

A Promising Low Cost Adsorbent Based on Tea-Waste for Confiscation of Crystal Violet Dye

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The goal of the current study is to eliminate Crystal Violet dye from aqueous solutions because it is one of the refractory dye molecules that can linger for a long period in the environment and cause hazardous effects. To potentially adsorb Crystal Violet (CV), a cationic dye from aqueous solution, tea-powder waste (TP) was used. Utilizing FTIR, SEM, EDX, XRD, and TGA analysis, the pore shape, surface characteristics, thermal stability and crystalline character of TP were evaluated. Several operating parameters, including contact time, TP dosage, and original CV concentration, point of zero charge (pH_{pzc}), solution pH and effect of temperature were examined in the adsorption of CV onto TP. Under ideal conditions of $pH 5.0 \pm 0.2$, 30 min of adsorption time and 100 mg of adsorbent dose, dye concentration 100 mg/L, TP was found to have highest possible adsorption capacity of 200.64 mg g^{-1} . From kinetics of adsorption analysis it was found to follow a kinetic model of pseudo-second order. The results for CV adsorption onto TP were found to be accurately characterised by the Langmuir isotherm. Increase in the temperature favoured the adsorption of CV on TP and therefore adsorption process was proven to be spontaneous and exothermic. Studies on fixed bed columns were also done. Column analysis demonstrated the TP's applicability to the greater sample volumes. For the elimination of crystal violet from aqueous solution, TP showed great promise as an adsorbent.

Keywords: Adsorption, Biosorption, Crystal violet, Isotherm studies, Tea-powder waste

1 Introduction

The most difficult hazard to humans is now environmental contamination as a result of fast industrialisation and population increase. The first pollutant to be identified in wastewater is colour. It is not acceptable for dyes to be present in water in even small concentrations (less than 1 ppm for some dyes). These hazardous contaminants have been discharged into water systems due to the steadily growing use of organic colours in a variety of industries, including textile, paper, plastics, rubber, and leather treatment.¹ Due to complex aromatic molecular structures of dyes, they are more environmentally stable. Because dyes prevent light from penetrating water, they have an adverse effect on aquatic life. The complex structure of dyes makes its biological degradation a challenging process. Approximately ten to fifteen percent of all colour are wasted to sewage water directly during the dyeing process.² Reverse osmosis, coagulation, biological techniques, oxidation procedures, electrochemical techniques and other approaches are utilised to treat dye-contaminated effluents. Unfortunately, majority of them have

substantial running costs when expanded to effluent treatment plant (ETP) scale.³ Adsorption was shown to be the most successful, efficient, and environmentally friendly way when researchers compared the methods for dye removal. Most of the parameters of the liquid substance, such as absorption, solubility, polarity, particle size, as well as the solid-state properties of surface area, density, pore size, and moisture, affect the adsorption process. Also through a few factors including temperature, medium pH, and solvent type. An adsorbent consists of biomass is referred to as a biosorbent. Biological adsorption is a simple and highly effective method of eliminating pollutants from the water. It generates less waste and uses fewer chemicals, which lowers the risk of chemical-induced environmental pollution.⁴ The waste from agriculture, such as rice husk, ginger waste, soy oil extract, lemon peelings, tamarind shells, maize, cane sawdust, papaya fruit seeds seashells powder and pepper seed spent are low cost bioadsorbents.⁵ India's most popular beverage is tea, hence every home uses tea powder. Current research demonstrates that the use of microbial biomasses and waste organic materials is gaining popularity as efficient, inexpensive, and environmentally

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responsible sorbents for the removal of organic dyes from bodies of water. Metal content, carbon, nitrogen, carbohydrates, phosphorus, and other chemical components of algal biomass and waste from the production of tea powder are practically same. These two factors may be utilized to detoxify dye-effluents using the vast amounts of tea waste that Indian kitchens create.⁶⁻⁷

The primary structural components, chromophores, and dyeing methods can all be used to classify dyes into different groups. CV is a triarylmethane class of dye. There are many uses for CV in the medical industry. It is applied for classifying microorganisms using Gram's staining. The textile business, where it is used as a purple dye, as well as the paint, paper dyeing and printing ink industries, are where this dye finds its greatest use. Even eye and skin illnesses of animals are treated with the dye.⁸ But being non-biodegradable it must be removed from wastewater. It persists in the longer periods of time and may contain harmful effects on the human body that are carcinogenic and mutagenic, including kidney failure, severe eye irritation, skin disorders, skin cancer, and skin problems with respiratory issues.⁹

This investigation focused at tea-powder waste's capacity to adsorb crystal violet from aqueous solutions.

2 Materials and methods

2.1 Materials

We bought tea dust from a resident supplier (India-specific Brook Bond Red Label product code is F12B). Hydrochloric acid (HCl), Crystal violet dye (95% dye concentration), and sodium hydroxide (NaOH) (98% analytical grade) were purchased from Loba Chemie in India.

2.2 Adsorbent preparation method

A simple method was followed to obtain tea waste. Tea dust was continually removed with hot water until it gave up trying to develop colour, then the extract was removed and dried for 24 hours at 50°C in oven. A normal sieve with a diameter of 500 µm was used to filter the dried material. In order to be used later, the adsorbent material was preserved in a sealed container.

3 Results and Discussion

3.1 Characterization techniques and instruments

Various instruments were used to characterization of TP include Bruker Alpha for FTIR, Rigaku-

Miniflex 300 for XRD, model Carl Zeiss EVO 18 for surface morphology of adsorbents. Energy dispersive spectrum (EDX) analysis was used to do the elemental analysis utilising an X-ray analyzer (Ametek) and a scanning electron microscope (Zeiss EVO 18). For measuring CV in the solution, an EQ-824 spectrophotometer with matching cuvettes was utilized. Thermal stability experiments based on thermogravimetry and differential thermal analysis were conducted using the Shimadzu DTG-60.

3.2 Batch adsorption experiments

In order to conduct batch tests, a stoppered conical flask was used to equilibrate 25 mL of CV dye solution at various concentrations (25-400 mg L⁻¹) and with a known quantity of adsorbent varied from 10 to 150mg. At room temperature, systems were stirred for the required amount of time duration. Using diluted HCl or NaOH solutions with a pH range of 4.0 to 9.0, the solution's pH was adjusted. These flasks were continuously stirred using a mechanical shaking machine at 180 rpm. After filtering the solutions, the absorbance value of the filtrate at 590 nm was recorded with the help of spectrophotometer. Amount of adsorbate adsorbed at equilibrium is given by

$$q_e = \frac{C_0 - C_e}{W} \times V$$

where C₀ and C_e respectively represent the CV's initial and equilibrium concentrations.

3.3 Characterization of TP

Figure 1(a) depicts the FT-IR spectra of TP, which exhibits distinctive peaks corresponding to O-H and C-H stretching vibrations in the ranges of 3,650 cm⁻¹ and 2,860 cm⁻¹, respectively. The peak at 1,730 cm⁻¹ that can be attributed to the carbonyl stretch of the -COO group. A dip at 1,610 cm⁻¹ indicates the C-O stretching mode coupled with NH₂. -OH group with a potent C-O-C stretching vibration is the 1,095 cm⁻¹ band. The results are consistent with those that have been reported in the literature.¹⁰

Crystallinity is expected to be present in the XRD pattern. The XRD pattern of TP and TP-adsorbed-CV is depicted in Fig. 1(b). It is clear from the two XRD results that crystalline areas exist. After CV was adsorbed, there was no obvious change in the XRD pattern. However, following dye adsorption, the majority of the peaks' reduced peak heights are a sign of a reduction in crystallinity.¹¹

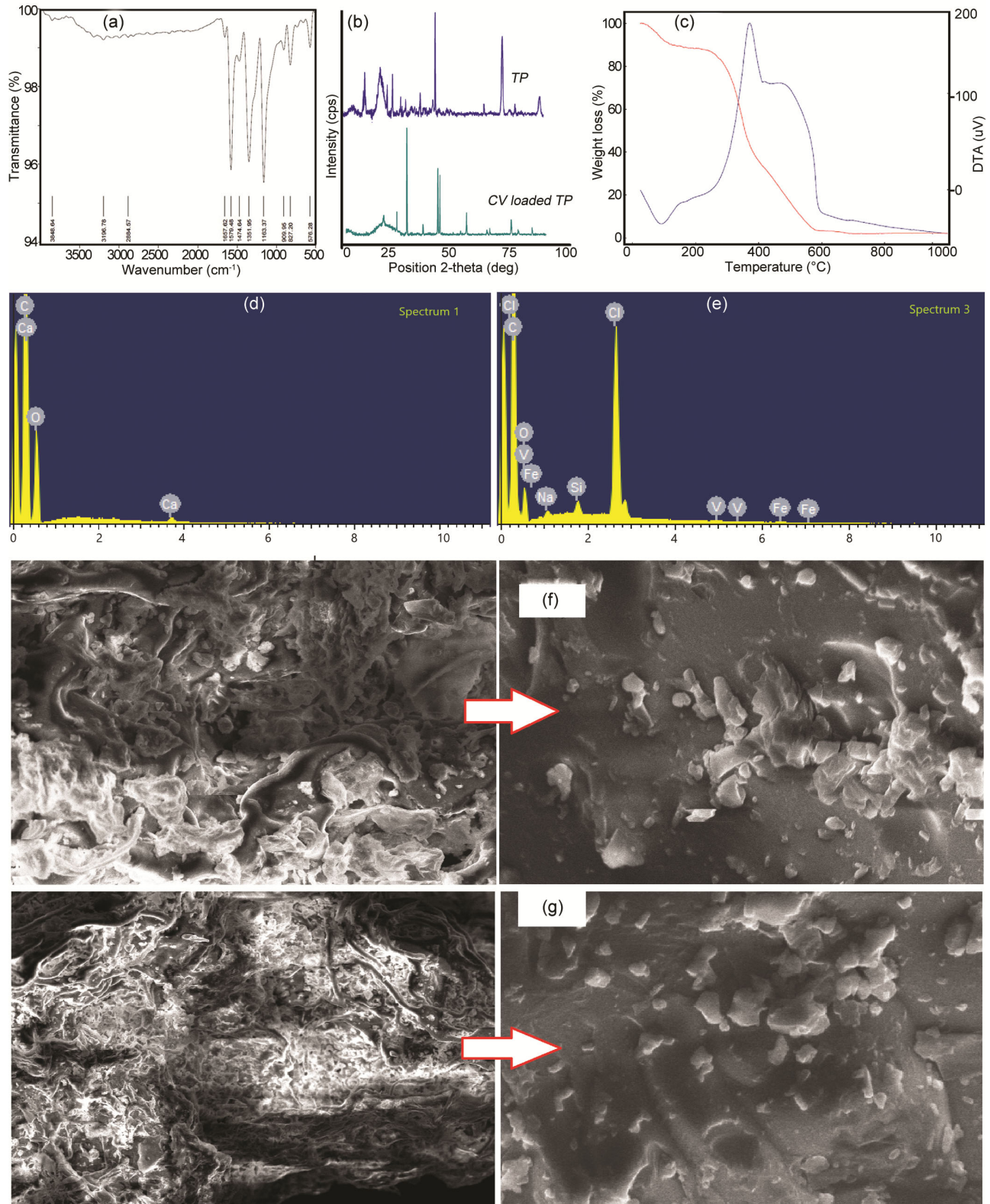


Fig. 1 — (a) FTIR analysis of TP, (b) X-ray diffractograms of TP and CV loaded TP, (c) TGA - DTA analysis of TP, (d) EDX spectra of TP, (e) EDX spectrum of CV loaded TP, (f) SEM micrographs of TP at two distinct resolutions, & (g) SEM pictures of CV loaded TP at two separate resolutions.

The thermal stability and decomposition temperature of the produced adsorbent was investigated using thermal examinations of sample materials. The TGA curve for TP is displayed in Fig. 1(c). Three stages of weight reduction are shown in the sample. In the first initial stage, 10% of the mass was lost due to moisture and retained water evaporation near 100 °C. Endotherm on the DTA curve coincided with this. A second stage degradation was seen between temperature 200 and 400 °C, when the cellulose and hemicellulose parts started to disintegrate. A third weight reduction started happening right quickly after the lignin broke down, which led the sample to totally degrade up to 600 °C. The DTA curve exhibits the exothermic nature of each of this breakdowns.¹²

The EDS curve of unloaded TP (Fig. 1d) revealed a significant concentration of calcium and carbon, similar to activated carbon. CV-loaded TP Fig. 1(e) shows distinct peak of chlorine which is present as a counter ion in CV dye. This is a clear indication that the entire CV molecule comprising of both cation as well as anion of the molecule get adsorbed on the TP surface.

Figure 1(f) of the SEM image of native adsorbent reveals some surface unevenness, structure density with noticeable porosity. SEM picture of TP sample following dye adsorption is shown in Fig. 1(g). It is revealed that upon dye adsorption, a systematic arrangement forms, with a smoothness and rigidity in the structure that are sustained throughout. The surface porosity of the TP was decreased when the adsorbent being uniformly covered with CV. This surface property will support the greater adsorption capabilities.

BET surface area analysis studies have suggested that the TP has a moderate surface area of 1.721 m²/g with total pore volume of 7.25×10⁻³ cc/g. The nitrogen adsorption-desorption curve and pore size distribution has been shown in Fig. 2.

3.4 Batch adsorption Studies

The state in which the electrical charge density on a surface is zero is known as the point of zero charge (pzc). A graph showing the pH change in relation to the starting pH was drawn. The x-intercept is the value of pH_{pzc} and it was discovered to be 6.2 Fig. 3(a). The findings demonstrate that at pH 6.2, an adsorbent immersed in an electrolyte displays no net electrical charge. This shows that the protonation of -OH groups to -OH²⁺ groups below pH 6.2 causes the

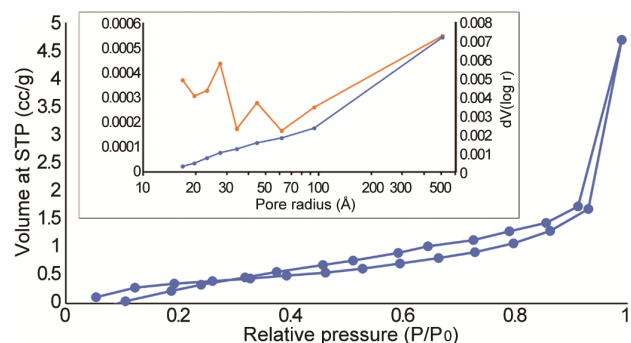


Fig. 2 — Nitrogen adsorption-desorption curve and pore size distribution.

adsorbent to acquire a positive charge, while the ionization of -COOH groups into -COO⁻ groups above pH 6.2 causes the -COOH groups to impart a negative charge. The ultimate pH of the filtrates was measured, and a graph of pH change relative to initial pH was drawn.

The capacity for adsorption is significantly impacted by the pH of the solution during the adsorption process. Throughout the range of pH (3-9) at 100 mg L⁻¹ initial CV concentration, the pH effect was examined. 100 mg of the adsorbent was used, and 60 minutes of stirring were other conditions maintained. The findings show that the dye elimination did not significantly alter across the entire pH range. For the whole pH range, the removal percentage for 100 ppm was between 94% and 96%. So, for the next investigation, the dye solution's original pH, which was about 5.20, was employed Fig. 3(b).

Since adsorption occurs on surfaces, its degree is directly correlated with the surface area that is accessible. Adsorbents of varied doses were applied to the adsorbate-adsorbent systems that had pH and contact time optimizations. Adsorption percentage increased with an increase in adsorbent quantity. This is possible due to the abundance of active sites for adsorption are available compared to the amount of dye molecules that can be adsorbed. Yet, since over 95% of the dye has been adsorbed, there appears to be little improvement in the removal efficiency after a particular dose. Equilibrium adsorption for TP was obtained at a dose of 100 mg Fig. 3(c), hence it was optimised for the next tests.

In order to optimize the contact duration for the experiment, it was changed from 0 to 120 minutes while maintaining all other factors constant. According to the results, % adsorption rose along with contact duration, but after a while, it progressively

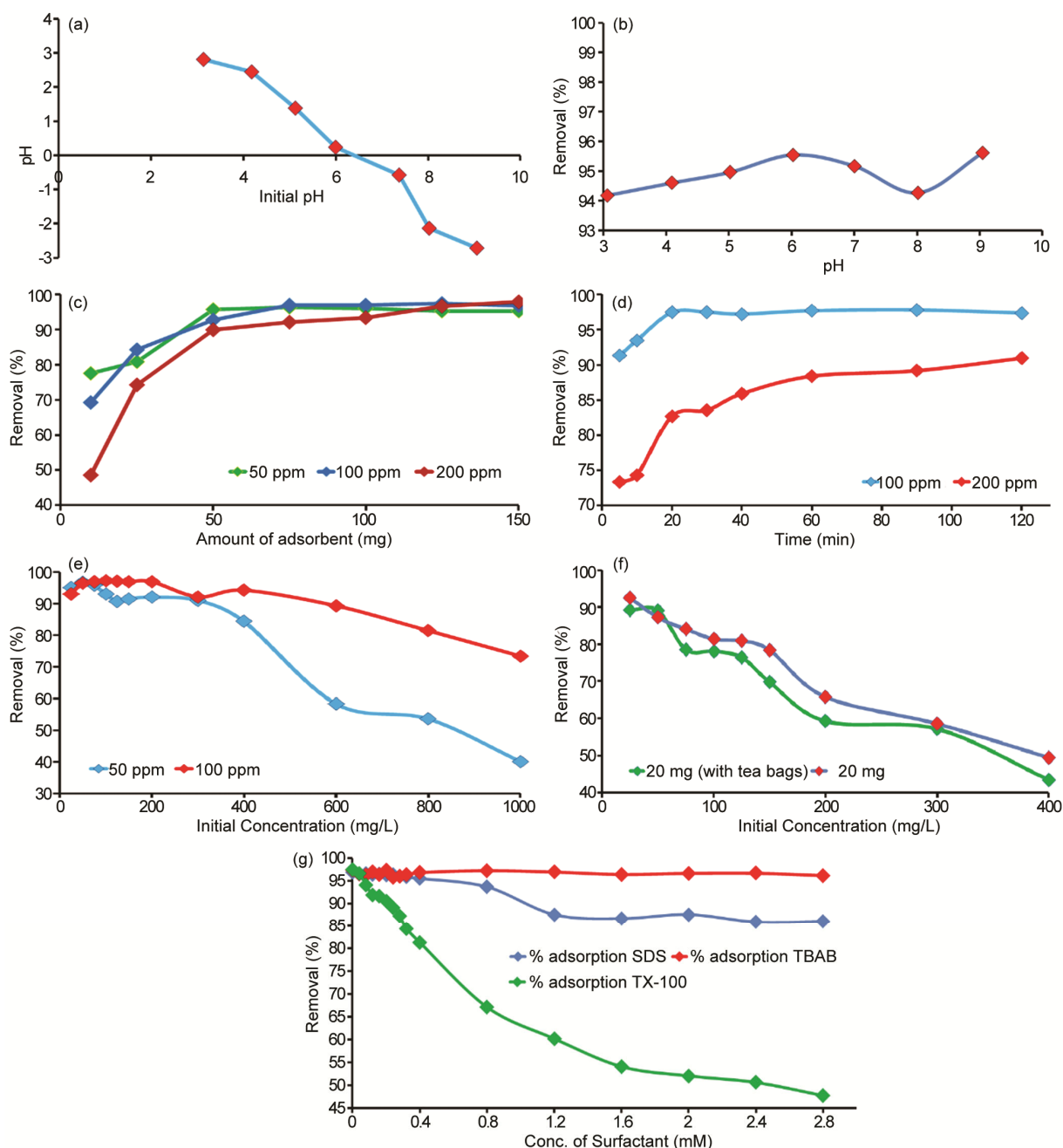


Fig. 3 — Effect of (a) pH_{PZC} , (b) pH, (c) TP dose, (d) contact time, (e) initial concentration of CV, & (f) surfactants on percentage CV elimination.

rose towards stable level, showing the achievement of equilibrium. From Fig. 3(d) it was observed that, equilibrium was reached after 30 minutes. Due to ion desorption on the accessible adsorption sites of the sorbent, a further increase in contact duration had no effect on adsorption. 30 minutes of contact time was therefore ideal for subsequent research.

The initial CV concentration parameter was examined for adsorbent doses of 50 mg and 100 mg

over a 30-minute contact period, with a range of 25–1000 $mg\ L^{-1}$ (Fig. 3e). The experiments were conducted at room temperature. Up to 200 $mg\ L^{-1}$ of high percentage adsorption was first noted, and as dye concentration increased, there was a corresponding decline in percent clearance. Being more active sites on the adsorbent's surface at low initial dye concentration, the majority of the dye molecules have been adsorbed on the surface of TP, resulting in a

higher percentage of adsorption, whereas at high starting CV concentrations, molecules have very little probability of being adsorbed because there are so few binding sites on the surface of the adsorbent. As a result, some dye molecules do not become adsorbed and instead stay in the solution. Since the adsorption effect was largest at a concentration of 100 mg L⁻¹, it was optimized for all studies.

For the first time, deflouridation of water utilizing tea-bag model has been proposed by Prabhu *et al*¹³. For this system also, the tea bag model was used. The benefit of this approach is that after adsorption, filtration is not necessary because the adsorbent material stays in the tea bag. In this model, each tea bag contained a constant weight of 20 mg of adsorbent that was placed in CV solutions of various concentrations. The bags were removed after whirling the liquids for 30 minutes. A comparison of the batch adsorption with tea bag and without tea bag has been depicted in Fig. 3(f). It was clearly observed that, the trend of change in adsorption efficiency with respect to CV concentration was identical when used in tea bag. However, the adsorption efficiency reduced a bit in case of tea-bag experiments. This observation is in accordance with that reported in the literature and can be attributed to incomplete exposure of the entire surface of the material to dye molecules as well as intermolecular repulsion in vicinity of the adsorbent surface¹³.

Based on these observations, it can be concluded that the interaction between TP surface and the CV molecules is mainly electrostatic in nature. At near neutral pH, one of the nitrogen atom of CV has positive charge while the TP surface has -NH₂ and -OH functional groups containing lone pairs of electrons. The electrostatic interaction between these charges leading to adsorption of the dye on TP surface may be the plausible mechanism of adsorption.⁶

Surfactants are also a problem in the majority of dye-containing effluents. They may affect the efficacy of the adsorbent to a great extent. Tetrabutyl ammonium bromide (TBAB), Triton X-100, and sodium dodecyl sulphate (SDS) were chosen as cationic, nonionic, and anionic surfactants, respectively, to examine the impact of surfactant concentration on the effectiveness of CV adsorption. It has been found (Fig. 3g) that as SDS concentration rises, adsorption percentage reduces. The decrease in CV adsorption on tea powder may be the result of SDS being more readily absorbed than CV. In case of

Table 1 — Isotherm studies for CV

Langmuir		Freundlich			
Q _m	K _L	R ²	K _F	n	R ²
200.64mg/g	8.06L/g	0.998	17.19mg/g	2.23g/L	0.984

TBAB effect, percentage adsorption has no appreciable impact as its concentration rises. This is to be expected as the CV dye and the surfactant are both cationic. They resist one another, and it was discovered that TBAB is not competitive for CV adsorption. When TX-100 concentration rises, adsorption decreases by a percentage. The solubilization of the CV dye molecule into the TX-100 micelles may be the cause of the decrease in CV adsorption on tea powder. Another reason could be complex formation between surfactant and dye molecules.

3.5 Adsorption isotherms

Adsorption isotherms are crucial for understanding how molecules that are being adsorbate and molecules that are being adsorbed interact. To comprehend the mechanism of adsorption, a variety of adsorption isotherm models are available. Two isotherms Langmuir and Freundlich were quantitatively examined in the current work. According to the Langmuir model, adsorption occurs on a homogeneous surface of sorbent, where all surface sites are equally attractive to adsorbate molecules and all positions have identical binding energies.¹⁴ While according to the Freundlich isotherm, the distribution of adsorbent sites is uneven because the adsorbent surface is heterogeneous.¹⁵ Variable CV concentrations of 25 to 1000 mg L⁻¹ were used to conduct the experiments under ideal conditions. Table 1 lists the specifics of the investigated isotherm models as well as the key parameters. The Langmuir model was found to be the best fit model for the dye when the results from the batch adsorption studies were subjected to these isotherm models. The high values of the correlation coefficient, R², led to this conclusion Fig. 4(a). The R_L values which range from 0 to 1, indicate how favourably the adsorption process is working. 200.64 mg g⁻¹ was observed to be the maximal monolayer adsorption capacity (q_{max}). Also, n > 1 values obtained using Freundlich isotherm implies the dye molecules and TP have potent interaction.

3.6 Kinetics of adsorption

Kinetics of adsorption was investigated by Intraparticle diffusion (IPD), pseudo first-order

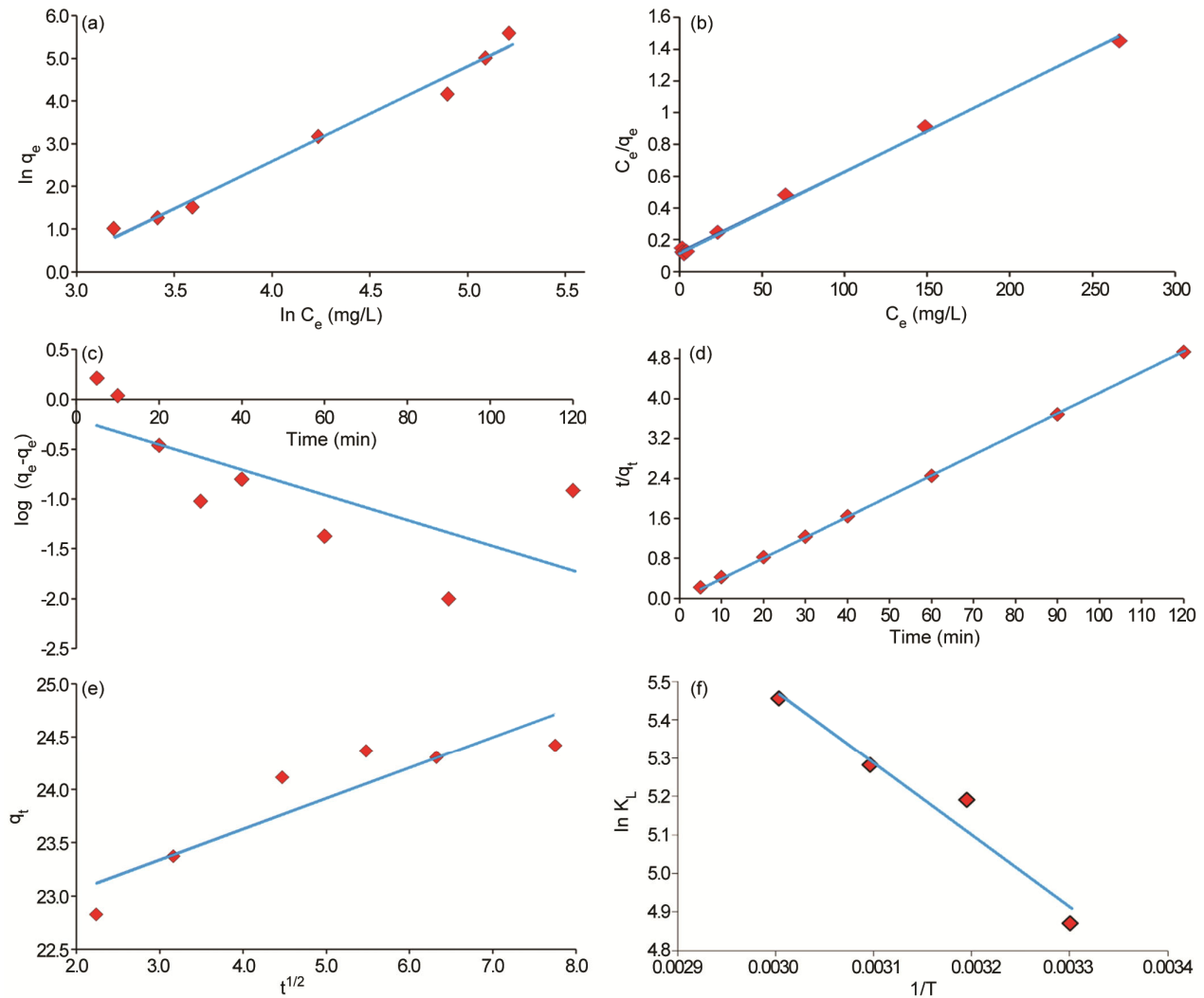


Fig. 4 — Adsorption isotherm models and kinetic models of CV dye on TP (a) Langmuir model, (b) Freundlich model, (c) Pseudo-first-order, (d) Pseudo-second-order, (e) plot for Intra-Particle Diffusion model, & (f) Van't Hoff graph.

(PFO), and pseudo second-order (PSO) models. Adsorption systems were maintained in contact for periods ranging from 5 to 120 minutes to examine kinetics. To determine which model would best match the experimental data, R^2 values were computed. The Table 2 provides thorough information on these three models and their associated variables. After comparing the three kinetic models, it was found that the model of pseudo second-order suited the data the most closely Fig. 4(b) as the value of R^2 , correlation coefficient was calculated to be unity. In Table 2, there is an overview of the specifics of the variables evaluated using kinetic models.

3.7 Thermodynamics of adsorption

To identify the thermodynamic components that determine the spontaneity and viability of the process,

Table 2 — Summary of Kinetic models studied

PSO		PFO		IPD	
$K_1(\text{min}^{-1})$	r^2	$k_2(\text{mg g}^{-1} \text{min}^{-1})$	r^2	$k_{\text{int}}(\text{mg g}^{-1} \text{min}^{1/2})$	r^2
0.023	0.8411	0.153	0.9999	0.29	0.8172

the impact of temperature on the adsorption process of CV was investigated. The thermodynamic equilibrium constant, K , provides simple information about these factors. The process's associated change in free energy is computed as follows:

$$\Delta G = -RT \ln K$$

The value of ΔH from the slope and ΔS value from the intercept of the plot of $\ln K$ against $1/T$ Fig. 4(c) were calculated.

$$\ln k = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$

Table 3 — Adsorption thermodynamics of TP

Temp (K)	ΔG (kJ mol ⁻¹)	ΔH (kJ mol ⁻¹)	ΔS (J K ⁻¹ mol ⁻¹)
308	-44.0005	-15.5783	92.28
313	-44.4619		
323	-45.3847		
333	-46.3075		

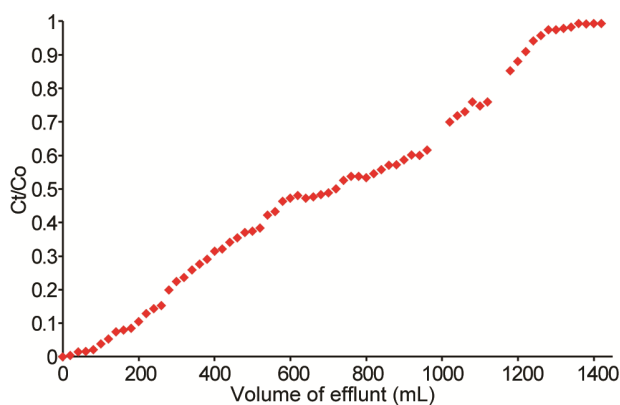


Fig. 5 — Column Studies.

where T is the absolute temperature (K) and R is the gas constant (8.314 J mol⁻¹ K⁻¹). To find the equilibrium constant K_L , it was necessary to compare CV on the adsorbent to that in the solution phase. The findings indicate that CV adsorption is an occurrence that occurs naturally because of the negative values of ΔG . The process is exothermic, as shown by the negative values of ΔH and the progression of dye adsorption on the adsorbent's surface is indicated by the positive values of ΔS , which suggest that the randomness in the mobile dye molecules is increasing. Table 3 shows that when the temperature rises from 303 to 333 K, the ΔG values turn increasingly negative, demonstrating that adsorption capacity rises as temperature rises.

3.8 Column parameters

One of the most popular and effective methods for removing contaminants from water is adsorption utilising columns. Due to the constant flow that is maintained across a fixed-bed in columns as opposed to batch, where the gradient of concentration between adsorbent and adsorbate gradually decreases, columns have an advantage over batch in terms of adsorbent capacity. To ascertain whether the adsorbent could be employed with a bigger sample, column investigations were also carried out. Using a 50 mg L⁻¹ CV solution, the column experiments were conducted. 0.5 g of the tea powder was placed inside glass column to column heights of 4 cm. The CV solution of necessary pH was

Table 4 — Column study for CV adsorption

Parameter	Observations
Concentration of CV	50 mg L ⁻¹
Exhaustion vol.	850 mL
Breakthrough vol.	250 mL
Exhaustion Capacity	80 mg g ⁻¹
Breakthrough Capacity	29 mg g ⁻¹
% column utilization	36.25%

Table 5 — Comparison of the capacity of the different adsorbents to adsorb Crystal Violet

S. No.	Material	Adsorption capacity (mg g ⁻¹)	Reference
1	Charred rice husk and xanthated rice husk	62.85 and 90.02	[16]
2	Sodium hydroxide-modified avocado shells	179.80	[17]
3	Chitosan/nanodiopside	104.466	[18]
4	Lysiloma latisiliquum seed powder	14.14	[19]
5	Olive leaves powder	181.1	[20]
6	Marine diatom alga, Skeletonema costatum	6.40	[21]
7	Corn stalk	9.6	[22]
8	Tea waste/Fe ₃ O ₄ magnetic composite	333.33	[23]
9	Used black tea leaves	184.1	[24]
10	Tea dust	175.4	[25]
11	TP	200.64	Present study

percolated through the column. 5 mL min⁻¹ was the optimal flow rate. The eluent solutions were collected in 20 mL aliquots and measured spectrophotometrically until the output concentrations achieved an equilibrium value. By plotting C_t/C_o versus the volume of CV solution eluted the values of different column parameters were calculated Fig. 5 and are listed in the given Table 4.

4 Conclusion

This work demonstrated that inexpensive adsorbent tea powder can be utilised to eliminate crystal violet dye from aqueous solutions over a wide range of concentrations. A simple and eco-friendly method for using TP as a strong, environmentally friendly adsorbent to eliminate CV dye from aqueous medium has been devised. There are no prerequisites or requirements for using tea powder because it is naturally occurring and easily accessible. The use of the column approach allows for the processing of larger sample quantities, as shown by column adsorption characteristics. The adsorption capacity of 200.64 mg g⁻¹ as obtained in accordance with

Langmuir isotherm model can be compared with those of other reported materials. This comparison has been presented in Table 5 which is self-evident and highlights the effectiveness of this waste material for water treatment application.

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