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Kinetic and Thermodynamic Studies of Crystal Violet Biosorption from Aqueous Solution using *Spathodea campanulata* leaves

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Abstract : The aim of the present work is to remove Crystal violet (CV) dye from their aqueous solution using *Spathodea campanulata* leaves powder as low cost biosorbent in a batch study. The effect of parameters in a batch study were contact time, solution pH, initial CV dye concentration, biosorbent dosage, average particle size of the biosorbent and temperature. The kinetic and isotherm studies of biosorption of CV dye onto *Spathodea campanulata* biosorbent was investigated. The maximum biosorption capacities of 12.65 mg/g of Crystal violet dye onto the *Spathodea campanulata* biosorbent fitted well with the Langmuir isotherm model. Thermodynamic parameters such as Gibbs free energy, enthalpy change and entropy change were also estimated for the biosorption of CV dye. The thermodynamic studies indicated that the biosorption of Crystal violet dye onto *Spathodea campanulata* biosorbent was spontaneous, feasible and endothermic.

Keywords : Crystal violet, *Spathodea campanulata*, pollution control, aqueous solution, Biosorption.

1. Introduction

Water is an irreplaceable natural resource on this earth on which all life depends as a valuable natural resource, it comprises marine, estuarine, fresh water (river and lake) and ground water environments, across coastal and inland areas. Even though there is a huge water resource in this world, about 97% of water is salt water (marine) and only 3% is fresh water, and in this small fraction of fresh water most of it is locked up in polar ice caps and just 0.003% is readily available to us in the form of ground water and surface water. Water pollutants are of different types such as oxygen demanding wastes, diseases causing agents, synthetic organic compounds, plant nutrients, inorganic chemicals and minerals, oils, thermal discharge and radioactive wastes. Of all these water pollutants, heavy metals and synthetic organic compounds cause majority of water pollution. Industries like paper and pulp, tanneries, textiles, and coke ovens, pharmaceutical, food processing, metal packing, dye-stuff and fertilizer discharge these pollutants into natural water bodies. Heavy metals and

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synthetic organic compounds are non-biodegradable and they can be accumulated living tissues causing various diseases and cause a great damage to human habitation[1].Dyes are also used in industries such as rubber, paper and pulp dye and dye intermediate industries, pharmaceutical, tannery, food Technology, hair colouring, plastic, cosmetic, etc. There are more than 10,000 commercially available dyes with over 7 lakh times of dyestuff being produced annually across the world [2].The textile industry consumes more than 107 kg of die per year of which 90% ending upon fabrics[3].Of this total usage 10 to 15% of the die is lost during the dying process and released with effluent. Colour is contributed by phenolic compounds such as tannins, lignans (two to three percent) and organic colorants (three to four percent) and with a maximum contributions from dye and dye intermediates which could be sulphur or mordant / reactive / cationic / disperse / acid / azo vat die [4].Crystal violet or gentian violet (also known as methyl violet 10B or hexamethylpararosaniline chloride) is a triarylmethane dye used as a histological stain and in Gram's method of classifying bacteria. Crystal violet has antibacterial, antifungal, and anthelmintic properties and was formerly important as a topical antiseptic. The medical use of the dye has been largely superseded by more modern drugs, although it is still listed by the World Health Organization. Crystal violet when dissolved in water has a blue-violet colour with an absorbance maximum at 590 nm. Biosorption technique was most favourable procedure among all the physico-chemical and adsorption, flocculation combined with flotation, membrane filtration, electrokinetic coagulation, ozonation, oxidation, precipitation and Ion exchange methods [5, 6].

2. Materials and Methods:

2.1.Preparation of dye solution:

Stock solutions of Crystal violet concentration 1000 mg/L was prepared by dissolving 1 g of 100% Crystal violet in 1000 ml of distilled water. The solution was prepared using standard flasks. The range of concentration of the prepared dye solutions varied between 20 and 200 mg/l was prepared using the stock solution of individual dye.

2.2.Preparation of biosorbent:

The green colored *Spathodea campanulata* leaves used in the present study were collected from the college of Engineering, Andhra University, Visakhapatnam, India. The collected leaves were washed with deionised water several times to remove dirt particles. The washing process was continued till the wash water contains no dirt. The washed leaves were then completely dried in sunlight for 20 days. The dried leaves were then cut into small pieces and powdered using domestic mixie. In the present study the powdered materials in the range of 53–152 μm particle size were directly used as biosorbents without any pretreatment.

2.3.Chemicals:

The other chemicals used in the present study were NaOH and HCl. The pH of solutions is adjusted with 0.1 N HCl and 0.1 N NaOH. The entire chemicals used were analytical reagent (AR).

2.4.Batch mode biosorption studies:

Batch biosorption equilibrium experiments were conducted in 250 ml conical flasks at a constant agitation speed (180 rpm). All the experiments were carried out at room temperature ($\pm 30^{\circ}\text{C}$). The concentrations of both the dyes before and after sorption were determined using UV- Vis spectrophotometer by monitoring the absorbance for the dye used.

3. Results and Discussion:

3.1.Effect of contact time:

The percentage of biosorption was determined at different contact times and the results are shown in Fig1. The figure reveal that the percentage of biosorption Crystal violet was steeply increased with an increase in contact time from 5 to 30 min and there after reached plateau after attaining equilibrium 30 min for Crystal violet. Therefore, contact time of 30 min is sufficient for the removal of Crystal violet under the experimental conditions used in this study. The percentage of biosorption of *Spathodea campanulata* leaves as biosorbent for

Crystal violet dye removal was increased from 12 to 66%, with an increase in contact time from 5 to 30 min for an initial dye concentration of 20 mg/L. For 200 mg/L, the percentage of biosorption increased from 7.5 to 59 %, respectively with an increase in contact time from 5 to 30 min.[7-13]

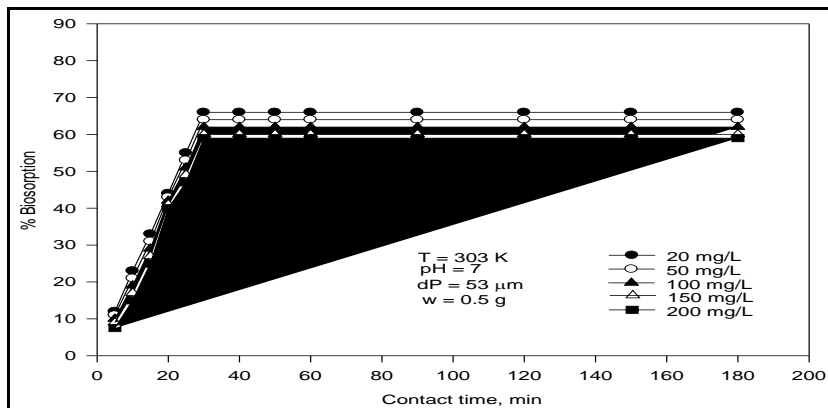


Fig.1. Effect of contact time on % biosorption of Crystal violet using *Spathodea campanulata* leaves as biosorbent.

3.2.Effect of solution pH:

The solution pH is one of the important controlling parameters of the biosorption process. The effect of solution pH on % biosorption for the removal of Crystal violet dye was studied From the Fig.2. It was observed that the increase in solution pH from 2 to 6 for Crystal violet resulted in an increase in percentage of biosorption from 39 to 75% for Crystal violet for an initial concentration of 20 mg/L. For an initial concentration of 200 mg/L, the percentage of biosorption was increased from 33 to 68 % for Crystal violet with an increase in solution pH from 2 to 6[14-20].

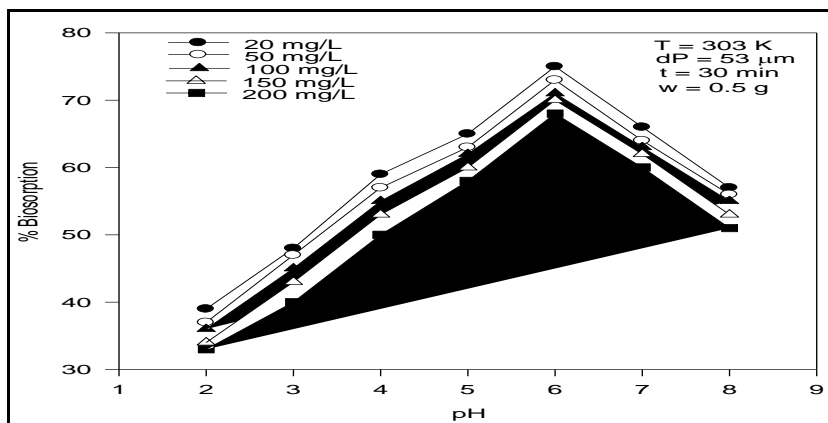


Fig.2. Effect of solution pH on % biosorption of Crystal violet using *Spathodea campanulata* leaves as biosorbent.

3.3.Effect of initial concentration of dye:

The effect of initial dye concentration on the percentage of biosorption is shown in Fig.3. It is evident from the figure that the percentage of biosorption decreased with an increase in initial concentration of dye from 20 to 200 mg/L at all temperatures. The percentage of biosorption of Crystal violet decreased from 71 to 42 % and 76 to 47 for Crystal violet with an increase in initial concentration of Crystal violet from 20 to 200 mg/L at the temperature of 283 K and 323 K, respectively [21, 22].

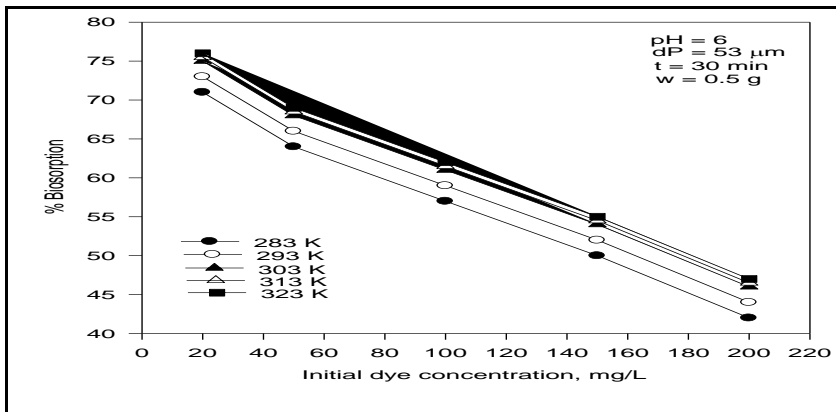


Fig.3.Effect of initial concentration of dye on % biosorption of Crystal violet using *Spathodea campanulata* leaves as biosorbent.

3.4.Effect of biosorbent dosage:

The result obtained is shown in Fig.4, illustrate that the percentage of biosorption was increased with an increase in biosorbent dosage. The percentage of biosorption increased from 75 to 86 % from biosorbent dosage 0.5 to 4 g for an initial concentration of Crystal violet 20 mg/L. For an initial concentration of Crystal violet 200 mg/L, the percentage of biosorption increased from 67 to 79% from biosorbent dosage 0.5 to 4g [23-29].

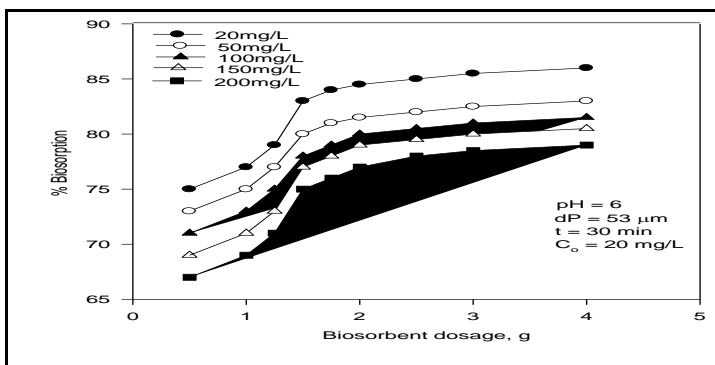


Fig.4.Effect of biosorbent dosage on % biosorption of Crystal violet using *Spathodea campanulata* leaves as biosorbent.

3.5.Effect of particle size of biosorbent:

The result obtained is shown in Fig.5. The result indicated that the percentage of biosorption was decreased with an increase in average particle size of biosorbent. The percentage of biosorption decreased from 83 to 60 % and 75 to 52% with an increase in average particle size of biosorbent from 53 to 152 μ m for an initial concentration of Crystal violet 20 mg/L and 200 mg/L, respectively[30-36].

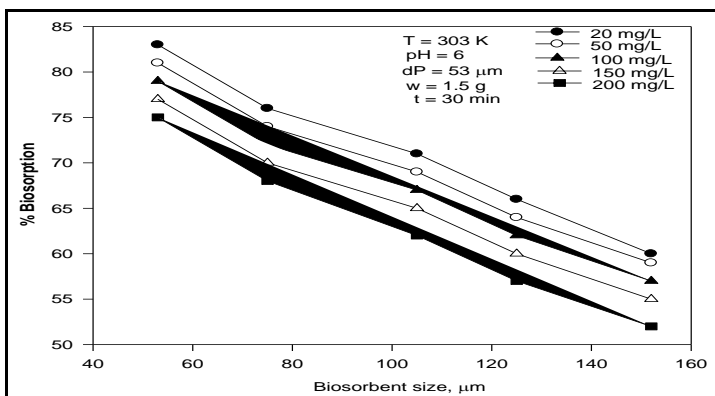


Fig.5. Effect of average particle size on % biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

3.6. Effect of Temperature:

The result obtained is shown in Fig.6, The figure indicated that the percentage of biosorption was increased with an increase in temperature of the solution. This suggests the endothermic nature of the biosorption process. The percentage of biosorption increased from 80 to 85% and 72 to 77% with an increase in solution temperature from 283 K to 323 K for an initial concentration of Crystal violet 20 mg/L and 200 mg/L, respectively [37-43].

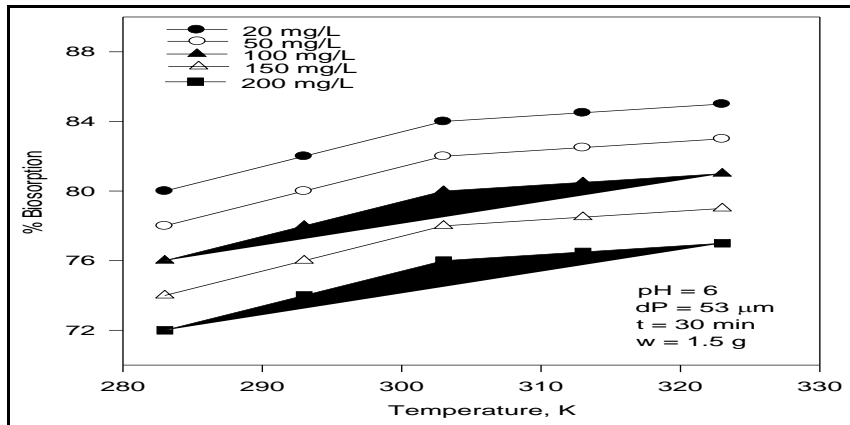


Fig.6. Effect of temperature on % biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

4. Equilibrium studies:

4.1. Langmuir adsorption isotherm:

The applicability of Langmuir adsorption isotherm model was analyzed using the experimental data by plotting C_e/q_e versus C_e . Fig.7. show the Langmuir plot for the biosorption of Crystal violet dye at a temperature 303 K and The separation factor (R_L) values at different initial dye concentrations for the dye was determined and shown in Fig.8. Langmuir constants and maximum biosorption capacity are compiled in Table 1. The high correlation coefficient indicates that the biosorption of dyes onto *Spathodea campanulata* leaves biosorbent followed the Langmuir isotherm. The maximum biosorption capacity (q_m) values were found to be 12.6566 mg/g for Crystal violet.

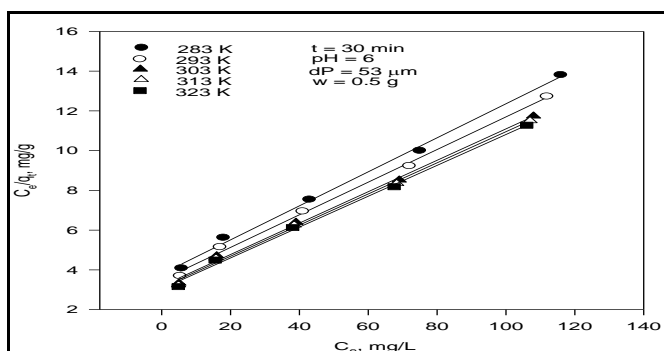


Fig.7. Langmuir Isotherm for biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

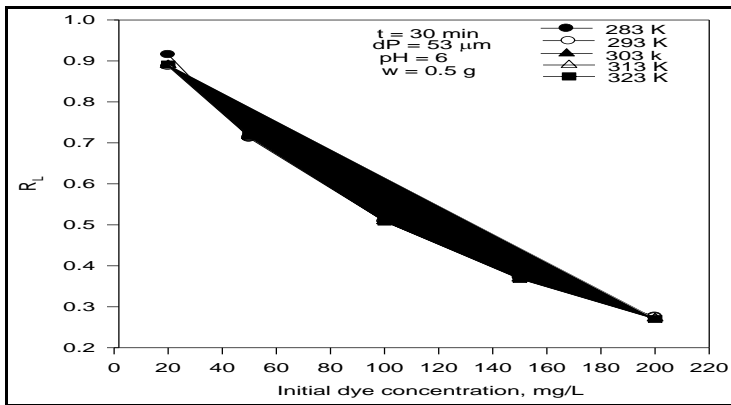


Fig.8. Separation factor for biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

4.2.Freundlich isotherm:

The experimental data was tested for the fitness of Freundlich isotherm model by using linear graphical method. The biosorption data was analyzed by plotting $\ln q_e$ versus $\ln C_e$ shown in Fig.9.

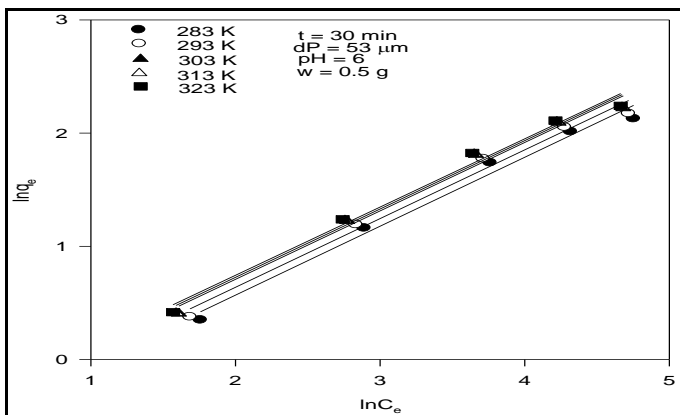


Fig.9. Freundlich Isotherm for biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

4.3.Temkin isotherm:

Temkin isotherm studies were conducted to evaluate the biosorption potentials and to assess the variation of biosorption energies during the biosorption of Crystal violet dye using *Spathodea campanulata* leaves as biosorbent. The biosorption data was analyzed according to the linear form of Temkin model and is shown in Fig.10 for the removal of Crystal violet dye. The linear Temkin isotherm constants B_T and A_T , were determined from the slope and intercept of the plots of q_e versus $\ln C_e$ [44-48].

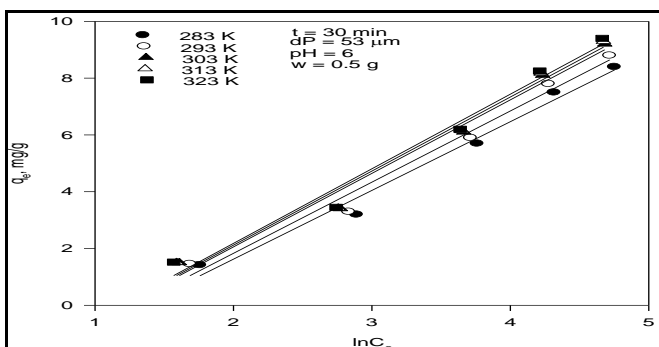


Fig.10. Temkin Isotherm for biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

Table - 1.Biosorption isotherm constants for Crystal violet removal using *Spathodea campanulata* biosorbent.

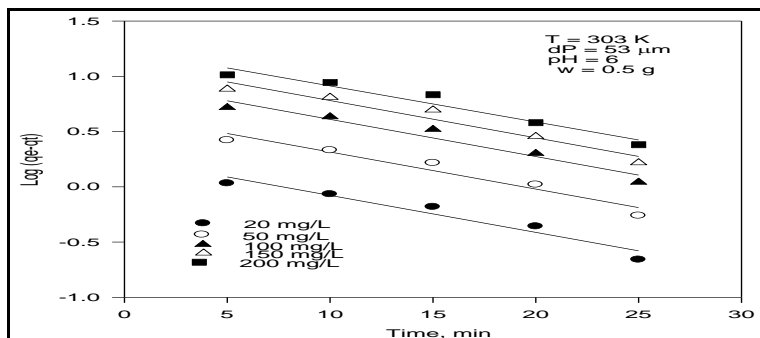
Temperature, T(K)	Langmuir isotherm			Freundlich isotherm			Temkin isotherm		
	q_{max} , (mg/g)	K_L , (L/mg)	R^2	n	K_f , ($\text{mg}^{1-1/n} \text{L}^{1/n} / \text{g}$)	R^2	b_T , (J/mol)	A_T , (L/g)	R^2
283	11.6959	0.0224	0.9968	0.6087	0.5227	0.9848	972.3736	0.2647	0.9823
293	12.1862	0.0235	0.9967	0.6071	0.5616	0.9859	971.6026	0.2805	0.9813
303	12.6566	0.0247	0.9964	0.6044	0.6059	0.9869	973.0164	0.2993	0.9801
313	12.7681	0.0251	0.9963	0.6036	0.6180	0.9872	997.6163	0.3045	0.9798
323	12.8799	0.0254	0.9961	0.6023	0.6305	0.9874	1022.006	0.31007	0.9795

High correlation coefficient, R^2 , values suggest that the biosorption process could be due to heterogeneous surface coverage. This is in good agreement with the result of Langmuir isotherm for Crystal violet dye.

5.Kinetic Studies:

5.1.Pseudo- first-order kinetic model:

The pseudo-first-order kinetic model was developed based on the solid adsorption capacity. The experimental results were analyzed to test the pseudo-first-order kinetic model and the linear plots of $\ln(q_e - q_t)$ versus t is shown in Fig.11.

**Fig.11.** Pseudo first order kinetic model for Crystal violet biosorption onto *Spathodea campanulata* leaves biosorbent.

5.2.Pseudo-second-order kinetic model:

The values of equilibrium biosorption capacity and second order rate constants were determined from the slope and intercept of the linear plot of t/q_t versus t in Fig.12. The values obtained are tabulated along with the correlation coefficient (R^2) values in Table.2 for Crystal violet dye.

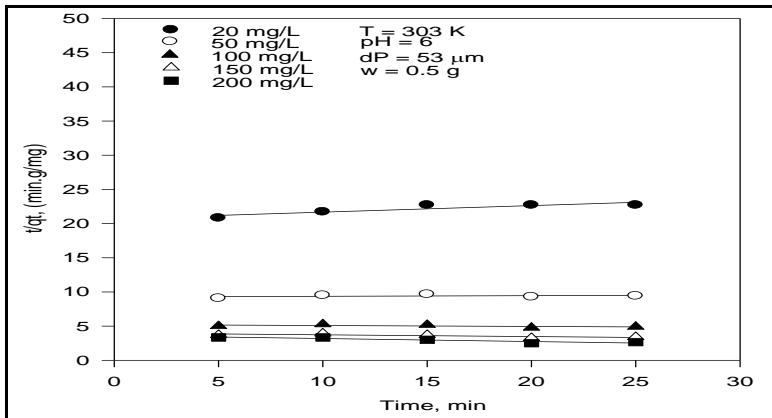


Fig.12. Pseudo second order kinetic model for Crystal violet biosorption onto *Spathodea campanulata* leaves biosorbent.

Table - 2.Kinetic model parameters for Crystal violet removal using *Spathodea campanulata* biosorbent.

Initial Conc, mg/l	Lagergren-first order			Pseudo-second order		
	q_e , (mg/g)	k_1 , (1/min)	R_1^2	q_e , (mg/g)	k_2 (g/mg.min)	R_2^2
20	1.2382	0.0563	0.9590	5.26	0.00155	0.7599
50	1.2499	0.0633	0.9385	4.2789	0.09153	0.9948
100	1.2606	0.066	0.9219	9.2165	0.1086	0.9990
150	1.2735	0.069	0.9022	14.1844	0.1124	0.9995
200	1.2893	0.0727	0.8795	19.15	0.1135	0.9997

6.Thermodynamic Studies:

Thermodynamic studies provide information about the feasibility of the process and nature of biosorption process. In order to estimate the thermodynamic parameters for the biosorption of Crystal violet dye using *Spathodea campanulata* leaves as biosorbent, the experiments were conducted and data were analyzed. The values of ΔH° and ΔS° were calculated from the slope and intercept of the linear Van't Hoff plot i.e.ln (q_e/c_e)vs(1/T). These plots are shown in Fig.13 for Crystal violet dye. The estimated thermodynamics properties along with the correlation coefficients (R^2) are tabulated in Table-3.

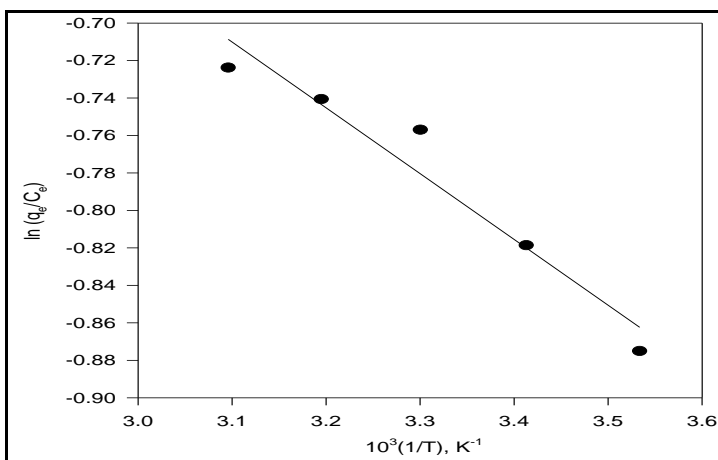


Fig.13. Van't Hoff relation for the determination of thermodynamic parameters for the biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

Table.3: Thermodynamic parameters for the biosorption of Crystal violet onto *Spathodea campanulata* leaves biosorbent.

Temperature, K	Crystal violet		
	ΔG^0 (kJ/mol)	ΔH^0 (J/mol)	ΔS^0 (J/mol)
283	-4.7111	15.4862	16.7020
293	-4.8781		
303	-5.0452		
313	-5.2122		
323	-5.3792		

7. Conclusion:

The data obtained from the biosorption studies showed that a contact time of 30 min was sufficient for the maximum removal of Crystal violet dye from aqueous solution using *Spathodea campanulata* biosorbent. The experimental data of biosorption of the dye onto the *Spathodea campanulata* biosorbent fitted well with the Langmuir isotherm model. The isotherm reveals that the biosorption of the dye onto *Spathodea campanulata* biosorbent was favourable. The maximum removal efficiency was predicted to be 89% for Crystal violet, which was obtained at a temperature of 303.2868 K, solution pH of 6.0684, initial dye concentration of 20.0908 mg/L and biosorbent dosage of 1.5039 g.

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