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Adsorption of Victoria Blue by Acid-Treated Maize Tassels

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Abstract : The increasing environmental pollution by industrial effluent is now a matter of concern. Biosorption is one of the cheaper ways to fight environmental pollution. In the present study biosorption of victoria blue using sulphuric acid treated maize tassels was studied. The effect of pH, initial concentration, adsorbent dose, contact time and temperature on the adsorption process was investigated. The feasibility of adsorption process was evaluated in the pH range of 2 to 10; concentration range of 50 to 100 ppm, temperature was varied from 25^{o}C to 100^{o}C, variation of contact time from 5 minutes to 30 minutes and dose of the adsorbent from 0.2 g to 1.0 g. The results suggested a high adsorbent dose is required for high adsorption capacity. A pH of 5 was effective with temperature set at 25^{o}C and contact time of 30 minutes. The maximum adsorption capacity obtained was 99.60%. The Freundlich and Langmuir isotherm equations were applied to the experimental data and values of parameters of these isotherm equations were calculated. The data fitted well into Langmuir isotherm. The adsorbent surface functional groups were studied with Fourier Transform Infrared (FTIR) spectroscopy and the results indicated that the adsorbent is a lignocellulosic material. The results suggest that sulphuric acid treated maize tassels can be employed as low-cost biosorbent for the removal of victoria blue dye from aqueous solution.

Keywords : Victoria blue, adsorption, maize tassel, Langmuir Isotherm, Freundlich Isotherm.

1.0. Introduction

Increasing water pollution is becoming a matter of great concern due of the growth in industries worldwide. Large quantities of coloured effluents are discharged into waters bodies each year. The discharge of dyes into natural water bodies presents a serious problem because of their persistence and non-biodegradable characteristics[1]. The disposal of highly coloured effluents containing dyes, pharmaceuticals and their derivatives products by industries causes extremely toxic effect to aquatic life even at very low concentration[2].

Dyes from textile industries are the most dangerous chemical pollutants in the environment, and it is estimated that there are more than 100,000 types of commercially available dyes with over 70×105 tons of dyes are produced annually[3]. Dye effluents reduce light penetration in water bodies. Light is essential for photo initiated chemical reactions necessary for aquatic life , especially to benthic organisms and the low biodegradability of these dyes present them as potential toxicants to marine lives.

Dyes used in textile industry are divided into anionic (acidic, direct, reactive), cationic that is all basic dyes and non-ionic (disperse dyes) groups[4]. Wastewater of textile industries commonly contains a variety of synthetic colours that have carcinogenic and mutagenic properties and are very stable in sunlight[5].

Basic dyes are salts of the coloured organic bases containing amino groups and amine also combined with a colourless acid, such as hydrochloric or sulphuric. They are brilliant and most fluorescent among all synthetic dyes but have poor light and wash fastness. Cotton dyes having higher fastness properties replace in dyeing cotton for them. Basic dyes are cationic which have positive electrical charge and are used for anionic fabrics which are negative-charge-bearing, such as wool, silk, nylon, and acrylics where bright dying is the prime consideration.

Victoria Blue is a non-biodegradable basic blue dye with molecular weight 506.1 and molecular formula $C_{33}H_{32}ClN_3$. The IUPAC name for victoria blue is (N-[4-[[4-(Dimethylamino)-phenyl] [4-(phenylamino)-1-naphthyl] methylene]-2, 5-cyclohexadien-1-ylidene]-N-methyl-Methanaminium chloride). Victoria blue is soluble dark blue to brown powder with a melting point of 206°C. Victoria Blue dye being cationic in nature carries a partial positive charge on nitrogen atom. Itis extensively used for dyeing wool, silk and cotton. It is also used for staining in microscopic work[6]. This dye is known to cause strong coloration and toxicity in the waste water and can produce irritation to eyes and respiratory system. It may even promote tumour growth in some species of fish[7]. Victoria blue and other similar dyes are a major concern as water contaminants. Dyes may cause cyanosis, convulsions, tachycardia, and dyspnoea, if consumed. They may cause irritation to the skin and arealso dangerous to biota. Dyes may also be problematic if they are broken down anaerobically in the sediments, as toxic amines are often produced due to incomplete degradation by bacteria[8]. Synthetic dyes have a complex aromatic structure which gives them physiochemical, thermal, biological, and optical stability[9]. Some of the dyes or their metabolites are not only toxic or mutagenic but also carcinogenic[10]. Most dyestuffs are designed to be resistant to environmental conditions such as light, heat, and microbial attack and also to oxidizing agents[11], making the biodegradation of dyes a very slow process[12].

The conventional technologies used for the removal of dyestuffs include chemical coagulation/ flocculation, ozonation, membrane filtration, anaerobic degradation. These methods suffer from having low efficiency for removal of reactive dyes and are very expensive. These processes are therefore not very effective in treating wastewater containing dyes. They are also not cost effective especially for developing countries like Zimbabwe. The use of lignocellulosic adsorbents has recently gained much attention as they are readily available. Biosorption is currently being used extensively for the removal of organic and inorganic micro pollutants from aqueous solutions. The most common and efficient adsorbent for waste water treatment is activated carbon. However its use is limited especially in developing countries, because of its high cost and loss during the regeneration step[13].Thus there is need for more research work in order to develop an equally efficient adsorbent that is readily available even to the poorest countries. Natural materials especially those from agricultural have recently gained much attention because of their low cost and availability[14].

Agricultural wastes have little or no economic value and they also pose a disposal problem hence using them as biosorbents will be of a great significance. Of late there has been an increase in the use of plant waste products for dye removal by biosorption from wastewater because of their natural availability and the high degree of dye removal achieved under laboratory conditions[15,16,17]. Agricultural waste materials such as coir pith, seed shell, corncob, tree bark, etc have been used for environmental remediation[2,18]. Several studies on removal of victoria blue on low-cost biosorbent materials have been reported. Meenakshi sundaram et al.[19] have studied the biosorption of victoria blue using bentonite which is natural clay. Kumar et al.[2] have studied the biosorption of victoria blue on Prosopis juliflora bark, Ayad et al.[20] on zeolite.

Adsorbents prepared from sugarcane bagasse, an agro-industrial waste, have been successfully used to remove the dye methyl red from an aqueous solution in a batch reactor. These adsorbents have been investigated by Saiful Azhar et al.[21] by pre-treating sugarcane bagasse with formaldehyde and sulphuric acid. However there has not been found an adsorbent that is effective as activated carbon thus there is need for further research on other alternative biosobents. In this present study the authors report the use of maize tassels for removal of Victoria blue. Maize tassel, a waste biomaterial, is the ‘male’ flower of the maize plant that forms at the top of the stem. The tassel grows at the apex of the maize stalk and end up as agricultural waste product after being involved in fertilisation. It is discarded by farmers in large quantities with the rest of the plant once the cobs have been harvested[22]. The gestation period of maize is about three to four weeks after pollination and fertilisation in which the tassel plays a vital role. This means that outside pollination and fertilisation the tassels serves no other useful purpose to the farmer hence they are usually cut off [22,23]. The fiber parts of the plant are rich carbohydrates. Apart from Zimbabwe maize tassels are found in large quantities from maize producing countries such as South Africa, Mexico, Brazil, China, Indonesia, Argentina and USA. Maize tassel adsorbent properties towards phenols have been investigated by Muchanyereyi et al.[24] and its potential to remove methylene blue has also been investigated [25]. In these studies maize tassels proved to have good adsorbent properties towards organic pollutants.

Fig 1: Maize Tassel

2.0 Experimental

2.1 Preparation of acid-treated maize tassels

The maize tassels were collected from Hermann Gmeiner High School garden in Chiwaridzo, Bindura, Zimbabwe. Maize tassels were plucked off the woody parts of the maize plant, thoroughly washed with water to remove sand and sun dried for 7 days. The dry biomass was milled and then fractionated using 100 - 300 µm analytical sieves. The fractionated maize tassel powder (150 g) was weighed in a clean dry beaker of capacity 1 L containing 150 mL, 97% H_2SO_4 for 24 h followed by refluxing in a fume hood for 4 h. After cooling, the reaction mixture was filtered, and the filtrate was washed repeatedly with distilled water and soaked in 1% $NaHCO_3$ solution to remove any remaining acid. The sample was then washed with distilled water until the pH of the acid treated maize tassel was between 6 - 7, dried in an oven at 120^{o}C overnight and kept in a glass bottle until use[26].

2.2 FT-IR Characterization of prepared acid-treated maize tassels

Functional groups present in the adsorbent were studied by Fourier Transforms Infrared Spectrophotometer(Fisher Thermo Scientific, Niolet iS5, UK)over the wavelength region of 400-4000 cm^{-1}.

2.3 Preparation of stock solution

A stock solution of VB was prepared by dissolving 1.000g of dye in 1dm^3 of distilled water. The test solutions were prepared by diluting the stock solution to desired concentrations. The concentrations range from 50 – 100ppm.

2.4 Adsorption studies

Victoria blue biosorption was performed by agitating Erlenmeyer flasks on a mechanical shaker at 160 rpm, the flask contained different amounts of acid treated maize tassels ranging from 0.2-1.0g and 50cm^3 of VB solutions at 100 ppm concentration. The suspensions were shaken at room temperature (25˚C ± 1˚C) and the pH was kept constant at 5. Samples of $15cm^3$ were taken from the mixture during stirring at 30 minutes intervals for determining the residual concentration in the medium.

2.5 Effect of initial VB concentration

The studies on the removal of victoria Blue dye on acid treated maize tassel was carried out at different initial concentration of dye from 50 to 100 ppm at room temperature and contact time 30 minutes. The pH was kept constant at 5 and biosorbent dosage of 1g was used.

2.6 Effect of contact time

Adsorption was measured at contact times 5; 10; 15; 20; 25 and 30 minutes. This was done at room temperature with pH kept constant at 5 and the biosorbent dosage also kept constant at 1 g.

2.7 Effect of adsorbent dosage

The effect of biomass dosage on the biosorption of victoria blue was studied using biomass dosage of 0.2; 0.4; 0.6; 0.8; and 1.0 g while holding the pH constant at 5, initial concentration VB of 100 ppm, contact time 30 minutes and at room temperature.

2.8 Effect of pH on VB adsorption

The effect of pH on the biosorption was investigated in the range 2-10 at room temperature. The pH of solution was adjusted to desired value by adding 0.1M HCl or 0.1M NaOH solution to the initial solution. The analysis was done at room temperature with all other parameters kept constant: pH at 5, initial concentration 100 ppm, biosorbent dosage 1 g.

2.9 Effect of temperature on adsorption

The effect of temperature was performed at different temperature range 25; 50; 75 and 100^oC. This was done with all other parameters fixed; pH 5, initial concentration 100ppm, biosorbent dosage 1 g and contact time of 30 minutes.

2.10 Analysis

The concentration of the VB dye solutions before and after biosorption was determined spectrophotometrically using UV/Vis spectrophotometer (Genesys 10S UV-Vis). The absorbance of colour was read at 599 nm (λ_{max}) (Meenaksh isundaram et al.[19] The values of percentage removal and the amount of VB adsorbed were calculated using equation (1) and (2)

$$Percentage removal = \left\lfloor \frac{(C_i - C_f)}{C_i} \right\rfloor \times 100 \qquad (1)$$

$$Amount adsorbed (Q_e) = \left(\frac{C_i - C_f}{m} \right) \quad (2)$$

Where: C_i is the initial concentration in ppm, C_f is the final concentration in ppm and m is mass of adsorbent in $g L^{-1}$.

3.0 Results and Discussion

3.1 FT-IR Analysis of acid –treated maize tassels.

The results of the FTIR analysis of the untreated and acid-treated maize tassels (Fig.2) show different stretches of bonds appearing at different peaks as summarized in Table 1.

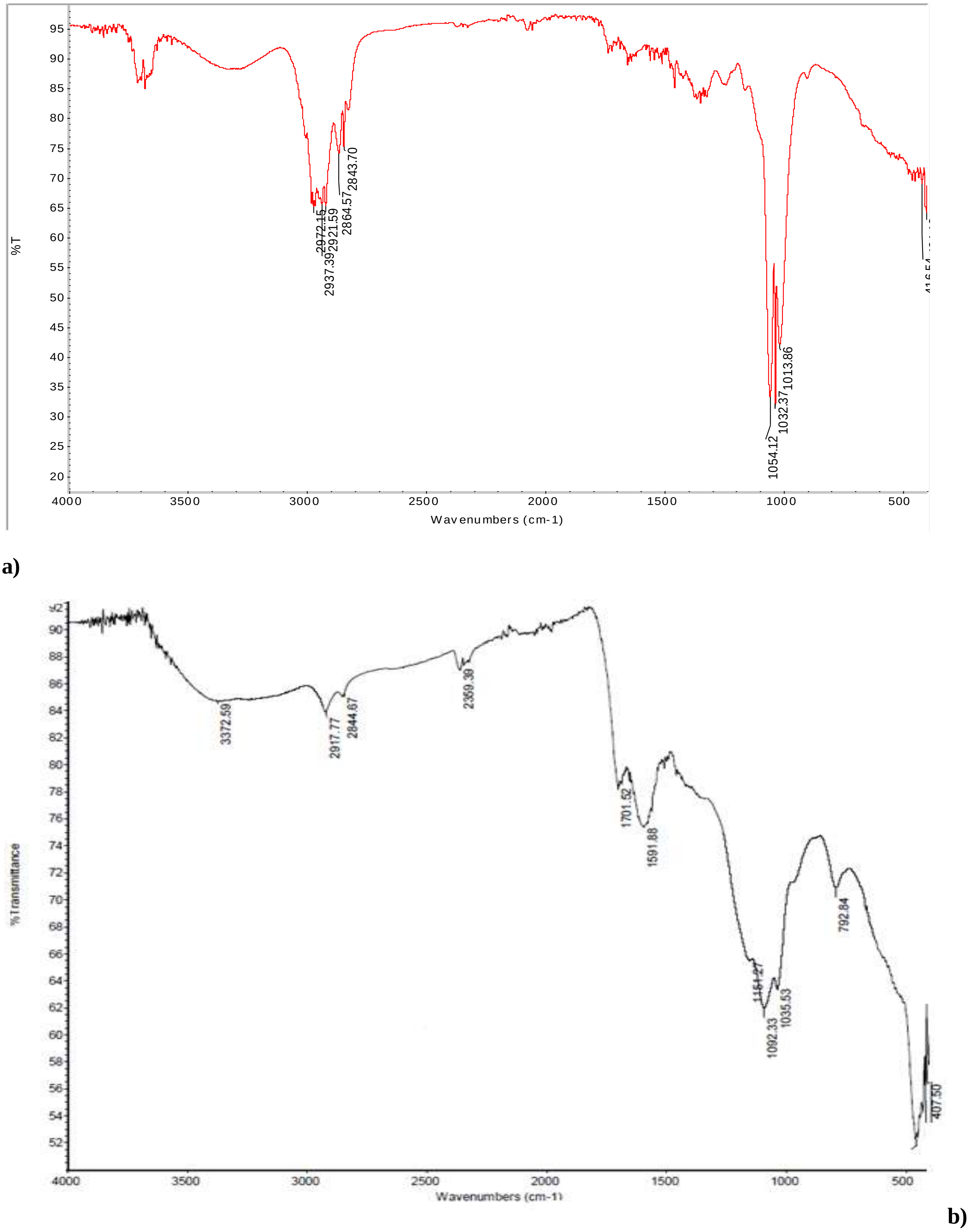


Figure2: FT-IR spectra of (a) untreated and (b)acid -treated maize tassels

Table 1: FT- IR bands

Bands/cm^{-1}	Assignment	Reference
3372.59	O-H Stretch (alcohols, phenols)	2,10,27
2917.77	-C-H (alkane), CH_2- asymmetric stretch.	28,29,30
2844.67	-C-H (alkane), aldehydic C-H stretching.	28,29
1701.52	-C=O stretch with conjugated aromatic ring, aldehydes, ketones, carboxylic acid.	1,31
1591.88	-C=C- (arene/aromatics)	28,29
1161.27	-C-O-C- asymmetric stretch in phase ring	29,30
1092.33	-C-O/ C-N	28,30
1035.53	-C-O or C-H.	30,32
792.84	$-CH_2$	29

The peak at 3372.59 cm^{-1} is an -O-H stretch, suggesting the presence of alcohols and phenolic compounds. The broad shoulder for this peak in the region 3300-3100 cm^{-1} can be attributed to the carboxylic acid O-H stretch. The presence of carboxylic acid functional groups is further confirmed by the C=O stretch at 1701.52 cm^{-1}. No peaks were observed in the regions 2220-2260 cm^{-1}and 1180-1360 cm^{-1} indicating the absence of nitriles (-C$\equiv$N) and amines(C-N stretch), respectively. The absence of amine functional groups is also further confirmed by lack of a doublet –N-H stretch within the region 3300- 3500 cm^{-1} for NH_2. The presence of aromatic compounds in the treated maize tassel is confirmed by the -C=C- stretch within the region 1500- 1600cm^{-1} i.e. at 1701.52 cm^{-1}. This is further confirmed by a broad =C-H stretch in the region 3000-3100 cm^{-1}. There is no clear peak observed in the region 3600-3650cm^{-1} indicating that almost all the -O-H stretches are due to H-bonded functionalities of alcohols and phenols i.e. no free –O-H stretches. However the intensity of these stretches were generally weak, and this is expected due to conc-H_2SO_4 treatment which causes reduction of the -OH functional groups. The stretch at 2369.30 cm^{-1} is not a common peak in literature but this is close to the maximum frequency range for nitriles (2260 cm^{-1}) or alkynes (2270 cm^{-1}). Again, this peak did not appear on the FTIR spectrum before treatment (figure 2). The peaks at 1701.53 cm^{-1} and 1591.88 cm^{-1} were not observed for the untreated maize tassel, indicating the effect of acid treatment i.e. introducing either carboxylic acid or aldehydic –C=O functional groups. However, alkynes should show a $\equiv$C-H stretch at around 3300 cm^{-1} with a strong and sharp intensity and this is not observed on the spectrum in that frequency range. Alkenes give a peak in the frequency range 1600-1680cm^{-1} due to –C=C- stretch. No peak was observed in this frequency region, hence no stretch can be assigned to the presence of alkynes.

3.2 Adsorption isotherms

The study of adsorption isotherm has been of important and significant in the waste water treatment by adsorption technique, as they provide an approximate estimation of the adsorption capacity of the adsorbent[19].The Langmuir and Freundlich models are the mostly widely used models in the case of adsorption and are the earliest and simplest known relationships describing the adsorption equation[33,34].The Langmuir model assumes a monolayer adsorption of solutes onto a surface comprised of a finite number of identical sites with homogeneous adsorption energy. It is based on the following assumptions. It assumes that the surface of the adsorbent is uniform, adsorbed molecules do not interact, adsorption occurs through the same mechanism and the adsorbed layer is made up of a single layer of molecules.The Langmuir isotherm is described by Equation 3:

$$q_e(1+bC_e) = q_m bC_e \quad (3)$$

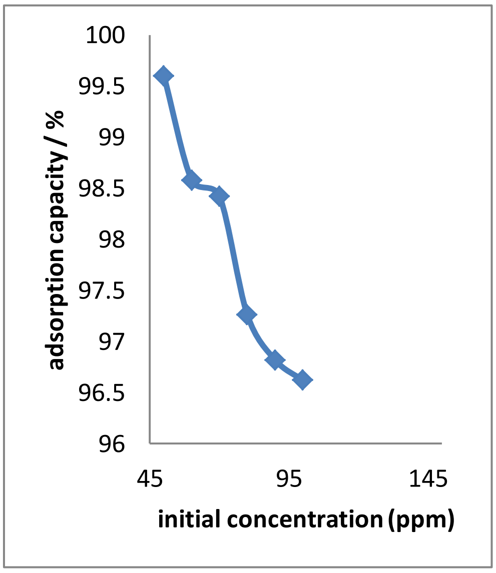
Where: qe is the amount of solute adsorbed per unit weight of adsorbed at equilibrium (*mgg^{-1}*), Ce isequilibrium concentration of the solute in the bulk solution (*mgL^{-1}*), q_m is maximum adsorption capacity (*mgg^{-1}*) and b is temperature dependent constant (*Lmg^{-1}*). The Freundlich isotherm is an empirical expression that takes into account the heterogeneity of the surface and multilayer adsorption to the binding sites located on the surface of the sorbent. This empirical model can be applied to multilayer adsorption, with non-uniform distribution of adsorption heat and affinities over the heterogeneous surface[35]. The Freundlichisotherm is given by equation 4

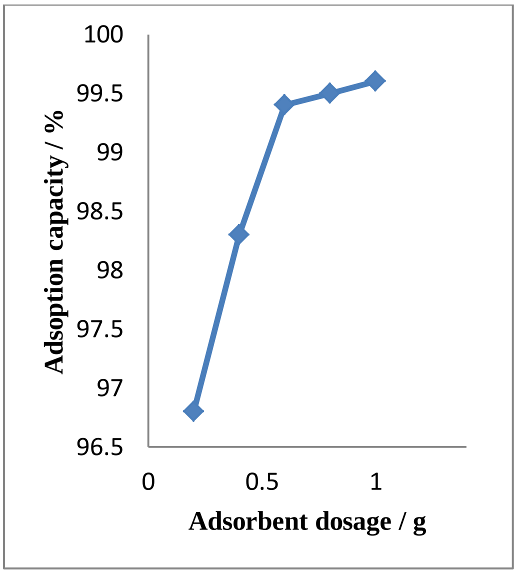
$$C_e^{1/n} Q_e = K_f C^{1/n}{}_e \quad (4)$$

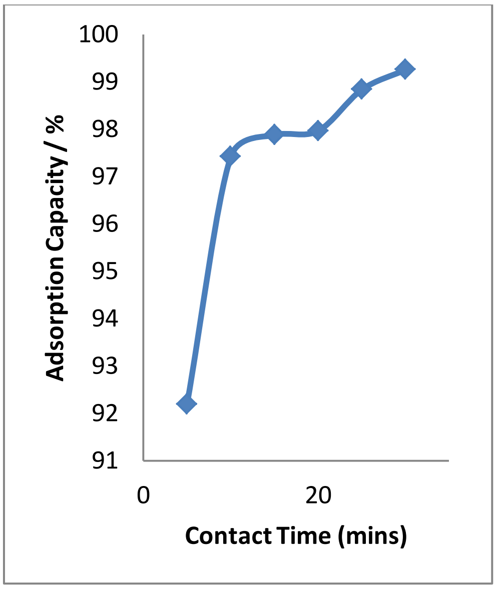
Where Q_e is equilibrium absorbent amount on the adsorbent, K_f is approximate indicator of adsorption capacity, C_e is equilibrium concentration of adsorbate, $^1/n$ is approximate indicator of adsorption intensity, when $^1/n < 1$ means there is high sorption

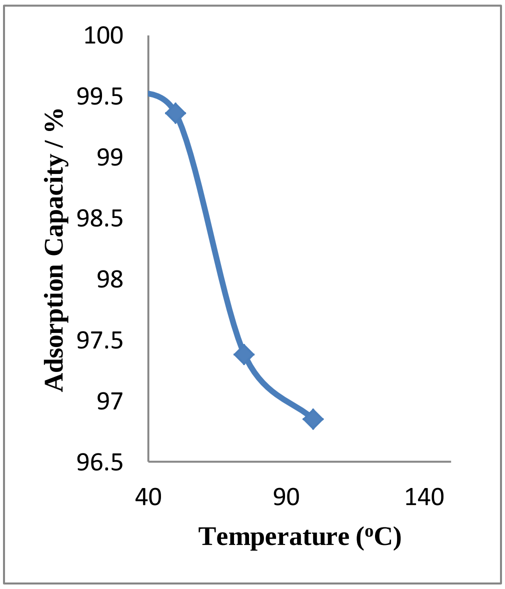
3.2.1 Effect of initial concentration

The studies on the removal of victoria blue dye on acid treated maize tassels were carried out at different initial concentration of dye from 50 to 100 ppm for contact time of 30 min. The percentage removal decreases exponentially with the increase in the initial concentration of dye. As shown on table 4.4 the maximum adsorption capacity reached was 99.60% when the initial concentration of VB was 50 ppm. This is due to the reduction in the immediate solute adsorbent surface compared to the relative larger number of required site for the high concentration of dye. The optimum removal of dye was at initial concentration of 50 ppm. Similar trend wasreportedbyMeenakshisundaram et al.,[19] who studied novel eco-friendly adsorbents for the removal of victoria blue dye.









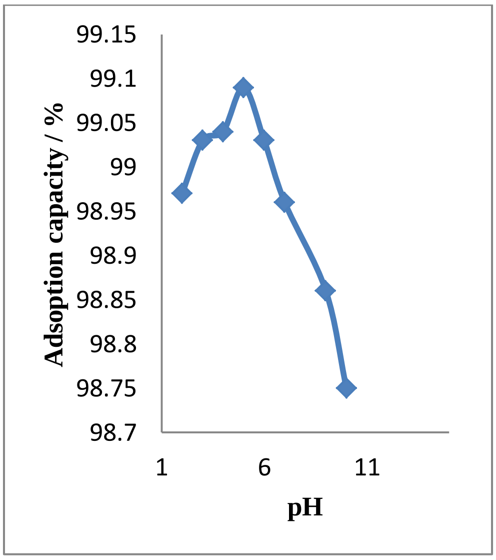


Fig 3: Effect of important parameters on adsorption capacity of acid-treated maize tassels for victoria blue

3.3 Effect of adsorbent dosage

The effect of adsorbent dosage was studied using adsorbent dosage ranging from 0.2 g to 1.0 g. This was investigated at an optimum pH 5 and the results showed that as the adsorbent dosage increased the adsorption capacity also increased, in this study from 96.8% to 99.6%. The maximum adsorption capacity was observed at 1.0 g dosage (Fig 3). This phenomenon can be due to the greater availability of activity sites or surface area as dosage amount increases thus making easier penetration of the victoria blue to the adsorption sites[36].

3.4Effect of contact time

Adsorption experiments were carried out at different contact time of 5, 10, 15, 20, 25 and 30 minutes, at a constant pH of 5, initial concentration 100 ppm and adsorbent dosage of 1.0 g. Figure 3 shows that the percentage removal of Victoria blue increases with increase in contact time. The adsorption capacity was found to be rapid at the initial period of contact time and then become slow and almost stagnant with increase in contact time. The maximum adsorption capacity of 99.25% was obtained at 30minutes contact time. The percentage of biosorption is almost constant indicating the attainment of equilibrium conditions. The presence of adequate external surface area on the acid treated maize tassel may have boosted the rate of adsorption to be fast in the initial stages which was followed by a slower internal diffusion process, which maybe the rate determining step [37].

3.5 Effect of temperature

Effect of temperature was investigated at pH of 5, contact time of 30minutes, adsorbent dosage of 1.0 g and temperature range of 25^{o}C to 100^{o}C. The adsorption capacity of victoria blue decreases with increasing temperature, this indicates that the adsorption is endothermic adsorption. The maximum adsorption capacity obtained was 99.53% at 25^{o}C. A decreased adsorption capacity of the victoria blue with increasing temperature couldbe attributed to the slight screen for the mobility of the dye molecules with temperature and is an indication of a physiochemical mechanism. An increaseintemperature decreases the viscosity of the solution. Changing the temperature does change the equilibrium capacity of the adsorbent during the adsorption process[38]and enhance the rate of intraparticle diffusion of the sorbate. The removal of victoria blue by acid treated maize tassels decreased from 99.53% to 96.85%. Similar trend was reported forthe adsorption of basic and reactive dyes [2].

3.6 Adsorption isotherms

The Freundlich isotherm (Fig 3-4) shows the correlation coefficient values (r^2: (0.7252-0.9923) are less than the Langmuir isotherm (Fig 3-4, Table 3) correlation coefficient value (r^2:0.9983-0.9999) (Table 3). These values indicate that theexperimental data fitted well the Langmuir isotherm than the Freundlich isotherm for the removal of VB dye. The monolayer (Langmuir) sorption process was the dominant one as compared with the multilayer process (Freundlich). Also from the Freundlich isotherm parameters(Table 2) the calculated n value in all cases are less than 1 suggestingunfavourable adsorption which is unbeneficial[39].

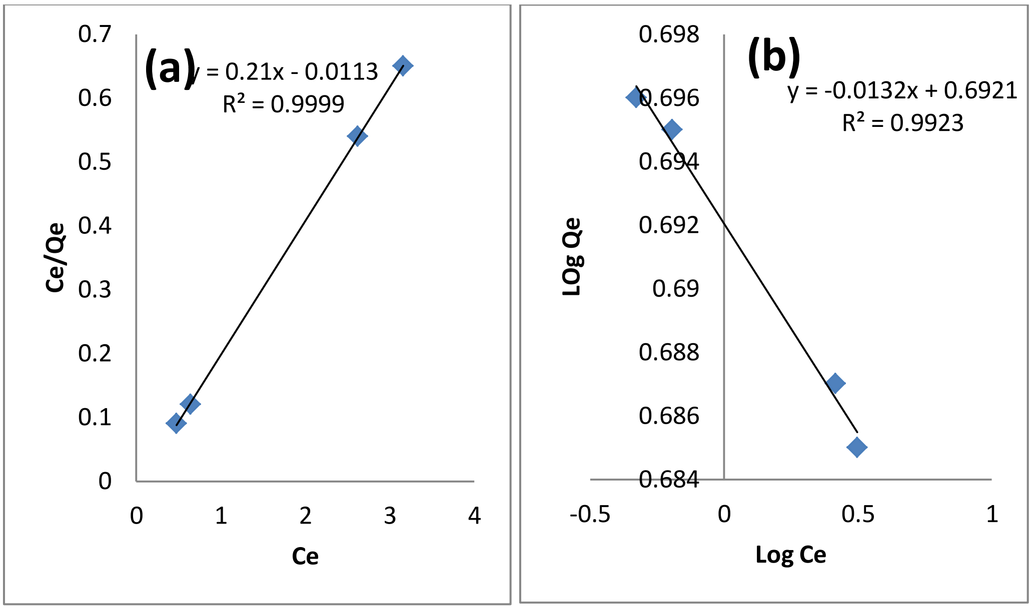


Fig 3: Plot of Langmuir adsorptionisotherm (a) and Freundlich adsorption isotherm for effect of temperature

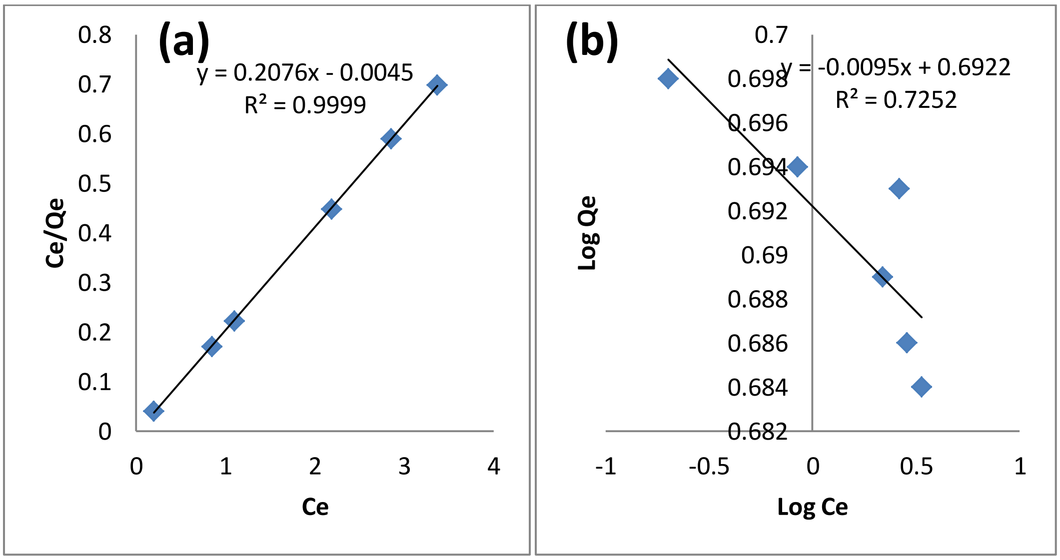


Fig 4.: a) Langmuir and b) Freundlich adsorption isotherm, for the effect of Initial VB Concentration

Table 2: Freundlich isotherm parameters

Parameter	**K_f**	**n**
Adsorbent dosage	0.6906	-59
pH	0.6944	-91
Initial Concentration	0.6922	-105
Contact Time	0.6962	-31
Temperature	0.6921	-75

Table 3: Table of R^2- Values obtained

Parameter	**Langmuir**	**Freundlich**
Adsorbent Dosage	0.9998	0.903
pH	0.9983	0.8407
Initial Concentration	0.9999	0.7252
Contact time	0.999	0.8771
Temperature	0.9999	0.9923

3.7 Adsorption Mechanism

The mechanism of the adsorption of organic pollutants it's still ambiguous many suggestions have been put forward. Adsorption is a surface phenomenon, thus the functional groups on the surface of the adsorbent play acrucial role in the adsorption process.For this present study the suggested mechanism is that of acid-base interaction. The dye acts as an acid that can receive electrons from the electron donating groups on the adsorbent (maize tassels)e.g. the oxygen atoms. The maize tassel acts as a base. There is evidence in literature that most lignocellulosic materials have basic functional groups on their surfaces[40].

Conclusion

Results of the present study suggest that acid -treated maize tassels can be used as a low cost biosorbent for effective removal of victoria blue dye from aqueous solution. Victoria blue dye adsorption was found to be pH dependent and maximum removal was observed at pH 5. An increase in the acid treated maize tassel dosage led to an increase in victoria Blue dye removal due to a corresponding increase in the number of active sites. The optimum adsorbent dosage required was 1.0 g, initial concentration of 50 ppm and contact time of 30 minutes. The adsorption process required low temperature 25 ^{o}C suggesting it can be done at room temperature. The data obtained fitted well in Langmuir absorption isotherm for all parameters suggesting the monolayer adsorption capacity of victoria blue dye on sulphuric acid treated maize tassels.

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