adsorption efficiency maximum (93.8%) at 250 mg/L dye concentration for commercial activated carbon. while the lowest (4.67%) was recorded at 25 mg/L surprisingly for modified flamboyant pod. This trend is in agreement with [17].

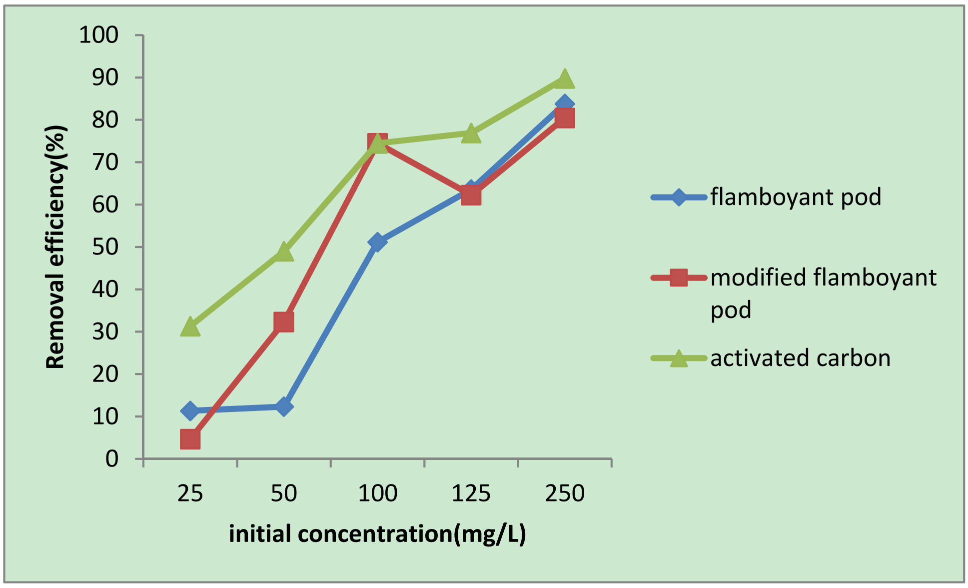

Figure 4. Effect of initial dye concentration on adsorption of indigo dye

3.3.2 Effect of Adsorbent Dosage on Adsorption of Indigo Dye

The result of different adsorbent dosages for the adsorption of indigo dye by flamboyant pods, modified flamboyant pods activated carbon and commercial activated carbon is presented in Figure 5 while all other conditions (pH, initial concentration and contact time) were kept constant. Dosage of adsorbent is one of the important parameters used to determine the adsorbent's capacity to adsorb a particular adsorbate at the operating conditions [33]. There is no clearly defined trend in the adsorption efficiencies for the three adsorbents, however the flamboyant based adsorbents had about 75% adsorption efficiency within adsorbent dosage of 0.2 and 0.4g while the commercial activated carbon was about 80% for the same range of adsorbent dosage.

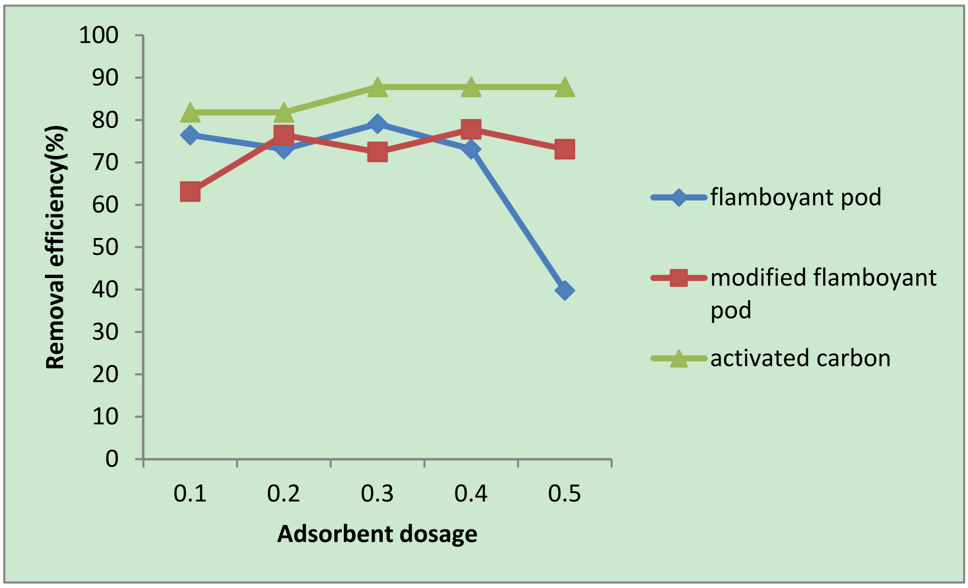

Figure 5. Effect of adsorbent dosage on adsorption of indigo dye

At 0.3 g, flamboyant pod reached its optimum of 79.13% and thereafter there was a decrease in removal efficiency to a value of 39.8%. Commercial ativated carbon also reached its optimum at 0.3 g and remained in equilibrium while modified flamboyant pod had a removal efficiency of 77.8% at 0.4 g and thereafter there was a decrease in removal efficiency to 73.13%. This may be attributed to saturation of adsorption sites on adsorbent surface due to particulate interaction such as aggregation, aggregation would lead to a decrease in total surface area of the adsorbent [28].

3.3.3 Effect of Contact Time on Adsorption of Indigo Dye

The results of the adsorption of indigo dye on flamboyant pod, modified flamboyant pod and commercial activated carbon by varying contact time (10, 20, 30, 40, 50 and 60 minutes) are presented in Figure 6 while other conditions (pH, adsorbent dosage and concentration) were kept constant.

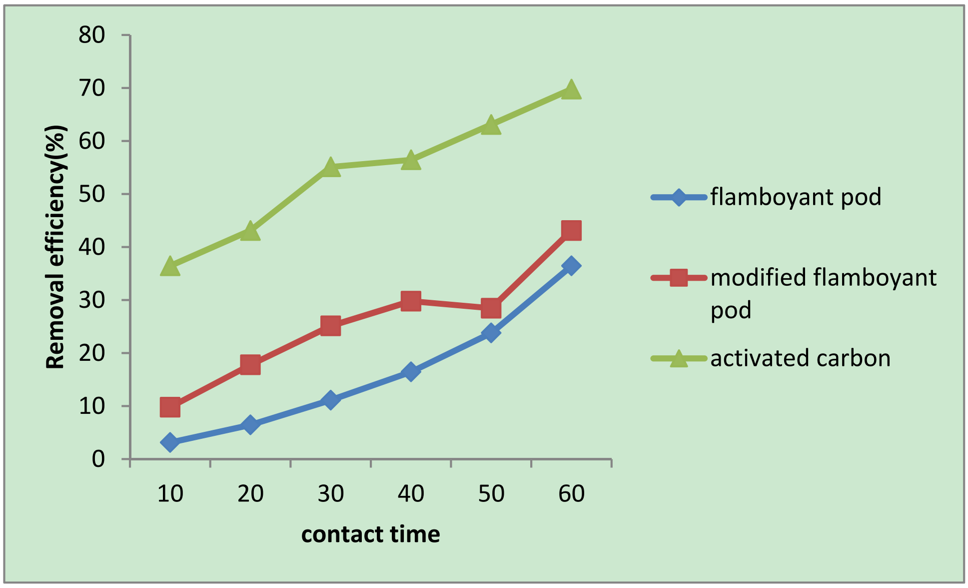

Figure 6. Effect of contact time on adsorption of indigo dye

Contact time was varied from 10 to 60 minutes, and it was observed that across the adsorbents, removal efficiency was optimum at 60 minutes. The lowest removal efficiency value of 3.14% was observed for flamboyant pod with commercial activated carbon having the highest adsorption efficiency value of 69.8%. The increase in removal efficiency with increase in contact time for the adsorbents may be due to increased interaction between indigo dye and adsorbent [34].

3.3.4 Effect of pH on Adsorption of Indigo Dye

The results of the investigation on the adsorption of indigo dye on flamboyant pod and modified flamboyant pod as well as commercial activated carbon with varied pH (3, 4, 6, 9, and 11.5) are presented in Figure 7 while all other conditions (adsorbent dosage, contact time and concentration) were kept constant. pH is a measure of acidity (pH < 7) or basicity (pH > 7) of an aqueous solution. The pH factor is very important in the adsorption process especially for dye adsorption. The adsorption of dye by both flamboyant pod and modified flamboyant pod adsorbents were generally high, there was higher adsorption efficiency from pH 3.0 to 6.0. Higher uptakes obtained at lower acidic pH range may be due to the electrostatic attractions between negatively charged functional groups located on the reactive dye and positively charged adsorbent surface. The change in solution pH will affect the ionization of these functional groups [35, 36].

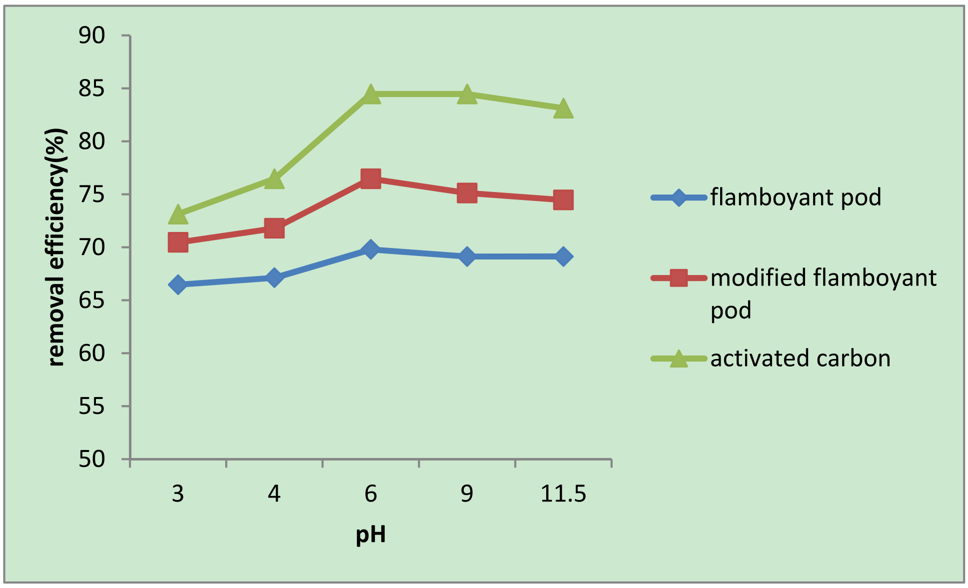

Figure 7. Effect of pH on the adsorption of indigo dye

3.4 Adsorption of Indigo Dye from Textile Industrial Wastewater using Flamboyant Pods and Chitosan Modified Flamboyant Pods Adsorbents

Adsorption studies were conducted on wastewater from the textile industry using flamboyant pods, chitosan modified flamboyant pods and commercial activated carbon adsorbents with the optimised conditions (pH -6.0, adsorbent dosage - 0.3 and 60 minutes contact time) obtained from simulated experiments. The indigo dye concentration of the industrial wastewater sample was determined using Ultraviolet-visible Spectrophotometer (Schimadzu uv-2700 model). Figure 8 shows the results of the adsorption process for the textile wastewater. It shows that the amount of dye adsorbed per unit mass (q) of flamboyant pods adsorbent increased from 0.068 mg/g to 0.335 mg/g. The amount of dye adsorbed per unit mass (q) of modified flamboyant pod increased from 0.089 mg/g to 0.339 mg/g. The best removal efficiency was observed at 30 mg/L initial concentration of the textile wastewater. The increase in the amount of dye adsorbed per unit mass of the adsorbents with increase in dye concentration is because a higher initial concentration enhanced the driving force between the aqueous and solid phases and increased the number of collisions between dye molecules and the adsorbent [37]. The enhanced performance of the modified adsorbents is due to the presence of chitosan on the flamboyant pod activated carbon [29, 38, 21].

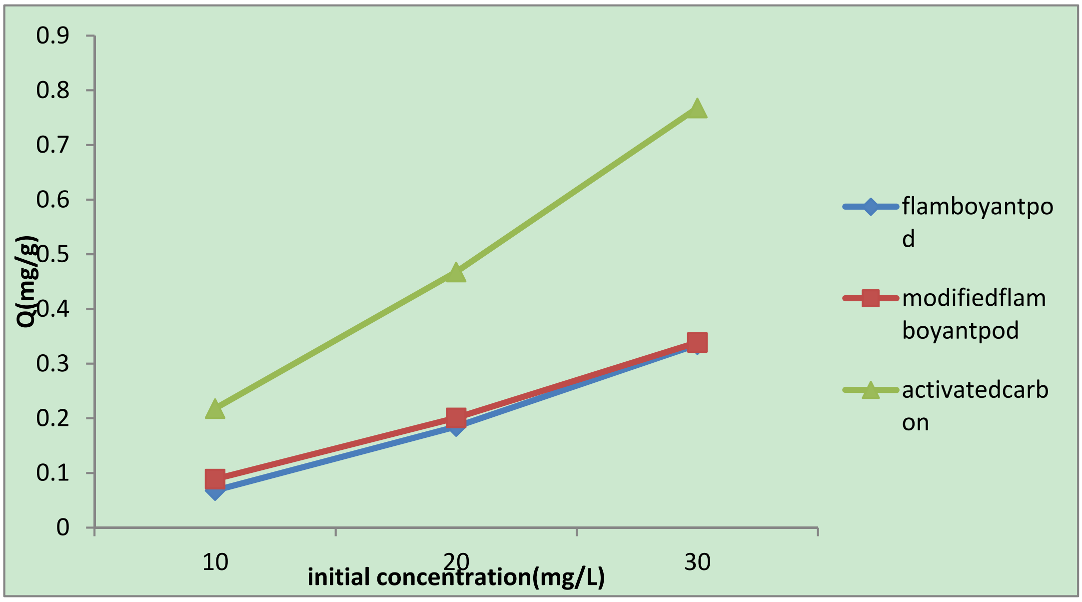

Figure 8. Adsorption of dye from Industrial wastewater

3.5 Adsorption Isotherm

The results of the sorption ability of flamboyant pod and modified flamboyant pod adsorbent was evaluated through determination of adsorption isotherm of indigo dye sorption system and were presented in Figures 9 and 10 (Langmuir isotherm) and Figures 11 and 12 (Freundlich isotherm) while Tables 2 and 3 show the coefficients of these isotherms (Langmuir and Freundlich) respectively. The results show that the adsorption process is suitable and well fitted with Langmuir isotherms model than Freundlich for adsorption of dye from the simulated wastewater.

The adsorption isotherm indicates how the adsorbed molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state [39]. The adsorption capacity is usually predicted from equilibrium sorption isotherm [40].

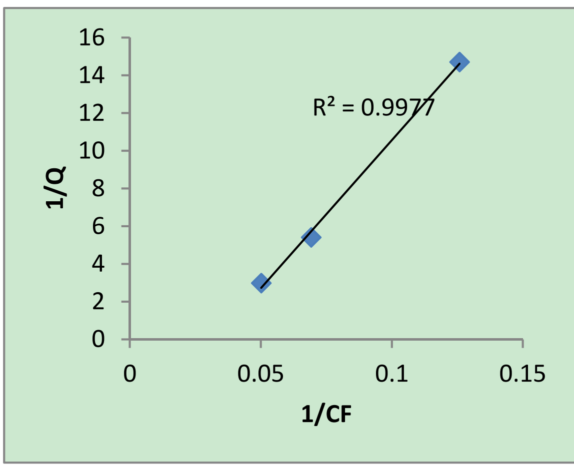

Figure 9. Langmuir isotherm for textile

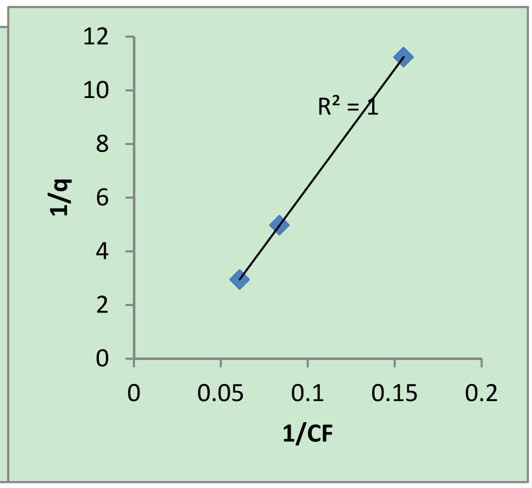

Figure 10. Langmuir isotherm for textile

Table 2. Langmuir adsorption isotherm constants for the adsorption of Indigo dye using flamboyant pod adsorbent and modified flamboyant pod adsorbent

Adsorbents	**Langmuir Constants**		
	b (L/mg)	Qmax (mg/g)	R^2
flamboyant pod adsorbent	-0.03272	-0.19459	0.9977
Modified flamboyant pod adsorbent	0.027154	0.419076	1

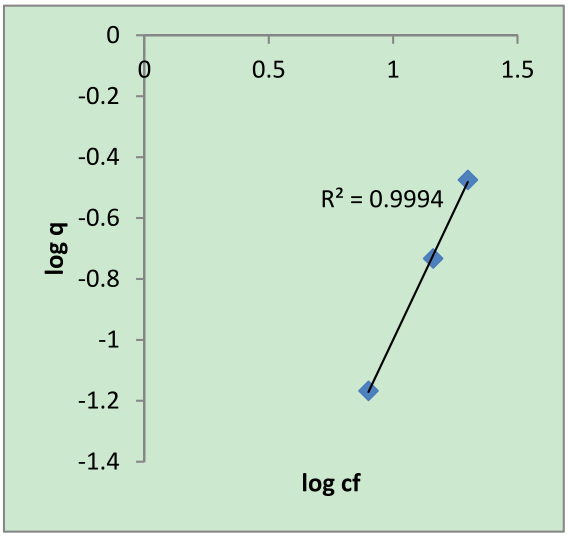

Figure 11. Freundlich isotherm for textile modified flamboyant pod

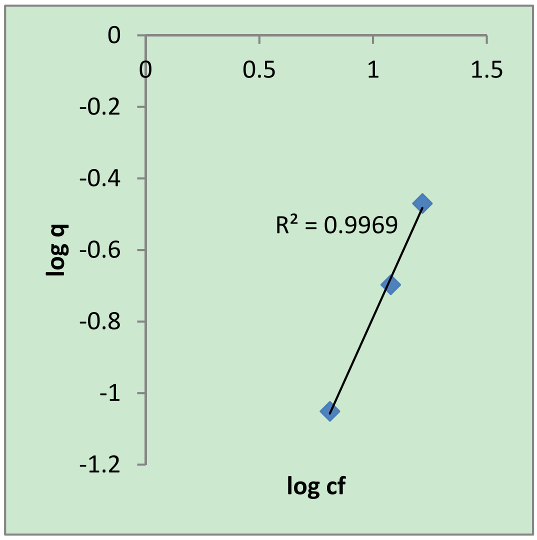

Figure 12. Freundlich isotherm for textile wastewater onto wastewater onto flamboyant pod

Table 3. Freundlich adsorption isotherm constant for the adsorption of Indigo dye using flamboyant pod adsorbent and modified flamboyant pod adsorbent

Adsorbents	**Freundlich constants**		
	K	1/n	R^2
flamboyant pod char	1.88E-03	1.7259	0.9994
modified flamboyant pod char	6.29E-03	1.414	0.9969

4. Conclusion

This study concluded that flamboyant pods activated carbon acts as an efficient adsorbent with improved performance upon modification with chitosan extracted from periwinkle shells. The adsorption process is effective at 0.3g dosage, pH of 6.0 at 250mg/L concentration for 60 minutes.

5. Recommendation/Further Work

The study recommends the consideration of flamboyant pods and periwinkle shells as economic and environment friendly adsorbent for dye removal in textile wastewater. The commercial production of activated carbon from these natural materials by small scale business investors can improve the economy of the country. Government and industries should take advantage of adsorption technologies by developing these natural adsorbents for household purposes.

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Data Availability

The data used to support the findings of this study are included within the article.

Ethical Issues

Authors have declared no ethical issues in the manuscript.

Conflicts of Interest

Authors have declared no conflict of interest in the manuscript.

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