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Studies of Flow Rate and Bed Height on the Fixed Bed Adsorption of Methylene Blue Dye, Bismarck Brown Y, and Indigo Dyes on to *Sphagnum Cymbifolium* (MOSS)

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ABSTRACT

The adsorption behavior of Methylene blue dye, Bismarck brown Y dye, and Indigo dye on to *Sphagnum cymbifolium* (moss) was investigated as a function of flow rate, and bed height through the fixed bed process.

One of the main objectives of this research is to expand the field of application of natural biomass for the treatment of dye waste waters from industrial effluents. Additionally, it is aimed at elucidating the dependency or otherwise of flow rate, and bed height on adsorption using the fixed bed technique method of adsorption.

The biomass was characterized by Scanning Electron Microscopy in order to examine the morphology of the biomass. The screened biomass samples were characterized at 1000 ×magnification, and 500 ×magnification respectively for their surface morphologies. This was done using a scanning electron microscope which was equipped with an energy dispersive x-ray spectrophotometer employed for the elemental composition analyses. It was equally characterized with Fourier Transformed Infrared Spectroscopy before and after adsorption to ascertain the functional groups responsible for the adsorption. This was done using a Fourier Transformed Infrared Spectrophotometer (Perkin-Elmer, England) in the wavelength range of 350-4000 nm.

Results for the biomass morphology obtained through the Scanning Electron Microscopy revealed the presence of tiny pores. These pores represent sites where dye molecules could be trapped in the course of the adsorption. The result from the Fourier Transformed Infrared Spectroscopy after adsorption show that C-H, C≡H, C≡C functional groups were responsible for the adsorption. For methylene blue dye, at the flow rate of 20 m^3/s, the amount of dye adsorbed was 18.80 mg/g, 22.70 mg/g at 30 m^3/s, and 25.40 mg/g at 40 m^3/s. For Bismarck brown Y dye, at the same range of flow rate, the amount of dye adsorbed ranged from 12.34 mg/g - 20.62 mg/g. For indigo dye, the values obtained ranged from 6.48 mg/g - 17.71 mg/g. In addition, at the bed height range of 4.0 - 6.0x10^{-2}(m), the amount of dye adsorbed ranged from 6.31 mg/g - 27.73 mg/g for methylene blue dye. Within the same range of bed height, the amount of dye adsorbed ranged from 16.40 mg/g - 25.60 mg/g for Bismarck brown y dye, and 12.57 mg/g - 17.71 mg for indigo dye.

Keywords*: Bio-sorption, Sphagnum cymbifolium, SEM, Adsorbent, Fixed bed, Dyes.*

1. Introduction

Bio-sorption can be defined as the sequestering of organic and in organic species by the use of live or dead biomass or their derivatives. This can be achieved through the batch or fixed bed techniques. But this work is based on achieving it through the fixed bed technique. This is achieved by allowing the dye solution to pass through a column containing the biomass from down of the column to the top by the use of a peristaltic pump. The use of synthetic organic dyes as coloring materials in textile and paper industries has been on the increase [1]. The annual world production of textile is about 30 million tones requiring 700,000 tons of different dyes which cause considerable environment problems [2].

These colored compounds usually contain molecules with aromatic structures which make their degradation difficult. The presence of very small amount of dyes in water is highly visible and undesirable. It is therefore necessary that they should be removed from waste waters before they are discharged to the environment.

Adsorption techniques are effective and attractive for the removal of non- biodegradable pollutants (including dyes) from waste waters [3]. Most commercial system use activated carbon as adsorbent to remove dyes from waste waters because of its excellent adsorption ability, but its wide spread use is limited due to its high running cost. Many low cost adsorbents including natural material adsorbent and waste materials from industry and agriculture have been proposed by several workers [4]. These materials do not require any expensive additional pretreatment step and could be used as adsorbent for the removal of dyes from solutions. For instance, a fresh water algae *pithophora sp* was studied by researchers [1] in finding out its bio-sorption properties on to malachite green (a cationic azo dye). In order to expand the field of application of natural biomass for the treatment of dye waste waters, and also to determine the adsorption capacity of *Sphagnum cymbifolium* (moss) on cationic, anionic and neutral dye molecules, this research work becomes more imperative since such comparison has not been done on this biomass. The results obtained from the work could add to the expansion of knowledge in this area.

2. Materials and Methods

The Methylene blue dye, Bismarck brown Y, and Indigo dye used in these investigations were obtained from qualikem laboratory, Owerri, Nigeria. Other materials obtained from here include analytical grade sodium hydroxide pellets, concentrated hydrochloric acid, distilled water etc. The *Sphagnum cymbifolium* (moss) used in this work was obtained from Ikorodu area of Lagos, Nigeria which lies within the following coordinates 6.6194º N and 3.5105º E. The sample was identified at the department of crop science at the Federal university of technology, Owerri, Nigeria with the voucher number of FUT/CR/003/15.

The biomass was washed severally with distilled water to remove any dirt from it. The washed biomass was air dried for ten days until constant weight was obtained. The biomass was grinded with a new sonic domestic blender to avoid any form of contamination. It was screened using 600-850 micron sized sieves and were stored in air tight containers ready for adsorption measurement.

The methods and techniques employed in this research are the standard methods which have been employed by other researchers [5].

2.1. Characterization of the Bio-Sorbent

The surface structure and morphology of the *Sphagnum cymbifolium* (moss) was characterized at 1000 ×magnification, 500 ×magnification and 250 × magnification respectively for their surface morphologies. This was done using scanning electron microscopy (SEM) (FEI- inspect oxford instruments x-max) which was equipped with an energy dispersive x-ray (EDAX) spectrometer employed for the elemental composition analysis. The biomass sample was further characterized for their fundamental functional groups before and after adsorption experiments using a Fourier Transform Infrared (FTIR) spectrophotometer (Perkin Elmer, England) in the wavelength range of 350-400 nm using KBr powder and fluka library for data interpretation.

2.2. The Fixed Bed Setup

The fixed bed was set up by packing wire gauze, glass wool, glass beads, glass wool, biomass and glass wool in that order in a graduated condenser. A dye solution of a known concentration and pH pressurized from down to top where a known amount of the bio-sorbent is placed with a peristaltic pump (CHEM-TECH, Model X030-XB-AAAA365, China). Subsequently, a sample was collected for analysis in a U.V spectrophotometer

(CAMSPEC M106 Model, England) by monitoring the absorbance changes at wavelength of maximum absorbance already determined for Methylene blue dye (600 nm), Bismarck brown Y dye (320 nm), and Indigo dye (360 nm) respectively. The variables investigated here include the effect of bed height and flow rate. The analysis was done in triplicates and mean values with the standard deviations reported.

2.3. Effect of Flow Rate on Adsorption

Experiments were carried out at different flow rates of 20 m^3/s, 30 m^3/s and 40 m^3/s while keeping constant the value of 1×10^{-2} m bed height, 40 mg biomass dose and 90 mg/L dye solution, and pH of 4 for Methylene blue dye, and pH of 2 for Bismarck brown Y, and Indigo dyes earlier determined as their best pH of maximum adsorption. The dye solution was subjected to pass through the column already prepared using the peristaltic pump. The samples collected were subjected to U.V analysis using a U.V Spectrophotometer (Camspec 106 Model, England) for absorbance measurements. Subsequently, the absorbance values were converted to concentration by the use of Beer Lambert's law. Similar experiment were carried out in triplicates and the mean values with their standard deviations reported.

2.4. Effect of Bed Height on Adsorption

Experiments were carried out at different bed heights. Bed height of 4×10^{-2} m, 5×10^{-2} m and 6×10^{-2} m were considered while keeping constant a flow rate of 10 m^3/s, 90 mg/L dye solution, pH of 4 for Methylene blue dye, and pH of 2 for Bismarck brown Y, and Indigo dyes earlier determined as their best pH of maximum adsorption.

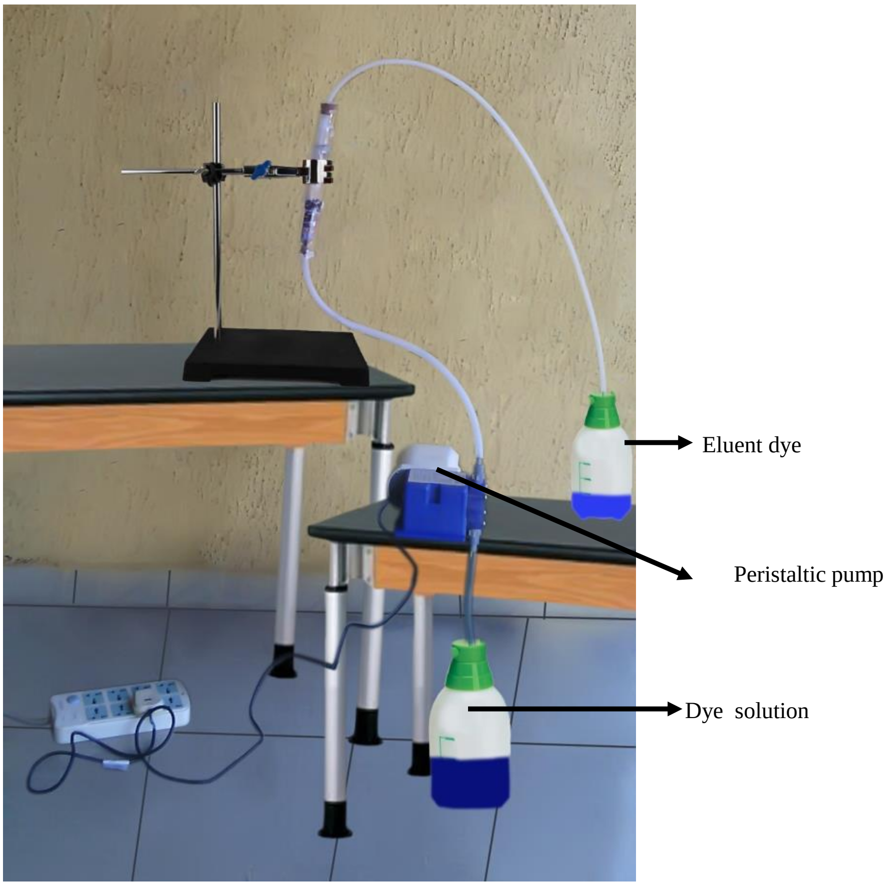

Fig.1. Fixed bed technique apparatus

The dye solution was subjected to pass through the column already prepared using peristaltic pump. The samples collected were subjected to U.V analysis for absorbance measurements. Subsequently, the absorbance value were converted to concentration by the use of Beer Lambert's law. Similar experiments were carried out in triplicates and the mean values with their standard deviations reported.

NOTE: The amount of dye adsorbed per gram biomass (q_e) was calculated using the expression below: $q_e = \frac{V(Co-Ce)}{M}$

Where V= Volume of the sample in dm^3

C_o= Initial dye concentration in mg/L

C_e= Equilibrium dye concentration in mg/L

M= Mass of the biomass in g

3. Results and Discussions

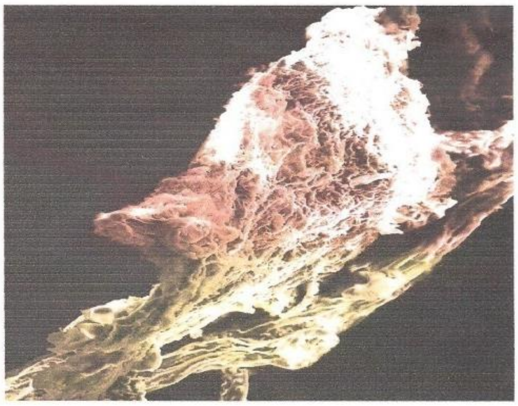

Fig.2. SEM Morphology of *Sphagnum cymbifolium* (moss) (×500)

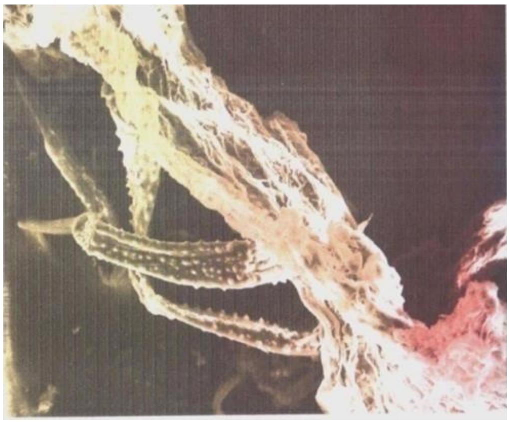

Fig.3. SEM Morphology of *Sphagnum cymbifolium* (moss) (×1000)

The scanning electron microscopy (SEM) micrographs of *Sphagnum cymbifolium* (moss) at (×500) magnification and (×1000) magnification shown in figure 2 and 3 reveal the presence of unevenly dispersed granules or cavities on the surface of the biomass. These cavities on the surface of the biomass provide sites where molecule of the dyes could be trapped thus giving rise to adsorption.

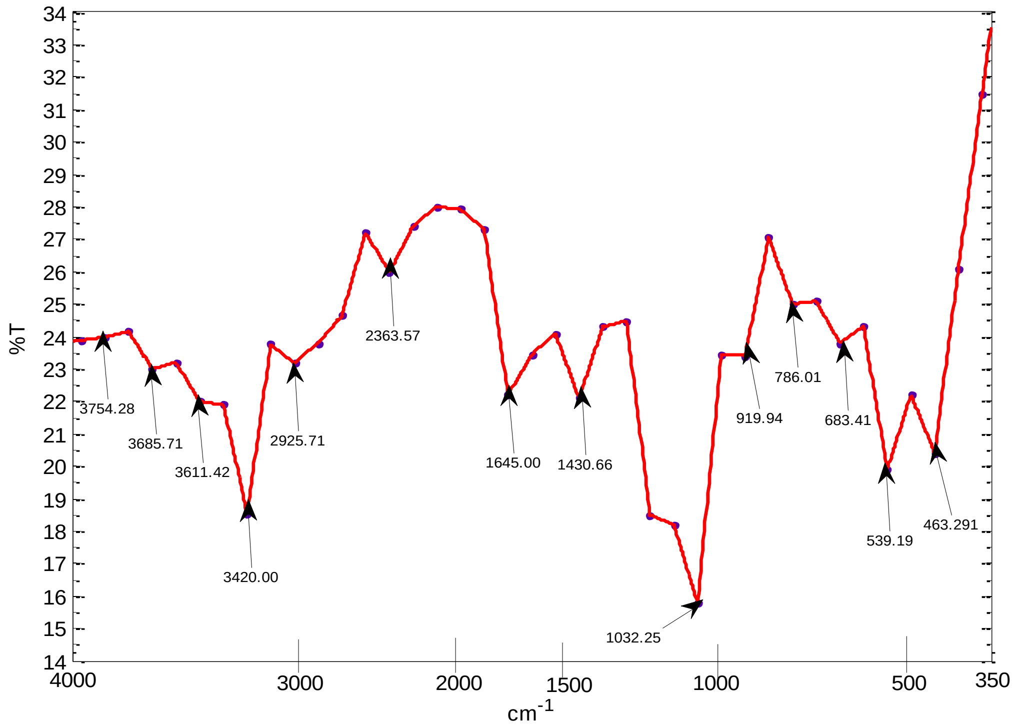

Fig.4. FTIR spectrum of *Sphagnum cymbifolium* (moss) before adsorption

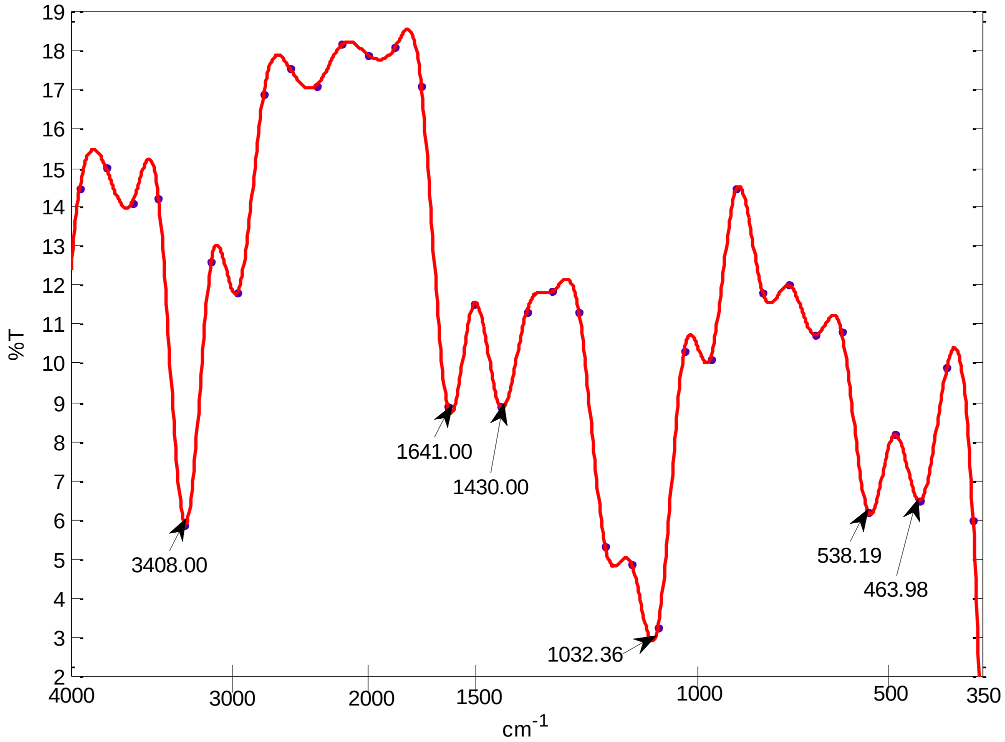

Fig.5. FTIR spectrum of *Sphagnum cymbifolium* (moss) with Methylene blue dye after adsorption

The FTIR spectrum of *Sphagnum cymbifolium* (moss) shown in figure 4 revealed the presence of five major functional groups. The functional groups include O-H or N-H at 3420nm, C-H at 2925.71nm, C≡N, C≡C at 2363.57nm, C=O, C=C at 1645nm and benzene at less than 1000nm. Similar findings were reported by other researchers for the characterization of the biomass pad*ina parvonica* [6].

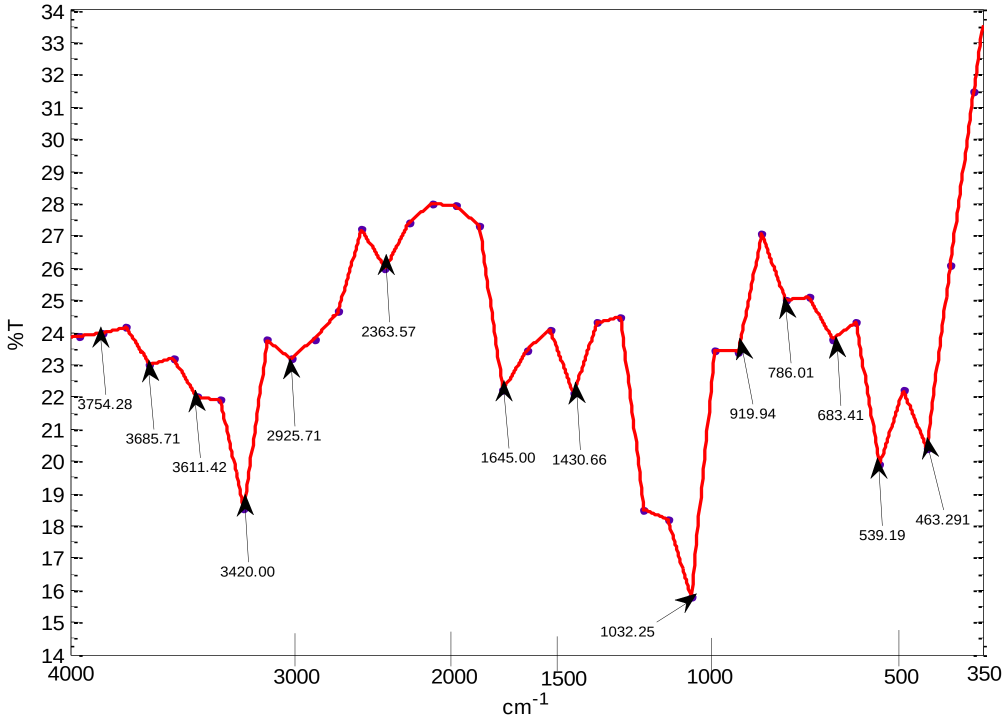

Fig.6. FTIR spectrum of *Sphagnum cymbifolium* (moss) before adsorption

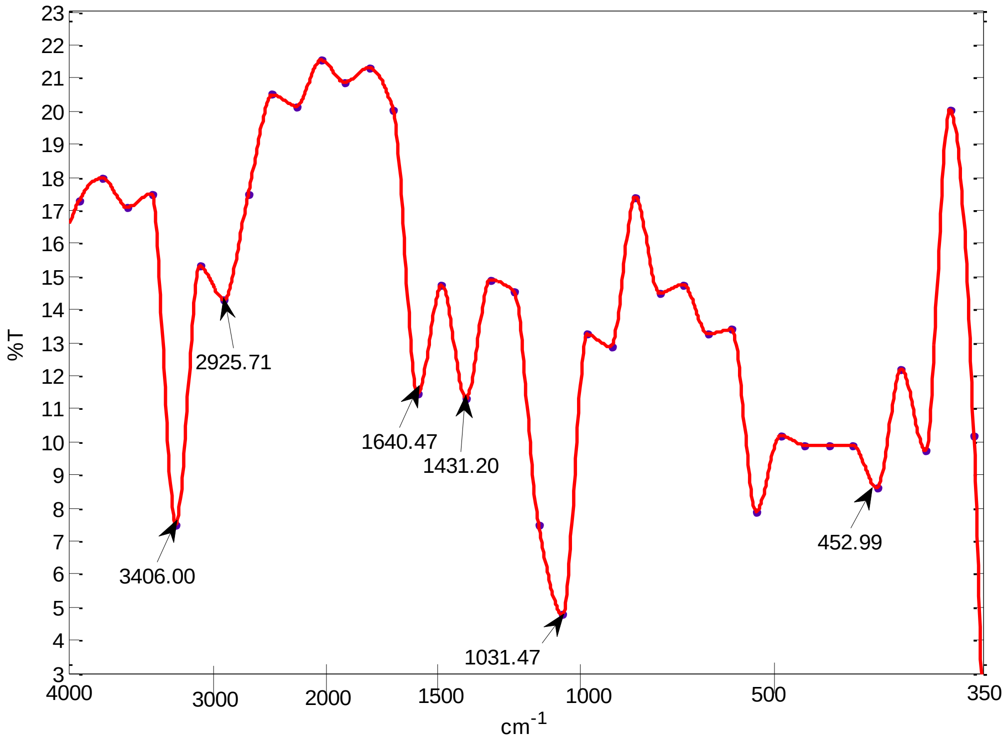

Fig.7. FTIR spectrum of *Sphagnum cymbifolium* (moss) with Bismarck brown Y dye after adsorption

The FTIR spectrum of *Sphagnum cymbifolium* (moss) after adsorption was used to ascertain the functional groups which were responsible for the adsorption. Figures 6, 8 and 10 show the FTIR spectra of *Sphagnum cymbifolium* (moss) with Methylene blue dye, Bismarck brown Y dye, and Indigo dye respectively after adsorption. The spectra showed prominent peaks at 3406nm (-OH, -NH), 1642nm which are characteristic of the –CO functional group which strongly predict the presence of carboxylic acid group in the biomass with the adsorbed dye molecule. After the adsorption, there were some bond displacement of the original peaks indicating the functional groups that were responsible for the adsorption. The displacement occurred at 2925.71nm and 2363.57nm which correspond to these functional groups C-H. C≡N and C≡C.

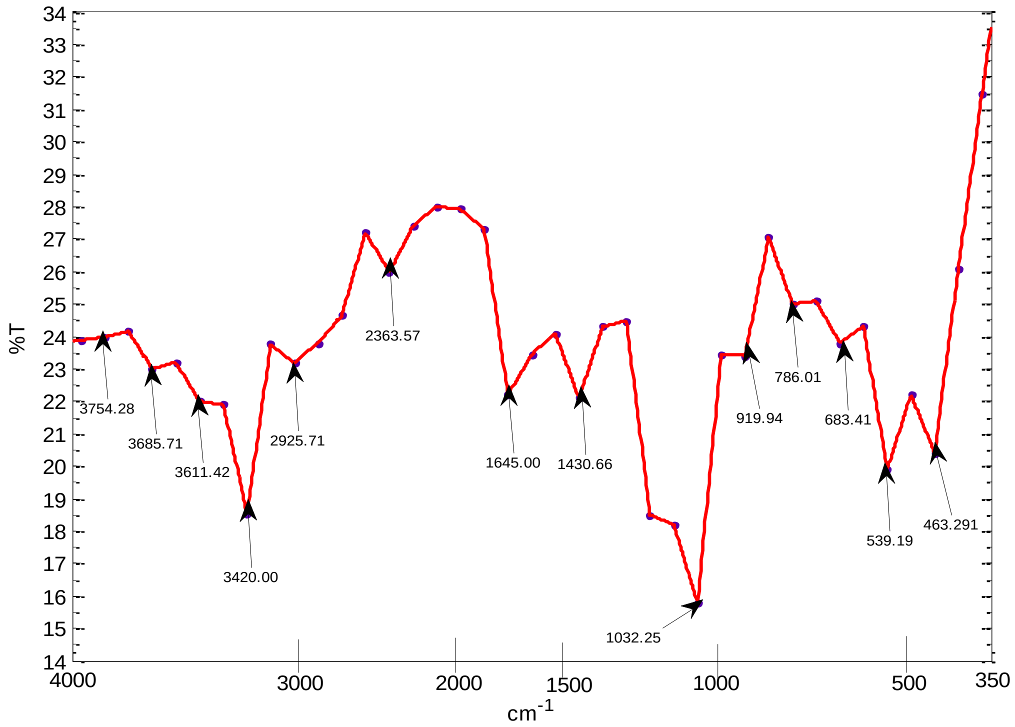

Fig.8. FTIR spectrum of *Sphagnum cymbifolium* (moss) before adsorption

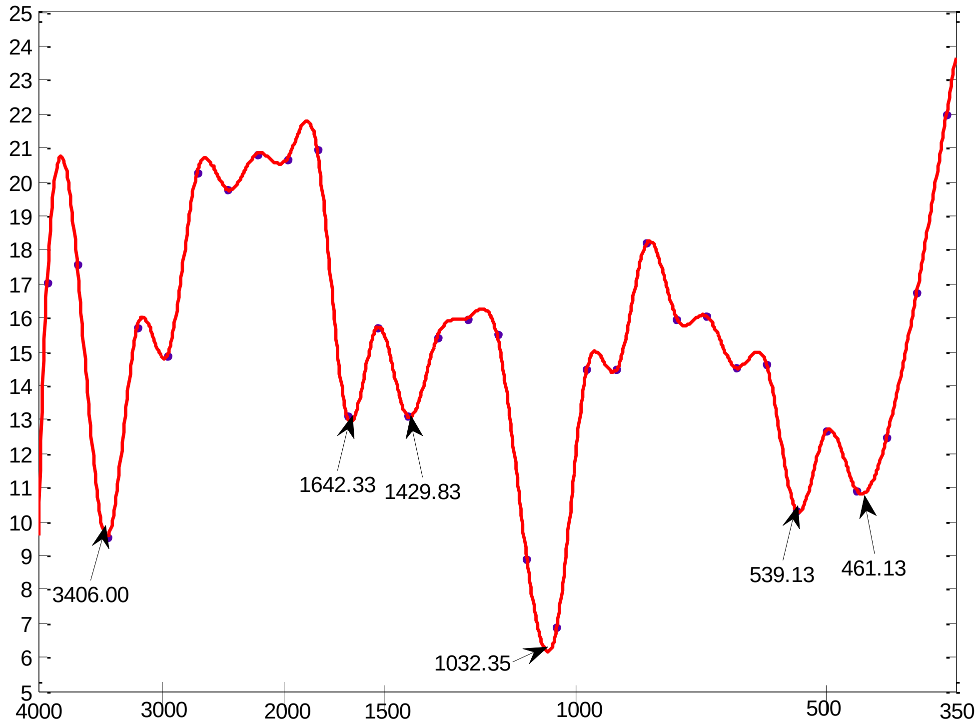

Fig.9. FTIR spectrum of *Sphagnum cymbifolium* (moss) with Indigo dye after adsorption

Furthermore, although the intensity of the peaks greatly decreased after adsorption, the functional groups in the biomass did not disappear totally during the biomass characterization after adsorption. This indicates that the interaction of the dye molecule with *Sphagnum cymbifolium* (moss) was merely a physical process [7].

Table 1. Effect of Flow Rate on the Fixed Bed Adsorption of Methylene Blue Dye, Bismarck Brown Y and Indigo Dye on to *Sphagnum Cymbifolium*

Flow rate (m^3/s)	**20**	**30**	**40**
Methylene blue q_e (mg/g)	18.80 ± 0.02	22.70 ± 0.08	25.40 ± 0.07
Bismarck brown Y q_e (mg/g)	12.34 ± 0.01	18.31 ± 0.01	20.62 ± 0.01
Indigo dye q_e (mg/g)	6.48 ± 0.01	14.28 ± 0.02	17.71 ± 0.02

Footnote

Three experiments were conducted in each case, and the values in the table show the mean with their standard deviations. As could be seen from table 1, increasing in the flow rate caused a corresponding increase in the q_e values for the biomass within the range of experimental consideration. This could be attributed to the increase in the force of interaction between the dye solution and the biomass surface area. Methylene blue dye was the most adsorbed while indigo dye was the least adsorbed. This agrees with experimental findings of other researchers [8].

Table 2. Effect of Bed Height on the Fixed Bed Adsorption of Methylene Blue Dye, Bismarck Brown Y Dye And Indigo Dye on to *Sphagnum Cymbifolium*

Bed height (10^{-2} m)	**4**	**5**	**6**
Methylene blue dye q_e (mg/g)	6.31 ± 0.02	24.66 ± 0.02	27.73 ± 0.01
Bismarck brown y dye q_e (mg/g)	16.40 ± 0.01	23.70 ± 0.02	25.60 ± 0.02
Indigo dye q_e (mg/g)	12.57 ± 0.01	16.51 ± 0.01	17.71 ± 0.01

Footnote

Three experiments were conducted in each case, and the values in the table show the mean with their standard deviations. Table 2 shows the effect of bed height on the quantity of each adsorbed on to the adsorbent. The q_e values for the biomass increase with increase in bed height within the range of experimental considerations. The result indicates that the longer the bed height, the higher the values. This could be due to the longer time of interactions between the biomass and the dye solutions. Methylene blue dye showed a better increment in q_e values while indigo dye was the least adsorbed. Reports from other researchers show a similar trend [9].

4. Conclusion

The results obtained show vividly that fixed bed adsorption is dependent on both flow rate and bed height. Increase in flow rate and bed height gave rise to a corresponding increase in the value of q_e of the bio-sorbent. A similar situation has been reported by other researchers. We can also conclude that the used bio sorbent can be used to treat waste water effluent containing Methylene blue dye, Bismarck brown Y dye, and Indigo dye. From the results obtained, methylene blue dye was the most adsorbed while indigo dye was the least adsorbed within the same experimental considerations.

Figures 2 and 3 revealed pores in the morphology of the biomass which is indicative of where the dye molecules could be trapped in the course of the adsorption.

Additionally, figures 5, 7, and 9 compared the adsorption peaks of dye after adsorption with the peaks before adsorption. There were depressions at some wavelengths which showed the functional groups which were responsible for the adsorption.

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Declarations

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Competing Interests Statement

The authors declare no competing financial, professional and personal interests.

Consent for publication

Authors declare that they consented for the publication of this research work.

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