IOSR Journal of Applied Chemistry (IOSR-JAC)
e-ISSN: 2278-5736.Volume 13, Issue 8 Ser. I (August. 2020), PP 41-54
www.iosrjournals.org

Comparative Study Of The Impact Of Flow Rate And Bed Height On The Fixed Bed Adsorption Of Methylene Blue, Bismarck Brown Y, And Indigo Dyes On To *Cedruslibani* (Elizabeth Leaf) And *Sphagnum Cymbifolium*(Moss) Biomass.

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Abstract
The adsorption behavior of methylene blue dye, Bismarck brown Y dye and Indigo dye on to Sphagnumcymbifolium (moss) and Cedruslibani (Elizabeth leaf) were investigated as a function of flow rate, and bed height through the fixed bed process.
OBJECTIVES*: 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. Also, it is aimed at achieving a comparative elucidation of the dependency or otherwise of flow rate and bed height on adsorption through the fixed bed technique using two different biomass.*
METHODS*: The two biomass were characterized by scanning electron microscope (SEM), in order to examine the morphology of the two biomass. The screened biomass samples were characterized at 1000x magnification and 500x magnification respectively for their surface morphologies. This was done using a scanning electron microscope (FEI- Inspect/ OXFORD INSTRUMENT- X- MAX) which was equipped with an energy dispersive x-ray (EDAX) spectrophotometer employed for the elemental composition analysis. It was equally characterized with Fourier Transformed Infrared spectroscopy (FTIR) before and after adsorption to ascertain the functional groups responsible for the adsorption. This was done using a Fourier Transformed Infrared (FTIR) spectrophotometer (Perkin- Elmer, England) in the wavelength range of 350-4000nm.*
RESULT: *Results for biomass morphology obtained through the scanning electron microscope (SEM) revealed the presence of tiny pores. These pores were more pronounced in the Sphagnum cymbifolium(moss) biomass than in the Cedruslibani (Elizabeth leaf) biomass. These pores represent sites where dye molecules could be trapped in the course of the adsorption. The results from the Fourier Transformed Infrared spectroscopy (FTIR) for both biomass after adsorption show that C-H, C≡H and C≡C functional groups were responsible for the adsorption.*
With the Sphagnum cymbifolium (moss) biomass, for methylene blue dye at the flow rate of 20m^3/s, the amount of dye adsorbed was 18.80mg/g, 22.70mg/g at 30m^3/s and 25.40mg/g at 40m^3/s. For Bismarck brown Y dye, at the same range of flow rate, the amount of dye adsorbed ranged from 12.34mg/g-20.62mg/g. For Indigo dye, the values obtained ranged from 6.48mg/g – 17.71mg/g.
In addition, at the bed height range of 4.0- 6.0 x 10^{-2}(m) the amount of dye adsorbed ranged from 6.31mg/g – 27.73mg/g for methylene blue dye. Within the same range of bed height, the amount of dye adsorbed ranged from 16.40mg/g – 25.60mg/g for Bismarck brown Y dye and 12.57mg/g – 17.71mg/g for Indigo dye. On the other hand, using Cedruslibani (Elizabeth leaf) biomass, for methylene blue dye, at the flow rate of 20m^3/s, the amount of dye adsorbed is 8.40mg/g, 11.30mg/g at 30m^3/s and 13.64mg/g at 40m^3/s. For Bismarck brown Y dye, at the same flow rate, the amount of dye adsorbed ranged from 4.71mg/g – 9.78mg/g. The values obtained with Indigo dye ranged from 2.80mg/g – 8.00mg/g. In addition, at the bed height of 4.0 – 6.0 x 10^{-2} (m), the amount of dye adsorbed ranged from 5.15mg/g – 24.62mg/g for Bismarck brown Y dye and 5.66mg/g – 14.86mg/g for Indigo dye.
CONCLUSION*: From the results obtained, it is clearly seen that methylene blue dye was the most adsorbed, while Indigo dye was the least adsorbed within the same flow rate and bed height ranges. In addition, the three classes of dyes used in these investigations which represent cationic, anionic and neutral dye molecules can adsorb on to Sphagnum cymbifolium (moss) andCedruslibani (Elizabeth leaf) biomass at various degrees. The amount of dye adsorbed is dependent on the flow rate and bed height within the range of experimental consideration. Furthermore, Sphagnum cymbifolium (moss) adsorbed better than Cedruslibani (Elizabeth leaf). In each of the analyses, three different experiments were performed and the mean values reported with their standard deviations.*
Keywords*: Bio-sorption, Sphagnumcymbifolium. Cedruslibani, SEM, adsorbent, fixed bed.*

Date of Submission: 11-08-2020 Date of Acceptance: 27-08-2020

I. Introduction

Bio-sorption can be defined as the abstraction of organic and in organic species, including metals, dyes and odor causing substances using live or dead biomass or their derivatives through the batch or fixed bed technique. The batch process of adsorption occurs due to agitation between the biomass and the dye solution. Such agitation is normally provided by a shaker or magnetic stirrer. On the other hand, the fixed bed adsorption process are ubiquitous throughout the chemical process industry[1]. Separation in a fixed bed is virtually in all practical cases, an unsteady state rate controlled process. Adsorption only occurs in a particular region of the bed known as the mass transfer zone (MT2) which moves the bed. This is practically achieved by allowing the dye solution to pass through the column containing the biomass from down of the column to the top by the use of a peristaltic pump. The removal of dyes from solutions has been attempted in the past using such techniques such as advanced oxidation process (AOP), Nano filtration (NF) and reverse osmotic membrane[(2,3)].

However, in recent times, the use of bio-sorption techniques for the removal of dye contaminants from waste water solutions have been found to be superior to other techniques based on the simplicity of design and operation[4]. Most commercial systems use activated carbon ad 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. This has resulted in attempts by various workers to prepare low cost alternative adsorbents[5].

Adsorption techniques are effective and attractive for the removal of non- biodegradable pollutants (including dyes) from waste waters[6]. Many low cost adsorbents and waste materials from industries and agriculture have been proposed by several researchers[7]. These materials do not require any expensive additional pretreatment step and could be used as adsorbents for the removal of dyes from waste water solutions. In the light of the above, an investigation was carried out on the kinetics and thermodynamic studies of adsorption of malachite green onto unmodified and EDTA modified groundnut husk, using the batch technique[8].

This work is carried out with the view of expanding the field of application of natural biomass for the treatment of dye waste waters and also to do a comparative study on the impact of flow rate and bed height on the fixed bed adsorption of methylene blue, Bismarck brown Y and Indigo dyes on to *Sphagnum cymbifolium* (moss) and*Cedruslibani* (Elizabeth leaf) biomass since such an in depth comparisms has not been done on the two biomass, the result obtained from the work will add to the expansion of knowledge in this area.

II. Material And Methods

The methylene blue dye, Bismarck brown Y dye, and Indigo dye used in these investigations were obtained from qualikem laboratory, Owerri, Nigeria. Other materials obtained here include analytical grade sodium hydroxide pellets, concentrated hydrochloric acid, distilled water etc. The *Cedruslibani* (Elizabeth leaf) and *Sphagnum cymbifolium* (moss) biomass used in this work were obtained from Ikorodu area of Lagos, Nigeria which is located within the following coordinates 6.6194°N and 3.5105°E. The samples were identified at the department of crop science at the Federal University of Technology, Owerri, Nigeria with the voucher specimen number of FUT/CR/002/15 and FUT/CR/003/15 respectively. The two biomass were washed severally with distilled water to remove any dirt from it. The washed biomass were air dried for ten days until constant weights were obtained. The biomass were 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 these determinationsare the standard methods which have been used by other researchers[(8,10)].

CHARACTERIZATION OF THE BIO-SORBENT

The surface structure and morphology of the *Cedruslibani* (Elizabeth leaf) and the *Sphagnumcymbifolium* (moss) biomass were characterized at 1000x magnification, 500x magnification and 250x magnification respectively for their surface morphologies. This was done using scanning electron microscopy (SEM) (FEI- Inspect oxford instrument x-max), which was equipped with an energy dispersive x-ray (EDAX) spectrometer employed for elemental composition analysis.

The biomass samples were further characterized for their fundamental functional groups before and after adsorption experiment using a Fourier Transformed Infrared (FTIR) spectrophotometer (Perkin Elmer, England) in the wave length range of 350-4000nm using KBr powder and fluka library for data interpretations.

THE FIXED BED SETUP

The fixed bed was set up by packing wire guaze, glass wool, glass beads, glass wool in that order in a graduated condenser. Then a dye solution of a known concentration and pH pressurized from down to top where a known amount of bio sorbent is placed with a peristaltic pump (CHEM- TECH model X030- XBAAA 365, China). Subsequently, a sample was collected for U.V analysis in a U.V spectrophotometer (CAMSPEC M 106 model, England) by monitoring the absorbance already determined for methylene blue dye (600nm), Bismarck brown Y dye (320nm) and Indigo dye (350nm) respectively. The variables investigated here include the effect of bed height and flow rate.

EFFECT OF FLOW RATE ON ADSORPTION

Experiments were carried out on the two different biomass at different flow rate $20m^3/s$, $30m^3/s$ and $40m^3/s$ while keeping constant a bed height of $1.0 \times 10^{-2}m$, 40mg biomass dose, 90mg/L dye solution and a pH of 4 for methylene blue dye and a 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 for absorbance measurements subsequently, the absorbance values were converted to concentration by the use of Beer Lambert's law. Similar experiments were carried out on the two biomass in triplicates and the mean values and standard deviations reported.

EFFECT OF BED HEIGHT ON ADSORPTION

Experiments were carried out on the two different biomass at different bed heights of $4.0 \times 10^{-2}m$, $5 \times 10^{-2}m$ and $6 \times 10^{-2}m$ while keeping constant a flow rate of $10m^3/s$, 90mg/L dye solution, pH of 4 for methylene blue dye, and a pH of 2 for both Bismarck brown Y and Indigo dye earlier determined as their best pH of maximum adsorption. The dye solution was subjected to pass through the column already prepared using a peristaltic pump. The samples collected were subjected to U.V analysis for absorbance measurements. Subsequently, the absorbance values were converted to concentration by the use of Beer Lambert's law. Similar experiments were carried out on the two biomass in triplicates and the mean values and standard deviation reported.

NOTE: The amount of dye adsorbed per gram biomass (q_e) was calculated using the expression below.

$$q_e = \frac{\mathrm{V}(c_o - c_e)}{\mathrm{M}}$$

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

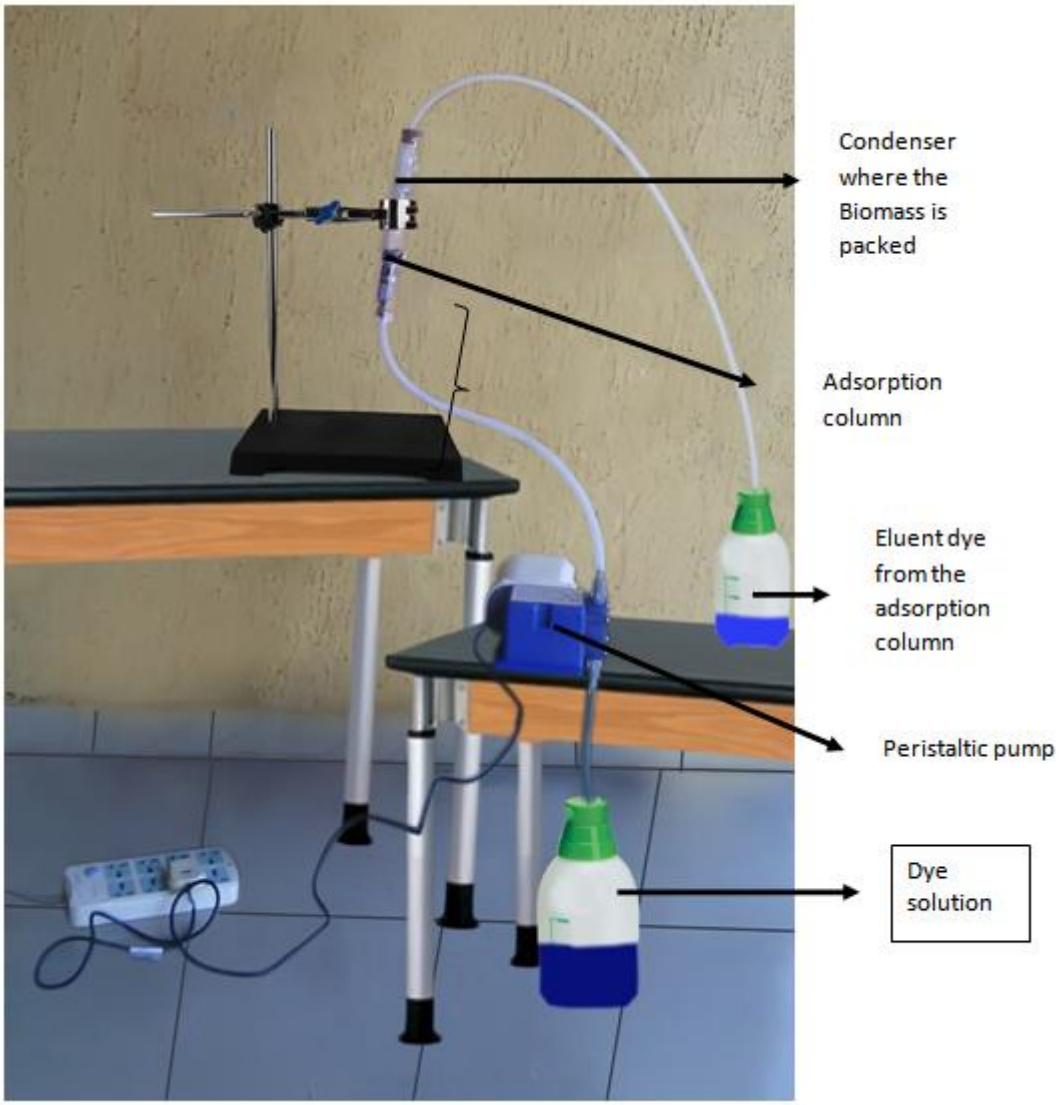

Fig 1: Fixed bed technique apparatus

III. Result And Discussion

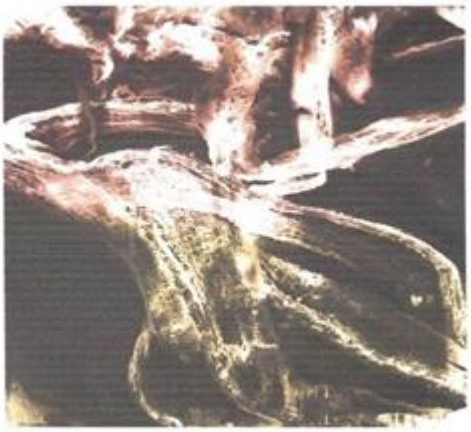

Fig. 2: SEM morphology of *Cedruslibani* (Elizabeth leaf X500)

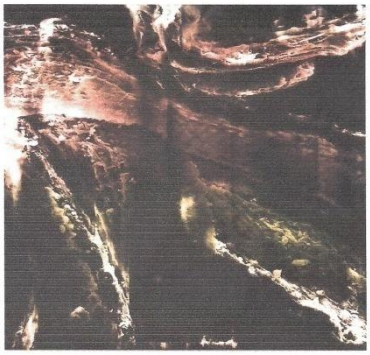

Fig. 3: SEM morphology of *Cedruslibani* (Elizabeth leaf X1000)

The SEM micrographs of *Cedruslibani* (Elizabeth leaf) revealed the presence of unevenly dispersed cavities on the surface of the biomass. The cavities provide sites where the molecules of the dyes could be trapped in the course of adsorption. The SEM micrographs of (X500) and (X1000) magnifications of *Cedruslibani* (Elizabeth leaf) are shown in figure 2 and 3 respectively.

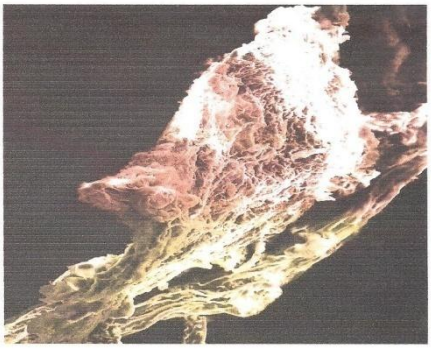

Fig. 4: SEM morphology of *Sphagnum cymbifolium* (moss X500)

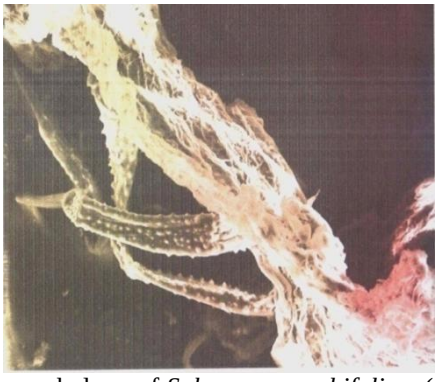

Fig. 5: SEM morphology of *Sphagnum cymbifolium* (moss X1000)

The scanning electron microscopy (SEM) micrographs of *Sphagnum cymbifolium* (moss) at (X500) and (X1000) magnifications shown in figure 4 and 5 also reveal the presence of unevenly dispersed granules or cavities on the surface of the biomass. These cavities appear larger than the ones revealed in *Cedruslibani* (Elizabeth leaf) biomass. This indicates that adsorption may be better in *Sphagnum cymbifolium* (moss) compared with *Cedruslibani* (Elizabeth leaf) biomass[11].

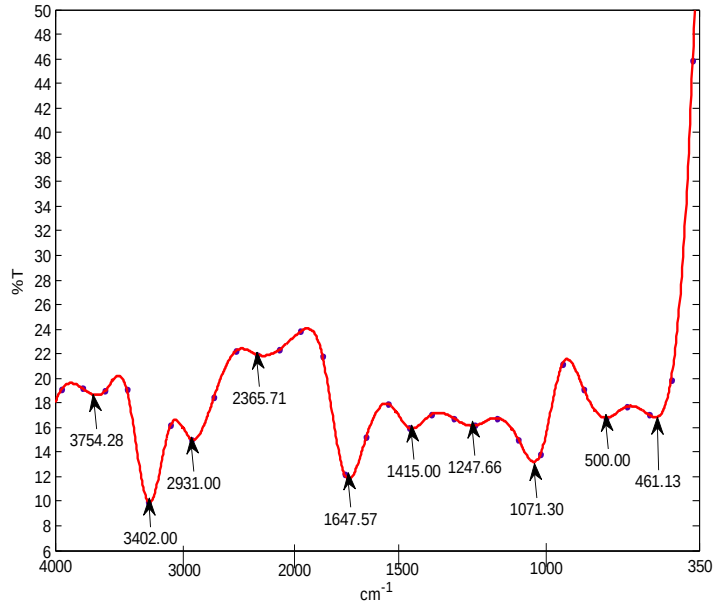

Fig. 6: FTIR spectrum of *Cedruslibani* (Elizabeth leaf) before adsorption.

The FTIR spectrum of *Cedruslibani* (Elizabeth leaf) shown in figure 6 reveals 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. As could be seen, the *Cedruslibani* (Elizabeth leaf) spectra (scanned between 350- 4000nm) revealed broad peaks around 3420nm, which lies well between 3200-3600nm. This corresponds to the presence of O-H functional groups on the surface of the biomass.[10] Other prominent peaks were observed around 1645nm and 1430nm and are due to carbonyl (C=O) stretching from aldehydes or ketones.[10] The peaks observed around 1031nm were attributed to the C=O stretch due to primary alcohol. The combination of these functional groups arising from OH and CO suggest the occurrence of a carboxylic functional group.

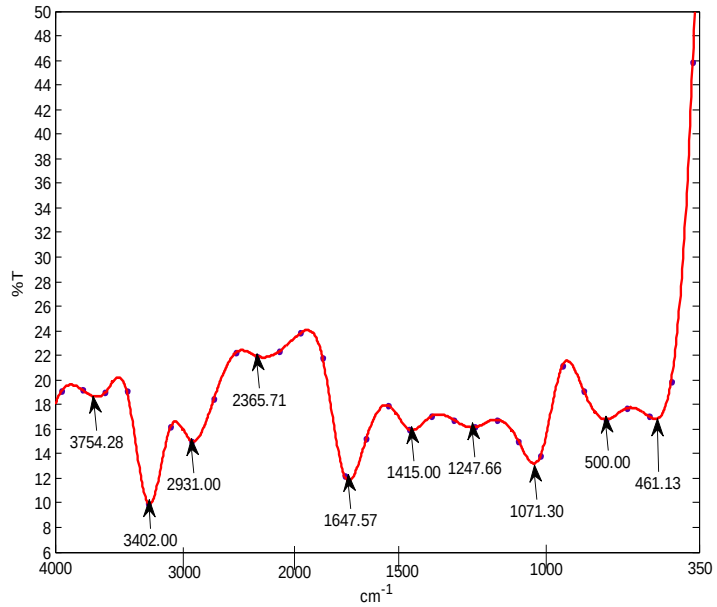

Fig. 7: FTIR Spectrum of *Cedruslibani* (Elizabeth leaf) before adsorption

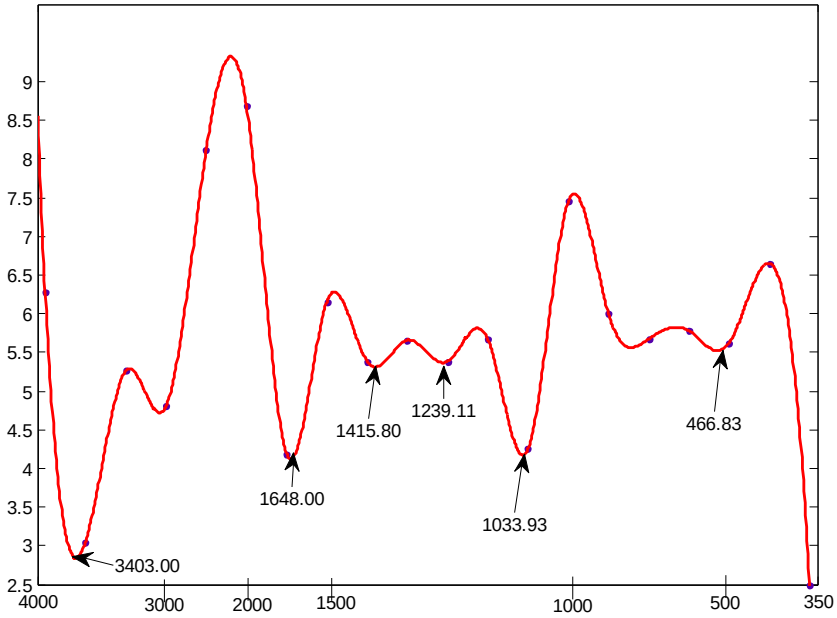

Fig. 8: FTIR Spectrum of*Cedruslibani* (Elizabeth leaf) with methylene blue dye after adsorption.

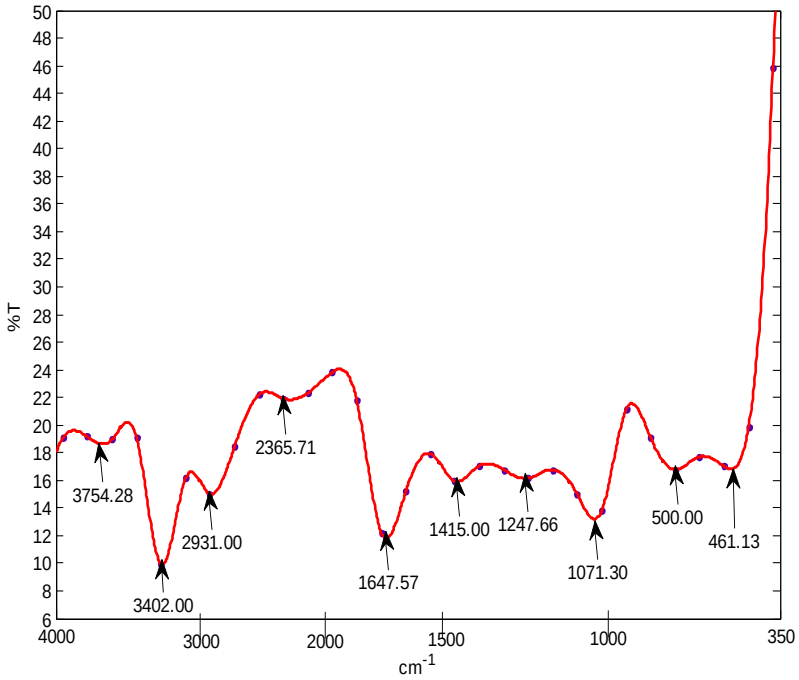

Fig. 9: FTIR Spectrum of *Cedruslibani* (Elizabeth leaf) before adsorption

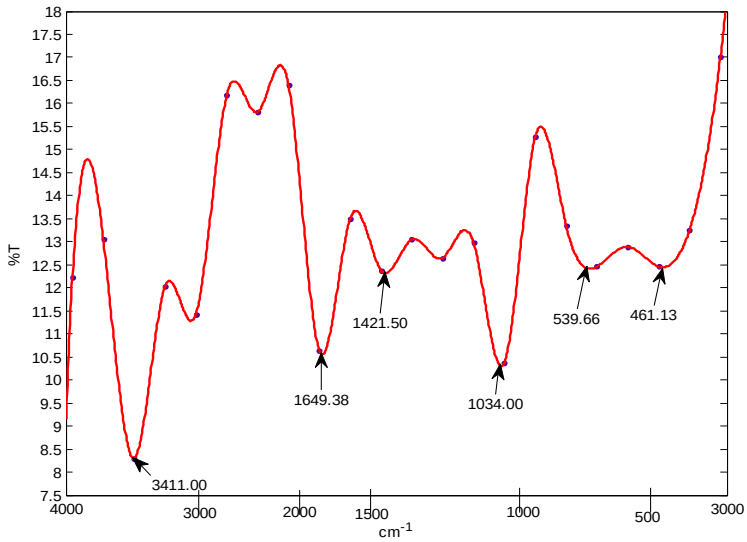

Fig. 10: FTIR Spectrum of *Cedruslibani* (Elizabeth leaf) with Bismarck brown Y dye after adsorption.

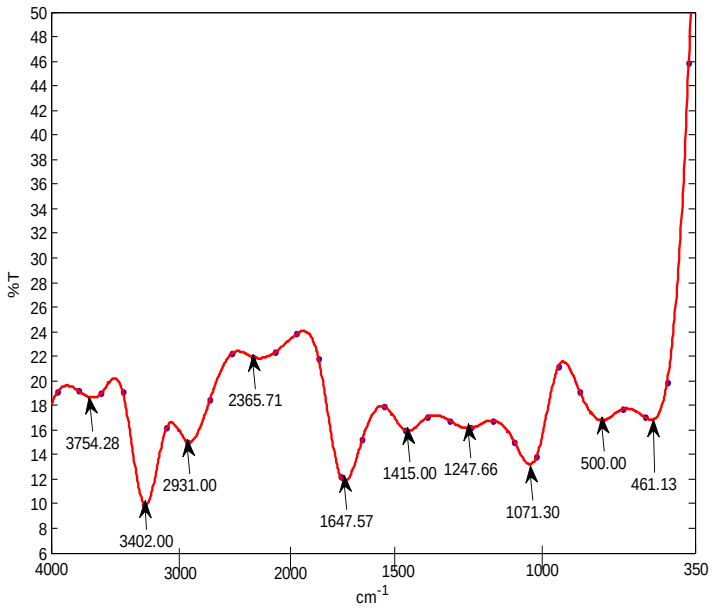

Fig. 11: FTIR Spectrum of *Cedruslibani* (Elizabeth leaf) before adsorption

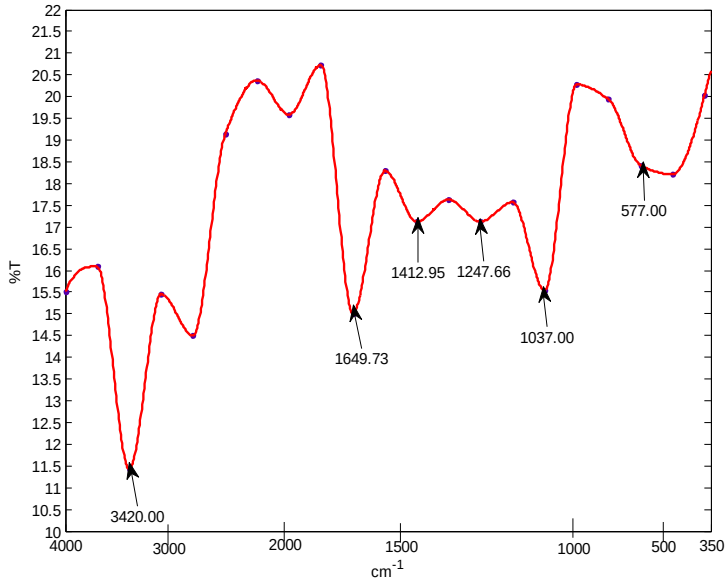

Fig. 12: FTIR Spectrum of *Cedruslibani* (Elizabeth leaf) with Indigo dye after adsorption.

After the adsorption process as shown in fig 8, 10 and 12 there were depressions of the original peaks as shown in fig. 7, 9 and 11 respectively. From the depressions observed, we can determine the functional groups that were actually responsible for the adsorption reaction. The displacements occurred at 2931.00nm and 3265.71nm, indicating that the following functional groups C-H, C≡N and C≡C were responsible for the adsorption process. Furthermore, the functional groups did not disappear totally after the adsorption process. This indicate that the interaction of the dye molecules with *Cedruslibani* (Elizabeth leaf) was indeed a physical process.

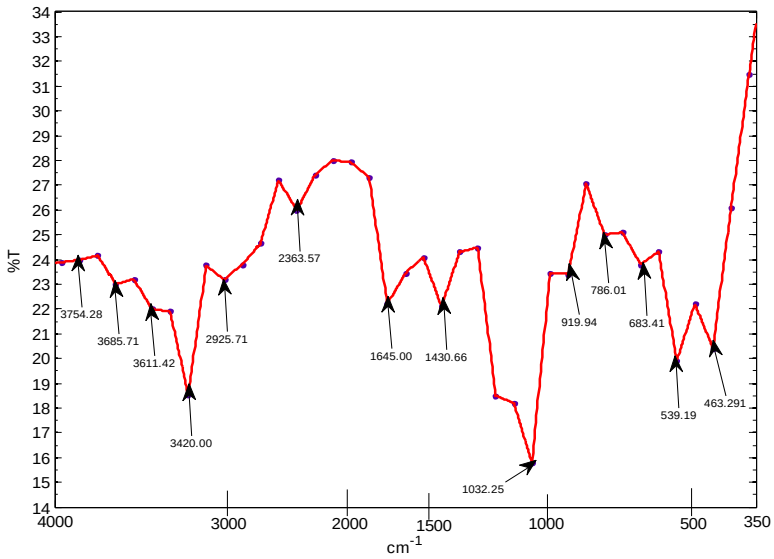

Fig. 13: FTIR Spectrum of *Sphagnum cymbifolium* (moss) before adsorption

The FTIR spectrum of *Sphagnum cymbifolium* (moss) as shown in fig.13 also 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 *Padina parvonica*[6].

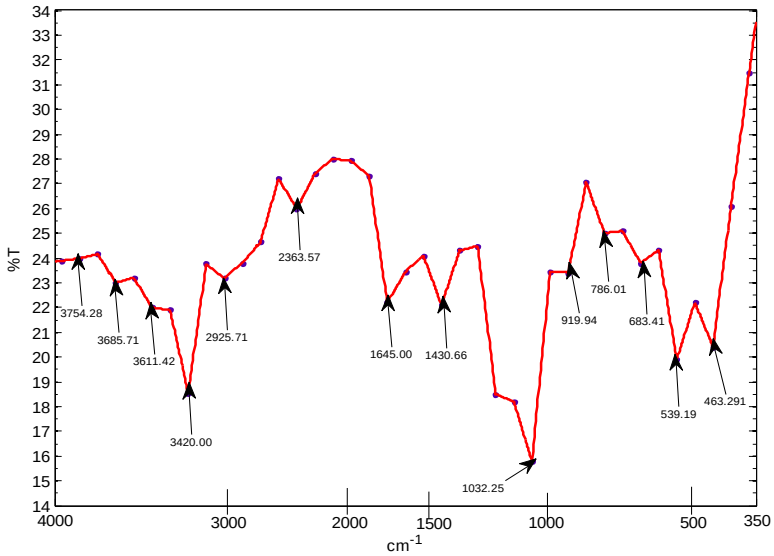

Fig. 14: FTIR Spectrum of *Sphagnum cymbifolium* (moss) before adsorption

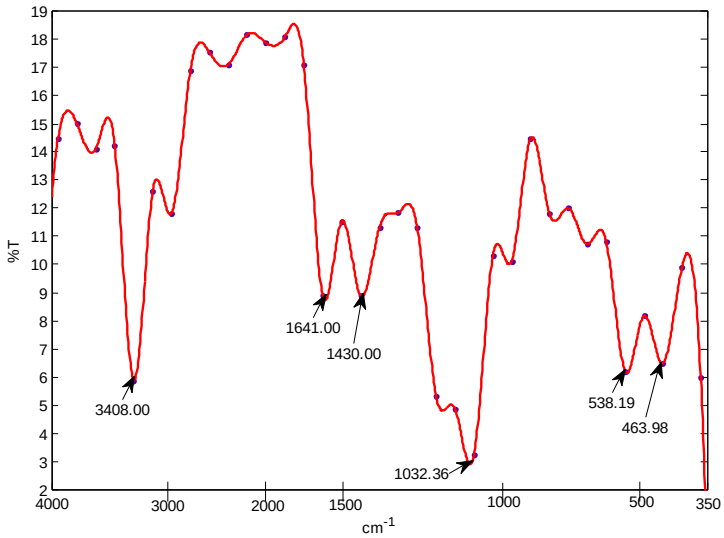

Fig. 15: FTIR Spectrum of *Sphagnum cymbifolium* (moss) with Methylene blue dye after adsorption.

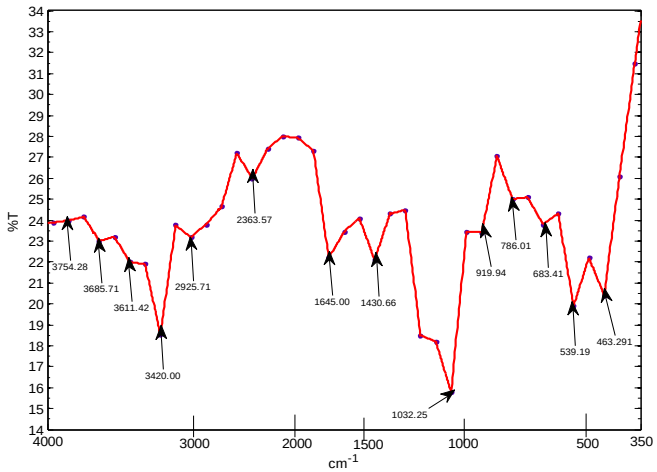

Fig. 16: FTIR Spectrum of *Sphagnum cymbifolium* (moss) before adsorption.

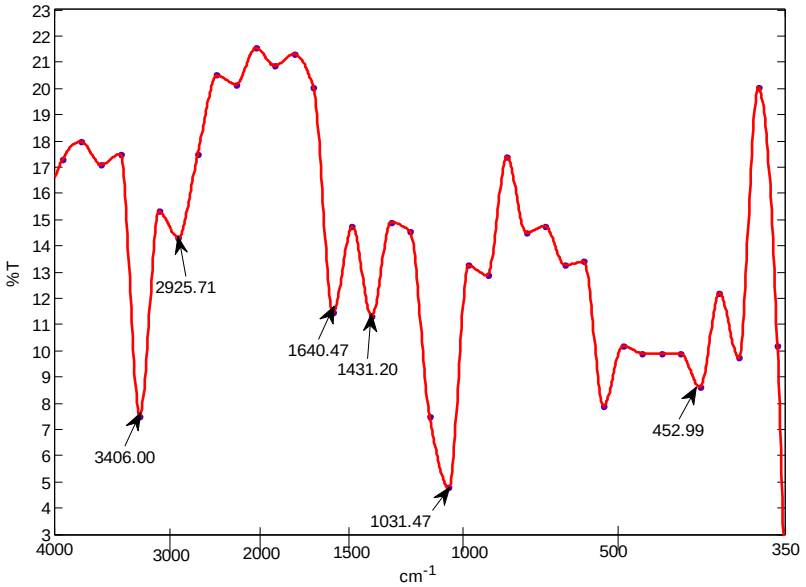

Fig. 17: FTIR Spectrum of *Sphagnum cymbifolium* (moss) with Bismarck brown Y dye after adsorption.

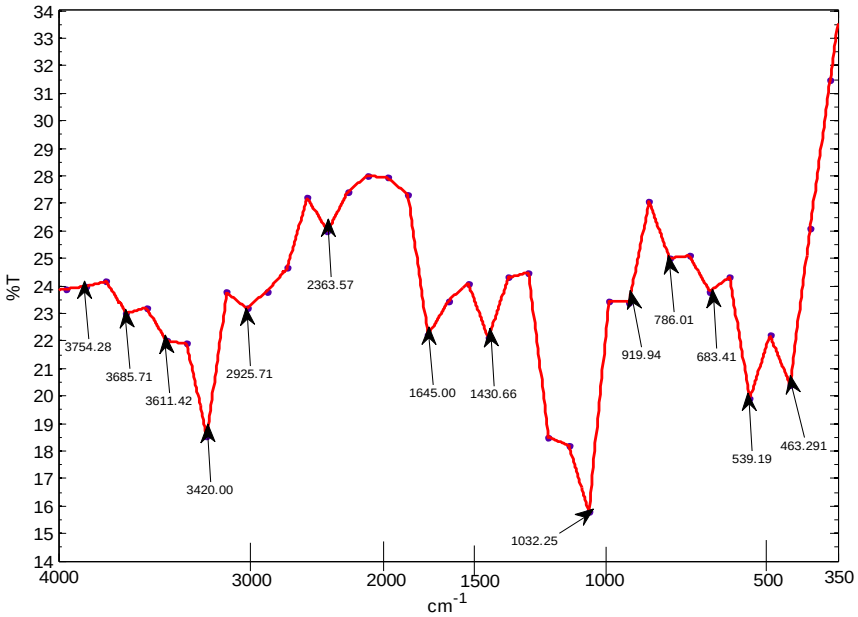

Fig. 18: FTIR Spectrum of *Sphagnum cymbifolium* (moss) before adsorption.

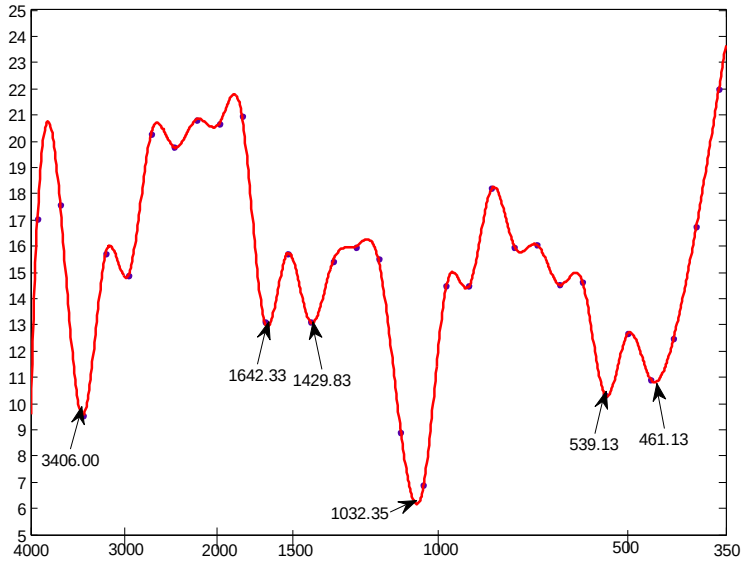

Fig. 19: FTIR Spectrum of *Sphagnum cymbifolium* (moss) with indigo dye after adsorption.

The FTIR Spectrum of *Sphagnum cymbifolium* (moss) after adsorption was used to ascertain the functional groups which were responsible for the adsorption. Figure 15, 17 and 19 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 group 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. 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. As was seen with *Cedruslibani* (Elizabeth leaf) biomass, it implies that the interaction of the dye molecule with *Sphagnum cymbifolium* (moss) was merely a physical process.

Table 1: Effect of flow rate on the fixed bed adsorption of methylene blue dye. Bismarck brown Y dye and Indigo dye on to *Cedruslibani*

Flow rate (m^3/s)	20	30	40
Methylene blue dye q_e (mg/g)	8.40 ± 0.01	11.30 ± 0.01	13.64 ± 0.02
Bismarck brown Y dye q_e (mg/g)	4.71 ± 0.01	8.80 ± 0.01	9.78 ± 0.01
Indigo dye q_e (mg/g)	2.80 ± 0.02	6.46 ± 0.02	8.00 ± 0.01

Footnote:
Three experiments were carried out in each case, and the values shown in the above table represent the mean values and their standard deviations.
As could be seen from table 1, increase in the flow rate caused a corresponding increase in the q_e values for the biomass within the range of experimental consideration. A similar effect was reported by other researchers[11].
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.

Table 2: Effect of bed height on the fixed bed adsorption of methylene blue dye. Bismarck brown Y dye and Indigo dye on to *Cedruslibani*

Bed height (10^{-2}m)	4	5	6
Methylene blue dye q_e (mg/g)	5.15 ± 0.02	20.35 ± 0.01	24.62 ± 0.01
Bismarck brown Y dye q_e (mg/g)	8.20 ± 0.02	11.00 ± 0.01	15.00 ± 0.01
Indigo dye q_e (mg/g)	5.66 ± 0.02	12.91 ± 0.02	14.86 ± 0.01

Footnote:
Three experiments were carried out in each case, and values shown in the above table represent the mean values with their standard deviations.

Table 2 shows the effect of bed height on to the quantity of each dye adsorbed on to the adsorbent. The q_e values for the biomass increased with increase in bed height within the range of experimental considerations. The result indicates that longer the bed height, the higher the q_e values. A similar situation has been reported in a similar investigations[11]. This could be due to the longer time of interactions between the biomass and the dye solutions. Methylene blue dye was adsorbed more while indigo dye was the least in these considerations.

Table 3: Effect of flow rate on the fixed bed adsorption of methylene blue dye. Bismarck brown Y dye 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[12].

Table 4: 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[13].

IV. Conclusion

The findings of this research vividly show that the two variables- flow rate and bed height can affect the adsorption properties of methylene blue dye, Bismarck brown y and indigo dye on to *Cedruslibani* and *Sphagnum cymbifolium*. Increase in bed height and flow rate gave rise to a corresponding increase in the q_e value of the adsorbent. It was also discovered that *Sphagnumcymbifolium* adsorbed better than *Cedruslibani* within the same experimental considerations. In both biomass, methylene blue dye was the most adsorbed, while Indigo dye was the least adsorbed.

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Idika, D. I, et. al. "1 Comparative Study Of The Impact Of Flow Rate And Bed Height On The Fixed Bed Adsorption Of Methylene Blue, Bismarck Brown Y, And Indigo Dyes On To Cedruslibani (Elizabeth Leaf) And Sphagnum Cymbifolium(Moss) Biomass." *IOSR Journal of Applied Chemistry (IOSR-JAC)*, 13(8), (2020): pp 41-54.