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Appraisal of bed height and flow rate effect on the removal of dyes on Pine biomass

D. I. Idika[1*], N. A. Ndukwe[2] and C. E. Ogukwe[3]

[1]Department of Basic Sciences, Chemistry Unit, Babcock University, Ilishan, Remo, Ogun state, Nigeria
[2]Department of Chemical Sciences, Mountain Top university, Ogun state, Nigeria
[3]Department of Industrial Chemistry, Federal University of Technology, Owerri, Nigeria
*Corresponding author's E. mail: idikad@babcock.edu.ng

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The impact of flow rate and bed height on the adsorption behavior of methylene blue, Bismarck brown y, and indigo dyes on to *Cedrus libani* was investigated. The biomass was characterized by scanning electron microscopy (SEM) as well as Fourier transformed infrared spectroscopy (FTIR) before and after adsorption to ascertain the functional groups responsible for the adsorption. The amount of dye adsorbed per unit mass of the biomass (q_e) was calculated and was found to be dependent on the variables investigated within the experimental range. It was discovered that increase in bed height and flow rate increased the value of the dye adsorbed on to the biomass. The results obtained show that methylene blue dye adsorbed more onto the biomass, while indigo dye adsorbed at the least level. Findings revealed that the *Cedrus libani* biomass is promising efficiency for the removal of dyes in textile effluents.

Capsule Summary: Impact of flow rate and bed height on the adsorption of *Cedrus libani* was studied and findings revealed that the *C. libani* could be used to treat dye wastewaters containing Methylene Blue, Bismarck Brown Y and Indigo blue dyes.

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INTRODUCTION

Biosorption can be defined as the abstraction of organic and inorganic species including metals, dyes and odor causing substances using live or dead biomass or their derivatives. This process can be achieved either through the batch or fixed bed technique. The batch process of adsorption occurs as a result of agitation between the biomass and the dye solution, such agitation is normally provided by a shaker or magnetic stirrer. On the other hand, fixed bed adsorption process is ubiquitous throughout the chemical process industries (Vannapusa et al., 2008). 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 (MTZ) 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 as advanced oxidation process (AOP), nano-filtration (NF) and reverse osmosis membrane along with biological approaches (Sotelo et al., 2009, Prased and Abdulkah, 2010).

Recently, the use of bio-sorption technique for the removal of dye contaminants from solution has been found to be superior to other techniques based on simplicity of design and operation (Tong et al., 2010). Activated charcoal is widely employed as an adsorbent. However, the use of activated charcoal is restricted due to high cost. This has resulted in attempts by various workers to prepare low-cost alternative adsorbents (Gupta et al., 2003).

Adsorption techniques are effective and attractive for the removal of non-biodegradable pollutant (including dyes) from waste waters (Robinson et al., 2002). Many low-cost adsorbent and waste materials from industries and agriculture have been proposed by several researchers (Amandurai et al., 2002). These materials do not require any expensive additional pre-treatment step and could be used as adsorbent for the removal of dyes from solutions. An investigation was carried out on the kinetics and thermodynamic studies of adsorption of malachite green on to un modified and EDTA modified groundnut husk, using the batch technique (Titilayo et al., 2008).

This work is carried out with the view of expanding the field of application of natural biomass for the treatment of dye wastewaters and also to determine the adsorption capacity of *Cedrus libani* (Elizabeth leaf) on Methylene Blue, Bismarck Brown Y and Indigo dyes. Since such an in-depth comparison has not been done on this biomass, the results obtained from this work will add to the expansion of knowledge in this area.

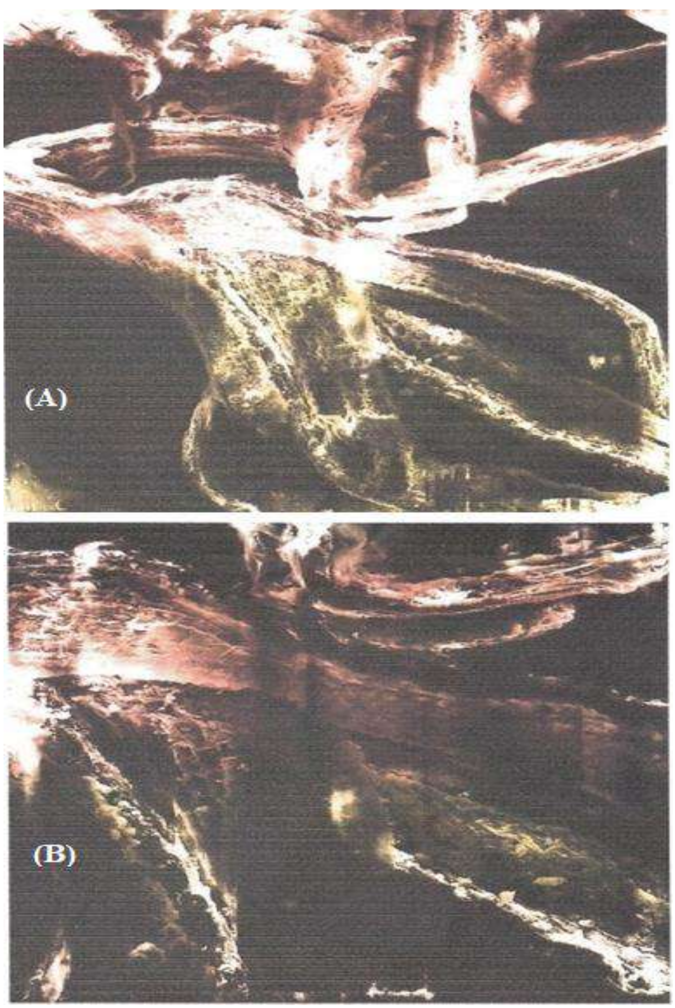

Fig. 1: SEM morphology of *Cedrus libani*, A (X500) and B (X1000)

MATERIAL AND METHODS

Biomass and sample collection

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 *Cedrus libani* (Elizabeth leaf) used in this work was obtained from Ikorodu area of Lagos, Nigeria which is located within the following coordinates 6.6194°N, and 3.5105°E. The sample was 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. The biomass was washed severally with distilled water to remove any dirt from it. The washed biomass was air dried for 10 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 600-800 µm sized sieves and were stored in air tight containers ready for adsorption measurement. The methods and techniques employed in these determinations are the standard methods which have been used by other researchers (Titilayo et al., 2008).

Characterization

The surface structure and morphology of the *Cedrus libani* was characterized at 1000X magnification, 500X magnification and 250X magnification respectively for their surface morphology using a scanning electron microscope (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 sample was further characterized for their fundamental functional groups before and after adsorption experiment using a Fourier Transformed Infrared (FTIR) spectrophotometer (Perkin Elmer, England) in the wavelength range of 350-400nm using KBr powder and fluka library for data interpretation.

Fixed bed set up

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. 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- XB- AAAA365, China) from there, a sample is collected for U.V analysis in a UV spectrophotometer (CAMSPEC M 106 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 (350 nm) respectively. The variables investigated here include the effect of bed height and flow rate.

U.V analysis for absorbance measurements. Subsequently, the absorbance values were converted to concentration by the use of Beer Lamberts law. Similar experiment was carried out in triplicates and the mean values reported.

Effect of bed height

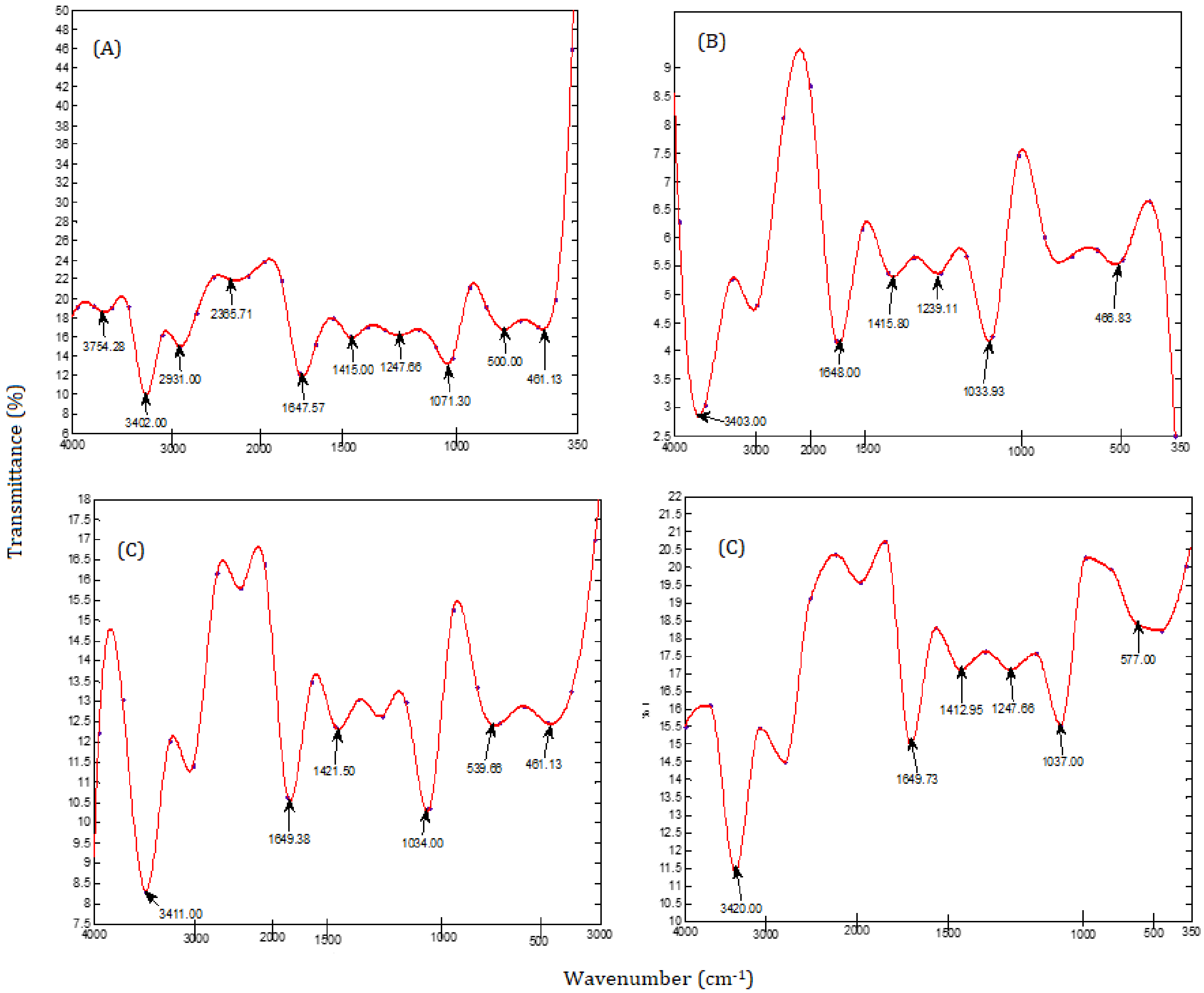

Fig. 2: FTIR spectrum of *Cedrus libani*, (A) before adsorption, (B) methylene blue loaded, (C) Bismarck Brown Y loaded and (D) Indigo dye loaded

Effect of flow rate on adsorption

Experiments were carried out at different flow rate of 20m^3/s, 30m^3/s and 40m^3/s while keeping constant a bed height of 1cm, 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 as earlier determined as the 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

Experiments were carried out at different bed heights of 4, 5 and 6 (cm) 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 a pH of 2 for both Bismarck brown y and indigo dyes as earlier determined as the 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 UV analysis for absorbance measurements. Subsequently, the absorbance values were converted to concentration by the use of Beer–

Table 1: Effect of flow rate on the adsorption of Methylene blue dye, Bismarck brown Y and Indigo dyes on to *Cedrus libani* biomass in a fixed bed

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

Table 2: Effect of bed height on the adsorption of Methylene blue dye, Bismarck brown Y and Indigo dyes on to *Cedrus libani* biomass in a fixed bed

Bed height (cm)	4	5	6
Methylene blue dye q_e (mg/g)	5.15	20.35	24.62
Bismarck brown Y dye q_e (mg/g)	8.20	11.00	15.00
Indigo dye q_e (mg/g)	5.66	12.91	14.86

Lambert law. Similar experiments were carried out in triplicates and the mean values reported. The amount of dye adsorbed per gram biomass (q_e) was calculated using the Eq. 1.

$$q_e = V(C_o - C_e)/M \quad (1)$$

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.

RESULTS AND DISCUSSION

The SEM micrographs of *Cedrus libani* revealed the presence of unevenly dispersed cavities on the surface of the biomass. These cavities provide sites where the molecules of the dyes could be trapped in the course of adsorption. The SEM micrographs of 500X and 1000X magnifications are shown in figures 2 and 3, respectively.

The FTIR spectrum of *Cedrus libani* shown in figure 4 reveals the presence of five major functional groups. The functional groups include O-H or N-H at 3420 nm, C-H at 2925.71 nm, C≡N, C≡C at 2363.57 nm, C=O, C=C at 1645 nm. As could be seen, the *Cedrus libani* spectra (scanned between 350-400 nm) revealed broad peaks around 3420nm which lie well between 3200-3600 nm. This corresponds to the presence of OH functional group on the surface of the biomass (Eman etal; 2013). Other prominent peaks were observed around 1645 nm and 1430 nm and are due to carbonyl (C=O) stretching from aldehydes or ketones as reported by Dotto (2013). The peaks observed around 1031nm was attributed to the C=O stretch due to primary alcohol. The combination of these functional groups arising from the OH and CO suggest the occurrence of carboxylic functional group.

After the adsorption process as shown in figures 6, 8, and 10, there were depressions of the original peaks as shown in figures 5, 7 and 9 respectively. From the depressions observed, we can determine the functional groups that were actually responsible for the adsorption reaction. The displacements occurred at 2931.00 nm 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 adsorption process. This indicates that the interaction of the dye molecules with *Cedrus libani* was indeed a physical process.

As it 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 (Ogukwe et al., 2015). 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 adsorption was minimum on to the *Cedrus libani* biomass.

Table 2 shows the effect of bed height on to the quality 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 investigation (Idika et al., 2019). 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. Hence, finding suggest that *Cedrus libani* biomass have potential to remediate the dyes in the textile wastewater.

CONCLUSIONS

The findings of this research showed that the two variables- flow rate and bed height can significantly affect the adsorption properties of methylene blue dye, Bismarck brown Y and Indigo dyes on to *Cedrus libani* biomass. Increase in flow rate and bed height gave rise to a corresponding increase in the q_e value of the adsorbent. Results revealed that *Cedrus libani* biomass has promising efficiency for the removal of dyes in textile effluents and also, this biomass is extendable for other dyes.

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