Removal of Methylene Blue and Neutral Red Dyes from Spiked Waste-Water Using Edtad Modified Cellulose Obtained from *Pennisetum purpureum*

Brenda Oyaro[1*], Elijah Ngumba[2] and Ali Mohammed Salim[2]

[1]*Department of Chemistry, College of Pure and Applied Sciences, Jomo Kenyatta University of Agriculture and Technology, P. O. Box 62000-00200, Nairobi, Kenya.*
[2]*Department of Chemistry, Jomo Kenyatta University of Agriculture and Technology, P. O. Box 62000-00200, Nairobi, Kenya.*

Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Original Research Article

ABSTRACT

The presence of dyes in aquatic effluents is highly visible, undesirable, carcinogenic to human beings and toxic to living organisms. EDTAD modified cellulose extracted from *Pennisetum purpureum* was studied as an adsorbent for the removal of cationic dyes from spiked waste-water. Shimadzu Fourier Transform Infrared spectrophotometer, was used to analyze functional groups of cellulose and EDTAD modified cellulose. The characteristic peaks at 1260 cm^{-1}, 1634 cm^{-1}, 1058 cm^{-1}, and 1740 cm^{-1} indicated formation of ester bond linkages associated with the introduction of EDTAD thus successful modification of cellulose. Shimadzu UV-VIS 1800 series determined the degree of dye concentration. Optimum adsorption was determined by varying the dosage, pH, contact time, and the initial concentration of the dyes.

An increase in adsorbent mass increased the percentage removal for both dyes up to a maximum of 98%. Low pH levels prompted low percentage removal as compared to higher pH values for both dyes. Kinetic studies were carried out using the Freundlich and Langmuir isotherms, to investigate the rate of adsorption. Methylene blue and neutral red dyes seemed to conform to langmuir with

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*Corresponding author: E-mail: brndkwmbk13@gmail.com;

high R^2 values of 0.999 and 0.997 respectively while freundlich isotherm had values of 0.9969 and 0.9835, indicating that the adsorption surface of the EDTAD modified cellulose was monolayer. Methylene Blue adsorption capacity was 200mg/g while for Neutral Red dye was 149.25mg/g. The kinetics were well depicted by pseudo second order model whose correlation coefficients were closer to unity than those of the first order model. The efficiency of EDTAD modified cellulose from *Pennisetum purpureum* as an adsorbent in the removal of dyes renders it a remedy to the various problems caused by dyes to aquatic life and human beings.

1. INTRODUCTION

Dyes are substances that provide color, when applied to a substrate, by a process that modifies temporarily the crystalline structure of the colored substances [1]. Dyes have found quite several applications as colorants in various industries such as textiles, plastics, pharmaceuticals, and food industries [2]. Due to the high stability of dyes to chemicals degradation processes and the environment, these dyes escape the conventional waste-water treatment processes and accumulate in the environment [3]. Over seven hundred tons of dyestuff are produced annually [4]. Two percent of produced dyes are discharged directly in aqueous effluent, and 10-15 % are lost during the textile coloration process [5]. A number of these dyes have been associated with some negative environmental and health risks such as toxicity to aquatic organisms, endocrine disruption, hypoxia, decrease light penetration, eutrophication, amongst others.

This phenomenon limits downstream beneficial uses of water [6-8]. There is a need to minimize levels of dyes in improving the quality of domestic water as a key factor in ensuring a healthy working community.

Commercial adsorbents such as reverse osmosis, flotation were initially used for dye removal. They are costly, need a lot of energy to operate and some form complexes, in availability hence rendered them less effective and hence there is need to look for the cheap way such as the introduction of bio-sorbents [9]. Various bio-sorbents have been used in dye and heavy metal removal such as rice husks, tea wastes and have proved to be efficient since they are eco-friendly, cheap and easily available. The aim of this study was to investigate the use *P. purpureum* which is an important feed-stock in the extraction of cellulose for use in the adsorption of dyes from waste-water. To the best of our knowledge, there is no available literature on the use of bio-sorbents from *P. purpureum* for the removal of dyes from wastewater.

2. MATERIALS AND METHODS

Pennisetum purpureum stem samples used in this research study were obtained from Jomo Kenyatta University of Agriculture and Technology University in Kenya. They were then washed, dried, crushed, and finally ground to a fine powder using a mechanical grinder. The main instruments used in the analysis were FTIR and UV-VIS. The reagents used were sodium hydroxide, nitric acid, anhydrous DMF, sodium hypochlorite, ethanol, sodium bicarbonate solution, and acetone.

All of these were of analytical grade and obtained from Chem-lab Limited-Kenya with a 99.8% purity. EDTAD was prepared by the method outlined by [10].

2.1 Extraction of Cellulose from Biomass

The ground samples of *Pennisetum purpureum* were mixed with 10% (wt/v) aqueous NaOH solution in a ratio of 1:10. The mixture was then heated at 70^0C for 3 hours, followed by filtration of the pretreated biomass, and then washed several times with distilled water until the residue became clean. The hemicellulose-free biomass cake was air-dried for 48 hours and then mixed with pre-prepared 10% nitric acid in a ratio of 1:10. The mixture was then allowed to stand for 3 hours at 70^0C in a water bath with a shaker. The residue was filtered using a muslin cloth and washed with distilled water repeatedly then air-dried. Sodium hypochlorite was added to the obtained dry residue in the ratio of 1:10 and then heated in a water bath for 3 hours at 70^0C. A muslin cloth was then used to filter the obtained mixture. The resultant residue was washed severally with distilled water until the residue

became clean. The residue was then air-dried to obtain cellulose.

2.2 Modification of Cellulose

Synthesized EDTA was mixed with extracted cellulose in the ratio of 1:3. The mixture was then put into 210 ml anhydrous DMF and then heated at 75 ^{0}C for 24 hours.

The resulting mixture was then filtered using muslin clothing. The obtained residue was washed with DMF then rinsed with distilled water. The filtrate obtained was washed with sodium bicarbonate solution, rinsed with distilled water and dried. The dried sample was reacted with 95% ethanol and 5% acetone and then dried in an oven for 1 hour at 80 ^{0}C. The resultant substrate was then cooled in a desiccator to obtain EDTAD modified cellulose.

2.3 Characterization of Cellulose and EDTA Modified Cellulose using FTIR

Shimadzu Fourier Transform Infrared spectrophotometer, Model FTS¬ 8400 was used to analyze functional groups of cellulose and EDTAD modified cellulose. The analysis was run using the KBr pressed pellet technique. The KBr pellets of samples were prepared by mixing 20 mg of samples, finely grinding, with 200 mg KBr (FT¬IR grade). The 13 mm KBr pellets were prepared in a standard device under a pressure of 75 kN cm^{-2} for 3 min. The spectral resolution was set at 4 cm^{-1} and the scanning range from 400 cm^{-1} to 4000 cm^{-1}.

2.4 Characterization of Cellulose and EDTA Modified Cellulose using UV-VIS

The degree of concentration of dye was determined by a Shimadzu UV-VIS 1800 series. The UV-VIS was used to determine the different wavelengths at which the dye solutions were absorbed before and after de-colorization by EDTAD modified cellulose.

2.5 Adsorption Experiments

2.5.1 Optimization of contact time, sorbent mass, PH and initial concentration

For contact time optimization, 6 conical flasks with 100 ml of synthetic wastewater were spiked with 8 ppm of the selected dyes (Neutral red or Methyl blue) they were in each case mixed with 0.5 g of the bio-sorbents at pH 7. The mixtures were then agitated using a mechanical shaker for different period ranging from 30 to 120 min.

In optimization of sorbent mass 0.4 g to 1.6 g of the bio sorbent were mixed with 100 ml of water sample containing 8 ppm of each dye separately in 6 conical flasks. The solutions were then agitated using a mechanical shaker for 90 min.

For pH optimization the pH of water samples in 6 conical flasks, containing 8 ppm of each dye was varied in the range of 2-10. Each sample was mixed with 0.5 g of the sorbent and the solution was agitated for 90 min.

For optimization of initial Concentration water samples of 100 ml in 6 conical flasks, containing varied initial dye concentration ranging from 2-20 ppm was mixed with 0.5 g of bio sorbent at pH 7. The solution was then subjected to agitation using a mechanical shaker for 90 min.

After each set period the solutions were then filtered using a Whatman® grade 1 filter paper. The amount of dye remaining in the solution was then determined using the UV/VIS machine (Shimadzu UV-VIS 1800 series). All the experiments were carried out at room temperature.

3. RESULTS AND DISCUSSION

Fig.1 represent raw reeds sample and EDTA modified cellulose. The spectrum was run concurrently to point out which components of the raw material were removed after chemical treatment to obtain EDTA modified cellulose. The fingerprint region between 1000–2000 cm^{-1} shows the differences between raw material and EDTA modified cellulose. The peak at 3400cm^{-1} in both raw material and EDTA modified cellulose samples is characteristic of O-H bonds. The absence of the characteristic peak at 1740cm^{-1} as a result of C=O stretching vibration in the raw material sample was due to the successful removal of lignin material from the raw sample after chemical treatment.

Aromatic skeletal vibration is observed at 1518 cm^{-1} wavenumbers for EDTA modified cellulose yet for the raw material was at 1512 cm^{-1} indicating a good quantity of lignin was stripped off after chemical treatment. The presence of the characteristic peak at 1740 cm^{-1} was as a result

of asymmetric and symmetric stretching of the ester C-O bond and this was similar to that reported by [10] on zinc ions removal by EDTAD modified sugarcane bagasse and wood dust. The presence of the characteristic peak at 1630 cm^{-1} is a result of asymmetric stretching of the carboxylate ion. The characteristic peak at 1260 cm^{-1} for the raw material is attributed to stretching or deformation of the C-N bond. The IR Spectra for C-N-C asymmetric stretching vibration is at 1058cm^{-1} this was similar to that reported by [11] on biosorption of methylene blue from aqueous solution on spent cottonseed hull substrate. The above IR Spectra's indicated the formation of ester bond linkages associated with the introduction of EDTA thus successful synthesis of EDTA modified cellulose.

It is evident from the Fig. 2 that methylene blue % removal increased with an increase in time from 20 minutes to 120 minutes after which it remains constant. The highest % removal was 96 at 120 minutes while the lowest was at 20 minutes with a 55% removal for neutral red. The highest % removal was 96 at 120 minutes while the lowest was at 20 minutes with a 50% removal for methylene blue. This behavior arises from the fact that initially there are numerous binding sites from the large surface area available leading to high adsorption capacity. As time elapses most of the binding sites become fully occupied and a further increase in contact time does not lead to any change. These findings are in agreement with what was reported by [12] on removal of methylene blue from water by cellulose/graphene oxide fibers.
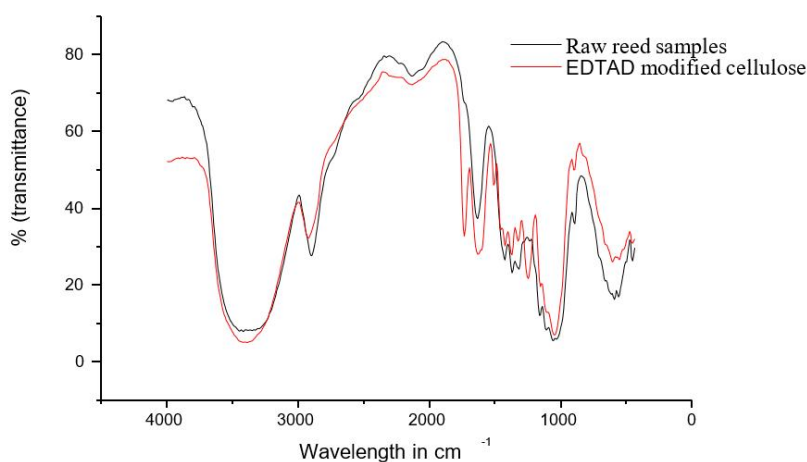

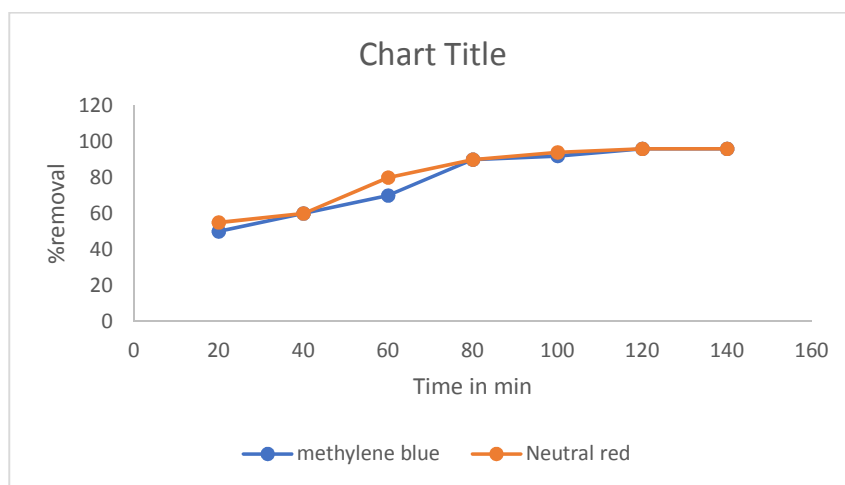**Fig. 1. FTIR spectrum of cellulose and EDTA modified cellulose**

Fig. 2. Percentage Removal of dyes with time at temp: 298k, dye concentration: 50 ppm, dosage: 0.05 g, pH:6
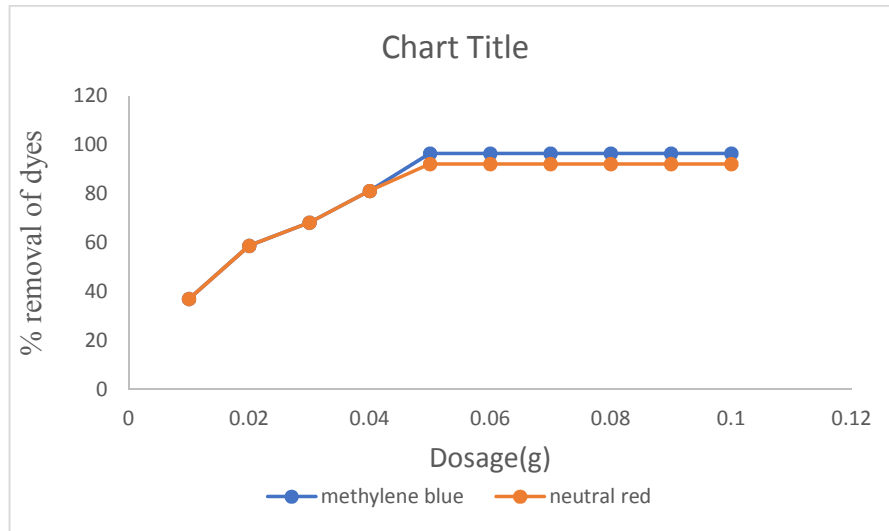
Fig. 3. Percentage removal of dyes with dosage at temp: 298 k, pH: 6, concentration: 50 ppm, time: 2 hrs

There was an increase in % removal of methylene blue with an increase in adsorbent dosage from 0.01-0.06 g after which % removal becomes constant. The % removal was highest at 96.5% for 0.05g and lowest at 0.01g with a % removal of 36.9%. For neutral red, there was an increase in % removal with an increase in adsorbent dosage from 0.01- 0.05 g after which % removal becomes almost constant. The % removal was highest at 92.2% for 0.05 g and lowest at 0.01 g with a % removal of 36.9%. This can be explained by the fact that there was an increase in the surface area hence active sites of the adsorbent as the adsorbent dosage increased thus leading to an increase in adsorption sites and adsorption capacity. After some time, the increase in adsorbent dosage does not lead to an increase in % removal due to the unsaturation of the adsorption sites. This is in agreement with what was suggested by [13] who reported similar findings with cottonseed hull substrate.
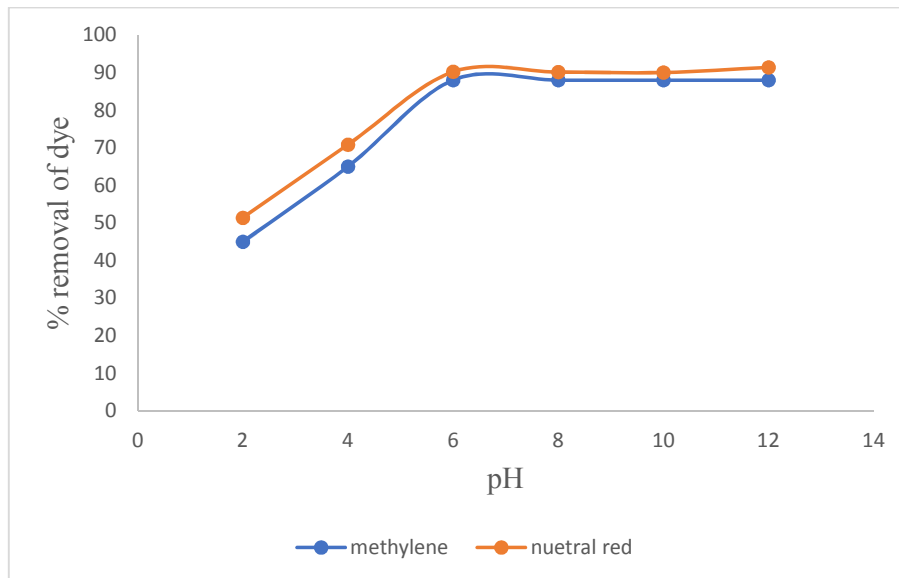

Fig. 4. Percentage removal of dyes with pH at temp: 298 k, dye concentration :50 ppm, time:2 hrs, dosage=0.05 g

It is observed that % removal for methylene blue increased with an increase in pH and reached a maximum at pH 6 then stagnated at basic pH values of 8. The highest % removal was obtained at pH 6 of 88% while the lowest was at pH 2 with a % removal of 45%. The highest % removal of neutral red was at pH 6 with a % removal of 95% and the lowest was at pH 2 with a % removal of 50%. In the acidic pH, it was suggested that there was an introduction of H_3O^+ ions which competes with positively charged methylene blue ions for active sites of the adsorbent having carboxylate ions thus low sorption capacity. At alkaline pH, OH^- ions are introduced and this results in a strong electrostatic attraction between the negatively charged active sites of the adsorbent and positively charged sites of the dye ions resulting in a high % removal. This observation was similar to that obtained by [14] who used cellulose/graphene oxide fibers. as an adsorbent.

The concentration was varied from 50 ppm to 200 ppm. From Fig. 4, the % methylene blue dye removal decreased with an increase in dye concentration. The highest % removal was 96% at 50 ppm with the lowest being 90% at 200 ppm. For neutral blue, the % removal decreased with an increase in neutral red concentration and the highest % removal was at 96% for 50 ppm and lowest at 93% at a concentration of 250 ppm. Generally, at low concentrations, there were high electrostatic forces between the dyes ions and the active sites of the adsorbent which resulted in high adsorption capacity as the dye

concentration increased this did not increase % removal, since the active sites of the adsorbent had been saturated and hence decreased the electrostatic attraction resulting in decreasing adsorption capacity. This result is similar to that obtained by [15] adsorption of safranin dye with *Bambusa tulda* as an adsorbent.

The observed scenario can be related to high repulsive forces, between the methylene blue ions and the adsorption sites of the adsorbent at low concentrations, thus high adsorption process. A further increase in concentration does not result in an increase in % removal since repulsive forces between the methylene blue ions and the active sites of the adsorbent reach saturation and this finding was similar to results reported by [16] who used nanocomposite of hydrolyzed polyacrylamide grafted xanthan gum and incorporated nanosilica for methylene blue removal.

3.1 Adsorption Isotherms

3.1.1 Langmuir sorption isotherm

This model suggests occurrence of sorption on the surface of the solid made up of elementary sites that can adsorb a sorbate molecule. Every sorption site has the ability of binding a sorbate independently. The linearized Langmuir model is given below:

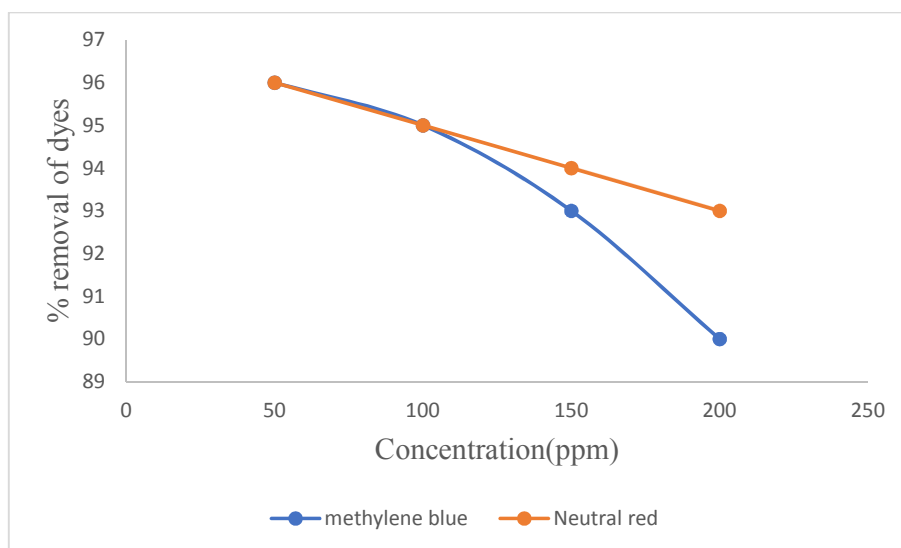$$\frac{C_e}{q_e} = \frac{1}{Q^o b} + \frac{C_e}{Q^o} \tag{1}$$

Fig. 5. Percentage removal of dyes with concentration at temp: 298 k, dye dosage: 0.05 g, time: 2 hrs, pH: 6

Where q_e (mg/g) and C_e (mg/L) are the sorbed metal ions on the sorbent and the metal ion concentration in the solution at equilibrium, respectively, b (L/mg) is the constant related to the affinity of sorbent for the sorbed species. Q^0 (mg/g) is known as the Langmuir constant, which represents the monolayer sorption capacity, i.e. a practical limiting sorption capacity when the surface is fully covered with metal ions. It assists in the comparison of sorption performances. The separation factor (RL) depicts the affinity amid the adsorbate and adsorbent evident from the equation:

$$RL = \frac{1}{(1+bC_o)}$$ (2)

With an RL value of more than 1shows that adsorption is less favorable. RL value less than1indicates the adsorption nature to be more unfavorable, linear (RL = 1), favorable and irreversible when (RL = 0).

3.1.2 Freundlich sorption isotherm

The Freundlich isotherm model describes the sorption of solute from liquid to solid surface and assumes that the stronger binding sites are occupied first and that the binding strength decreases with an increasing degree of site occupation. It proposes a monolayer sorption

with heterogeneous energetic distribution of active sites and/or interactions between sorbed species i.e multilayer sorption. The Freundlich model can be expressed by the following empirical equation which can be expressed further in logarithmic form:

$$q_{e} = K_{F}C_{e}^{n}$$ (3)

$$\log q_{e} = \log K_{F} + n \log C_{e}$$ (4)

Where KF (mg^{1-n} /g L^{n}) and n (dimensionless) represents the freundlich constants characteristic of the system. KF is indicative of the relative sorption capacity, whereas n is the measure of the nature and strength of the sorption process and the distribution of active sites. A slope ranging between 0-1 measures the adsorption intensity or surface heterogeneity. Whereas, a value below unity implies chemisorption process where 1/n above one is an indicative of cooperative adsorption [17].

In both dyes a monolayer adsorption is observed this is attributed to correlation coefficients in the langmuir model being close to unity than for the freundlich model. The maximum adsorption values for methylene blue and neutral red dyes were 200 mg/g and149.25 mg/g respectively.

Table 1. Langmuir adsorption isotherm and freundlch adsorption isotherm of methylene blue and neutral red dyes

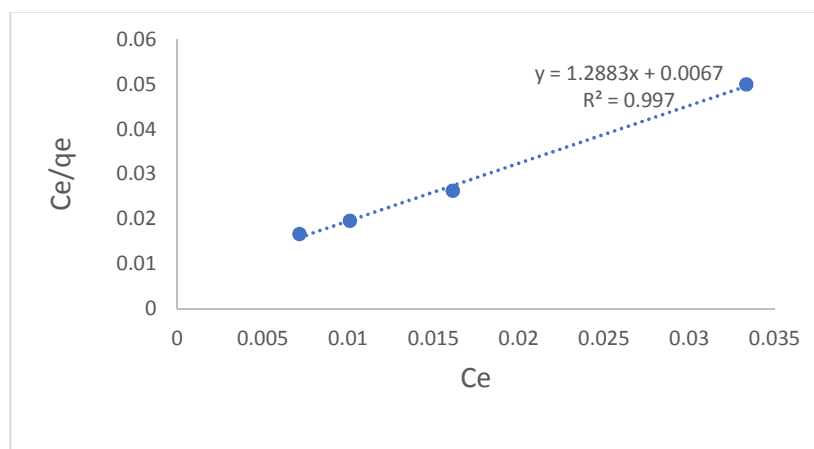|	Langmuir isotherm			Freudlch isotherm		
	q_o	k_l	r^2	k_f	1/n	r^2
Methylene blue	200	0.00543	0.9995	4.45	0.7624	0.9969
Neutral red	149.25	0.0052	0.997	1.8	0.7211	0.9835

Fig. 6. Langmuir adsorption isotherm of neutral red onto EDTAD modified cellulose temp: 298 k, dye concentration: 50 ppm, time: 2 hrs, pH: 6
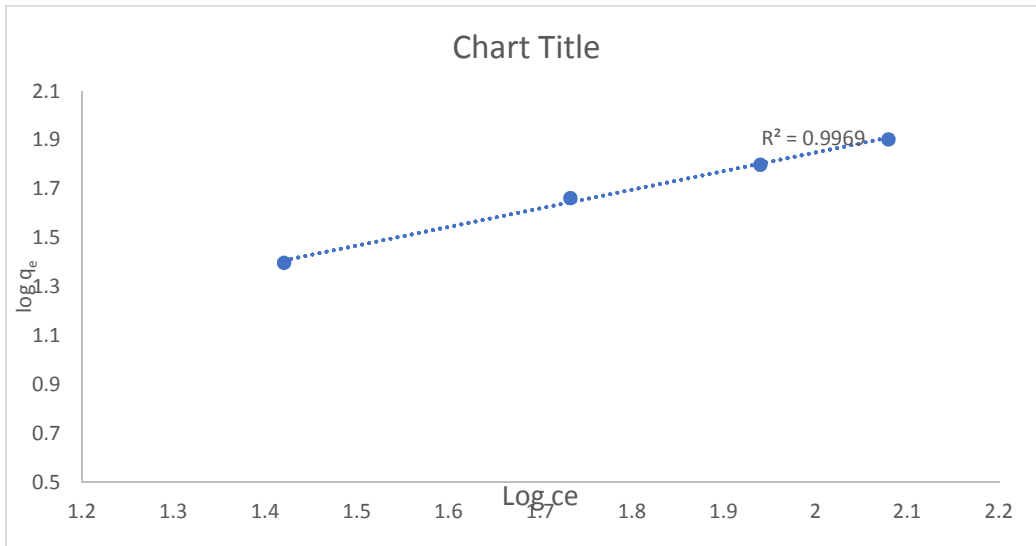
Fig. 7. Freundlich adsorption isotherm of methylene blue onto EDTAD modified cellulose

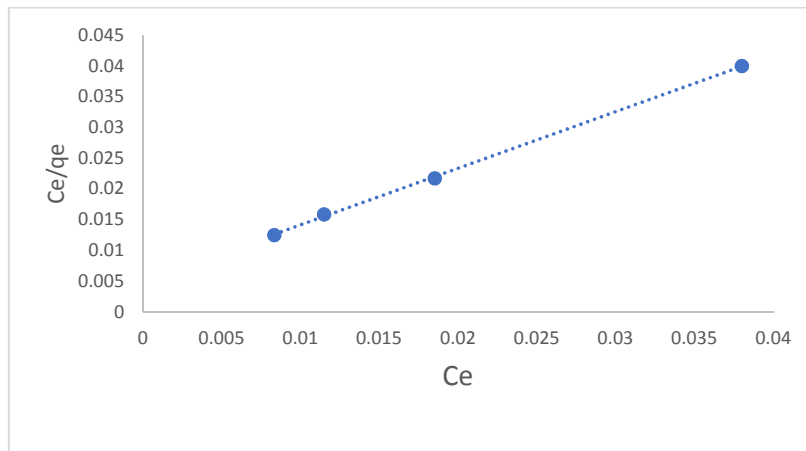**Fig. 8. Langmuir adsorption isotherm of methylene blue onto EDTAD modified cellulose temp: 298 k, dye concentration: 50 ppm, time: 2 hrs, pH: 6**
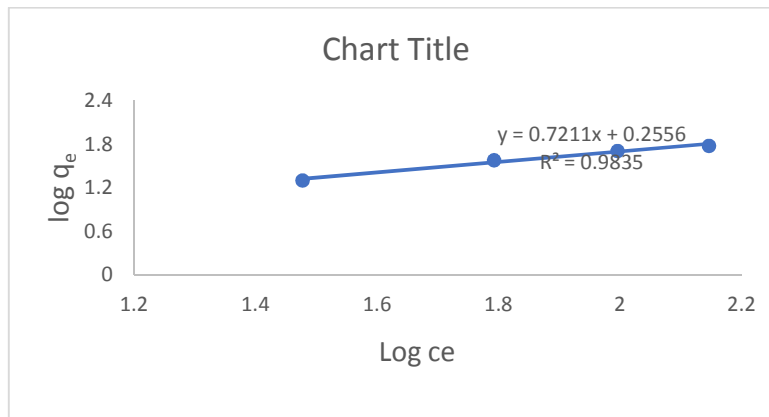

Fig. 9. Freundlich adsorption isotherm of neutral red onto EDTAD modified cellulose

3.2 Adsorption Kinetics

Pseudo first order model this is also known as lagergren's first order rate equation whose linear form is:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (5)$$

where k1 represents adsorption rate constant, qe mass adsorbed per unit at equilibrium, and qt mass adsorbed at a time t [18].

Pseudo second order model has a linear equation of

$$t/qt = (1/k_2 q_e^2) + (1/q_e)t \text{ (Ho)} \qquad (6)$$

where k_2 is the adsorption rate constant and qe mass adsorbed per unit at equilibrium [19].

In both dyes the second pseudo order model is relevant. This is attributed to the correlation coefficient r^2 which is closer to unity for the second pseudo order model than the r^2 values obtained by the first pseudo order model. The q_e calculated values of the second pseudo order model are close if not equal to the experimental values as shown in Tables 2 and 3.

Table 2. Pseudo first order and pseudo second order neutral red (N.R)

C_o(mg/g)	q_e experimental	Pseudo first order N.R			Pseudo second order N.R		
		q_e	k_1	r^2	q_e	K_2	r^2
50 ppm	30	3.106705	0.0228	0.9726	29.7619	0.039474	0.9997
100 ppm	62	4.470952	0.037539	0.9812	62.1118	0.038688	0.9999
150 ppm	99	4.272676	0.022339	0.9784	99.0099	0.02757	0.9999
200 ppm	140	8.064919	0.034315	0.9129	140.8451	0.021004	0.9999

Table 3. Pseudo first order and pseudo second order of methylene blue (M.B)

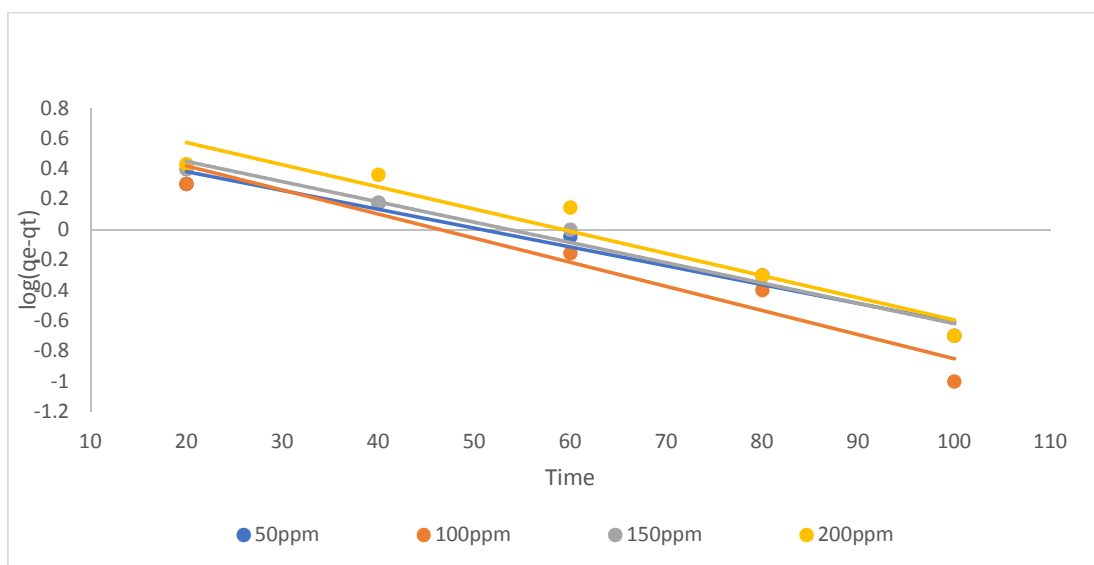| C_o(mg/g)	q_e experimental	Pseudo first order for M.B			Pseudo second order M.B		
		q_e	k_1	r^2	q_e	K_2	r^2
50 ppm	25	4.259906	0.028557	0.9614	24.87562	0.032256	0.9994
100 ppm	54	5.466382	0.036618	0.9404	54.05405	0.031986	0.9999
150 ppm	87	5.201157	0.03086	0.9742	86.95652	0.029389	0.9999
200 ppm	120	7.326558	0.033624	0.9323	120.4819	0.021528	0.9999

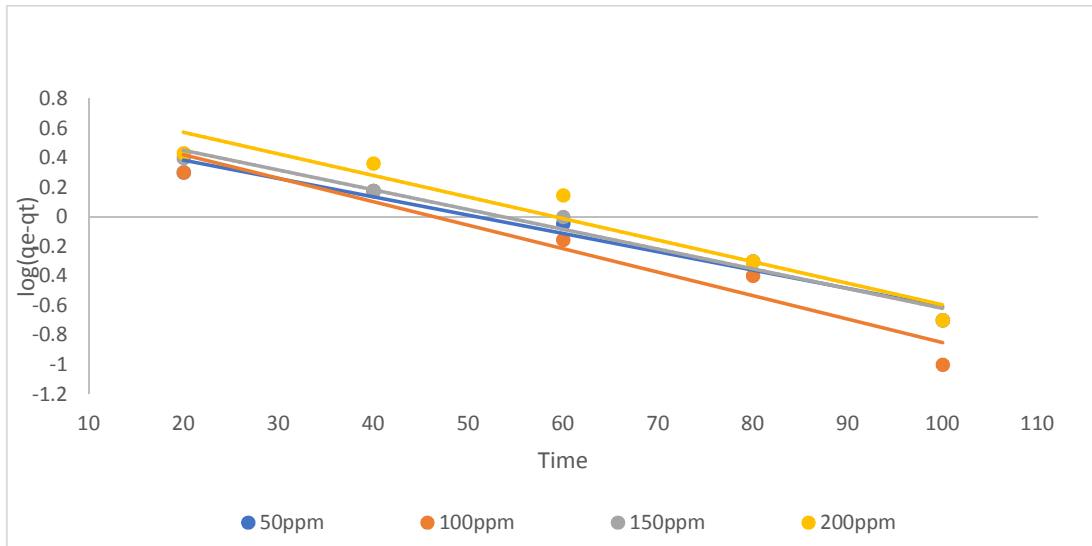**Fig. 10. Pseudo first order of methylene blue onto EDTAD modified cellulose**

Fig. 11. Pseudo first order of neutral red onto EDTAD modified cellulose
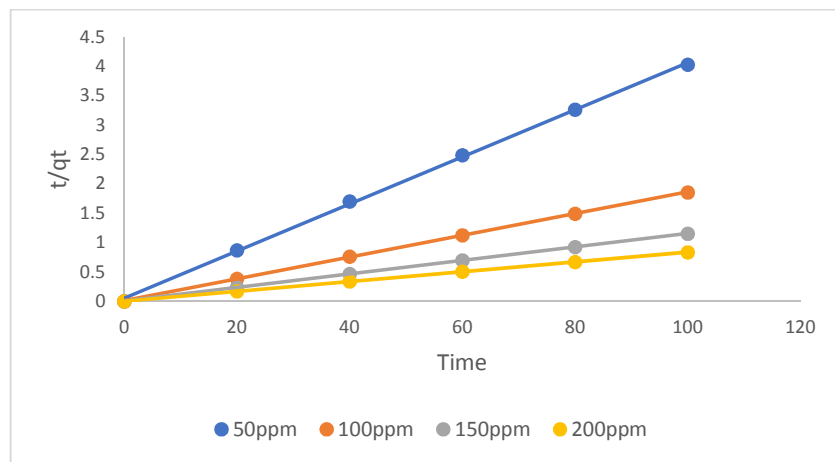

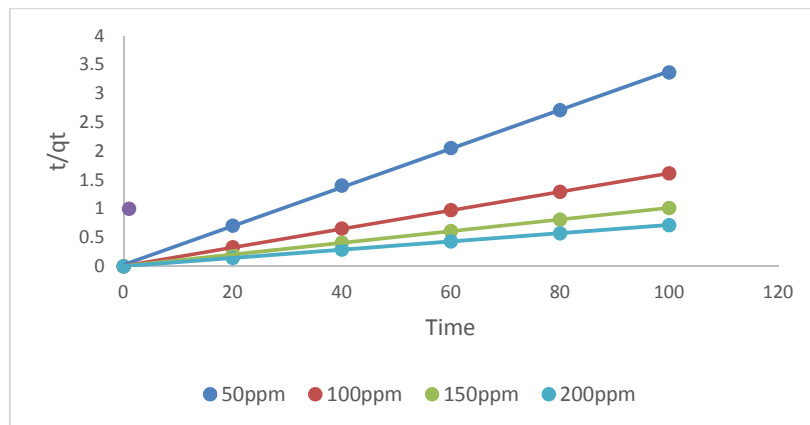**Fig. 12. Pseudo second order of methylene blue onto EDTAD modified cellulose**

Fig. 13. Pseudo second order of neutral red onto EDTAD modified cellulose

3.3 Adsorption Mechanism

There are a number of adsorption mechanisms associated with reactions ranging from mass transfer across boundaries, availability of binding sites and diffusion of adsorbates across a boundary. Electrostatic attraction was considered in explaining the adsorption mechanism of neutral red and methylene blue dyes (20). This correlates the pH of sorption of the dyes to the ionization of the surface functional groups in EDTAD modified cellulose. The functional groups acquire a surface charge when pH is varied. In the acidic pH, there was an introduction of H_3O^+ ions which competes with positively charged methylene blue and neutral red ions for active sites of the adsorbent having carboxylate ions thus low sorption capacity. At alkaline pH, OH^- ions are introduced and this results in a strong electrostatic attraction between the negatively charged active sites of the adsorbent and positively charged sites of the dye ions resulting in a high % removal.

4. CONCLUSION

In this study, cellulose obtained from *Pennisetum purpureum* was modified to obtain EDTAD modified cellulose. The modified cellulose was characterized by FTIR. The EDTAD modified cellulose was used as an adsorbent to determine its efficiency in the removal of methylene blue and neutral red dyes from waste-water. Various adsorption parameters such as dose, pH, contact time, and the initial concentration of the dyes were used to obtain its optimum adsorption capacity.From experimental findings and data analysis, it was found that an increase in adsorbent mass increased the % removal for both dyes with the highest % removal being 96.5% and 92.2% for methylene and Neutral red respectively and the lowest being 36.9% and 39.6% for methylene blue and neutral red respectively. The dyes seemed to favor the Langmuir isotherms as compared to , Freundlich isotherms, with high R^2 values of 0.999 and 0.997 for methylene blue and neutral red respectively while that of Langmuir isotherms had R^2 values of 0.4757 and 0.8125 for methylene and neutral red respectively indicating that the adsorption surface of the EDTAD modified cellulose was monolayer. Finally, it can be concluded that the EDTAD modified cellulose obtained from *Pennisetum purpureum* can be a good candidate for the removal of dyes from wastewaters.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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