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**Original Reserach Article** 

### Photocatalytic Degradation and Adsorption Crystal Violet Dye Using Capparis Spinosa Plant as Catalysts

Haitham H. Al-Gatea<sup>1\*</sup>, Share A. Ali Alshamkhawy<sup>1</sup>

<sup>1</sup>Department of Chemistry, College of Sciences, University of Thi-Qar, Thi-Qar, 64001, Iraq

\*Corresponding Author: Haitham H. Al-Gatea

Department of Chemistry, College of Sciences, University of Thi-Qar, Thi-Qar, 64001, Iraq

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Abstract: Natural products demonstrate high effectiveness as catalysts which aid dye removal in textile factories alongside water purification efforts in various industrial fields. Scientists conducted tests to examine how caper stem powder executes photocatalysis when treating crystal violet dye. The experiments that used crystal violet dye contaminated aqueous solutions ran their course in two different settings: in light conditions with ultraviolet radiation. The research evaluated how different elements affect dye crystal violet removal through adsorption by caper stem powder under light conditions along with unlightened circumstances. A solution containing 100 ppm dye solution needed 0.5 grams of powder during a 30-minute process to achieve the most effective result. The results showed that the removal percentage under light exposure reached 92.02% while the removal without light exposure reached 85.38%. The solution at pH level 11 showed the highest removal efficiency where the qualifying rate became 96.97% but without light exposure the rate was 11.88%. Researchers observed that increasing the dye concentration decreased the removal rate through 0.499% during irradiation and 3.295% without irradiation. The results indicated that the adsorption behavior followed Freundlich-type patterns better than Langmuir-type patterns according to linear equation correlation coefficients. Tests revealed that stem-based adsorption decreased as temperature increased while under irradiation treatment regardless of whether the stem was present or not. Mathematical thermodynamic analysis showed that the adsorption process was non-spontaneous while both the process and adsorption stopped providing randomness within the system.

Keywords: Photodegradation, adsorption, crystal violet dye, isotherm, thermodynamics.

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#### **1 - INTRODUCTION**

Human existence and evolution of all living organisms faces a severe danger from environmental pollution. The environment experiences substantial damage when polluting substances at various concentrations damage both living creatures and soil together with water and air. The extensive role that water serves in daily human existence makes it the principal reason why water pollution stands as the most critical pollution issue. The industrial sector heavily depends on water because various industries use large quantities of water which must persist within particular purity ranges determined by industrial demands and sector-specific requirements [2]. Numerous health problems arise in human populations from toxic substance formation and their aquatic environment entry [3]. Multiple businesses employ complex organic compounds known as dyes in order to stain their final products. Sexual problems and environmentally important dyes represent critical compounds because they function as essential components throughout our everyday activities. Synthetic dye components persist for a long time because they lack natural decomposition capabilities and build up within river zones and terrestrial environments thus generating ecological troubles [4]. Real-world examples of natural water as well as soil samples and plants contain heavy metals at detectable levels even though these substances remain toxic at small concentrations according to research [5].

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The rise of cadmium levels in specific regions stems from sewage as well as industrial emissions and practices [6]. During the last forty years governments from all around the world and especially developed nations have created environmental protection agencies as they recognized the worsening pollution crisis. Multiple researchers have started examining suitable techniques to eliminate compounds that now create environmentally dangerous conditions at different levels of concentration [7]. Environmental protection demands the use of crystal violet CV as representative cationic dye for risk control studies. The study evaluated the dye removal process through caper stem powder adsorption of dve solutions under different conditions which consisted of pH control and time stirring duration and temperature levels and solution concentration and powder quantity adjustments. Photodissociation describes how photons break chemical bonds either by straight or oblique exposure to light. Direct photodissociation involves photon strikes into bonds breaks whereas leading to bond indirect photodissociation occurs when a photocatalyst interacts with photons to produce an oxidation zone and reduction zone which generate strong cellular oxidants that can be either O2- or OH [8].

The most important techniques for solving this problem include photocatalysis and several physical chemistry methods such as coagulation, fusion, ozone saturation and adsorption on activated carbon, magnesium oxide, silica and clay [9]. The application of photocatalytic technology proves highly effective for eliminating various pollutants throughout multiple fields of use. This research implemented a decomposition and absorption method using caper plants that consist of pure carbon atoms organized into a two-dimensional structure. Researchers and industrialists aim at this material because of its exceptional features. A scientific assessment investigated how titanium dioxide nanoparticles function under photoactive conditions while decomposing methyl orange dye [10]. The use of

plant extracts in photoremediation provides better results while being more environmentally friendly than traditional chemical photocatalysts. Plant-based and plant extract photocatalysts facilitate wastewater cleanup operations alongside waste management from the dying industries and other similar sectors [11].

Natural plants exist in the environment since humans have not managed or altered them. Plants growing naturally in the environment contain active chemical substances that have useful biological properties. The caper plant as well as flavonoids and alkaloids represent the most significant active compounds found within these plants. The medicinal along with nutritional features of capers result from multiple chemical components which also afford other properties to this plant. The plant contains several biological compounds which include flavonoids alkaloids and organic acids in addition to other substances [12]. Flavonoids exist as natural phenolic compounds which are widely distributed among plants with capers being an example. The chemical compounds absorb UV radiation and create free radicals through which they function as effective natural photocatalysts. Flavonoids amplify organic pollution degradation like crystal violet dye (CV) degradation through their use with mercury lamps operating at 254 nm or 365 nm wavelength [13].

The saturated electron structure with  $\pi$ conjugated double bonds of flavonoids enables them to absorb ultraviolet (UV) radiation while promoting electronic excitation followed by electron movement from HOMO to LUMO states. Flavonoids gain activation by UV rays to create free radicals including the strong oxidant OH-• and the contributing pollutantoxidizing O2-•. Free radicals in solution use OH• as their attacking mechanism to break organic bonds of crystal violet pigment during the degradation process [14]. Flavonoids + hv (UV)  $\rightarrow$  Flavonoids\*  $\rightarrow$  ROS  $\rightarrow$  Dye Degradation



Figure 1: Shows the stem of the caper plant

#### **2- MATERIALS AND METHODS**

#### 2-1 Preparation of the Absorbent Material

Plant collectors obtained caper plant (stem) which received distilled water washing for dust particles elimination. The plant dried under sunlight for a duration of seven days. Several methods processed the cut caper plant material through grinding and sieving with a 200-micrometer sieve mesh.

#### 2-2 Preparation of the Dye Solution

Table (1) lists the crystal violet dye as a selected organic pollutant found in water. A solution containing 1000 ppm of crystal violet dye was prepared through dissolving 1 gram of dye into 1000 ml of distilled water. The dilution method served to create the other solutions from the prepared solution which followed the specified concentrations.



#### 2-3 Photochemical Study

A mercury vapor lamp served as the source of ultraviolet radiation in the photolysis experiment according to [15]. A medium-pressure type produces fast reactions that generate strong light power suitable for various material polymerization reactions. A 365 nm wavelength is the maximum emitted by this chargedischarge lamp [16]. Tests to evaluate caper plant photocatalytic performance utilized ultraviolet light for measuring crystal violet dye decomposition in water solution.

# 2-4- Practical experiments for removing (CV) dye with and without a light source when using stem powder.

The research used dilute aqueous solutions and evaluated crystal violet dye removal with and without the presence of light throughout the experiments. A Beaker contains both a specified volume of reacting solution and a certain amount of adsorbent before their mixture under magnetic stirring for a precise duration. [17]

The adsorbed solution gets separated from the adsorbent material by utilizing a centrifuge. The absorbance reading becomes a concentration value through analysis of a standard calibration graph. The evaluation of the remaining solution concentration proceeds after solution separation. The analysis process leads to determining both the residual concentration of solutions and the total amount of dye. A similar expression is used to determine the adsorptive removal rate according to [18].

$$R = \frac{(\text{Co} - \text{Ce})}{\text{Co}} \times 100$$
$$Qe = \frac{(\text{Co} - \text{Ce})}{\text{mg}} \times VL$$

R: Dye removal rate (%)

Qe: Amount of adsorbent (mg/g)

C\_0: Initial concentration of adsorbent (mg/L)

Ce: Concentration of adsorbent in solution at equilibrium (mg/L)

V: Volume of adsorbent (L)

m: Mass of adsorbent (g)

#### **3 - RESULTS AND DISCUSSION**

#### 3-1 Optical Absorption Spectrum

The UV-Visible spectrophotometers evaluated the absorption level of UV-Visible radiation. Hydrogen peroxide samples received natural lighting during preparation in regular quartz cells, using the distilled water dilution as Planck's solution. The multivariate analysis determined the crystal violet dye peak occurred at 591 nm since the measured peak appeared at 590 nm [19].



Figure 2: Maximum wavelength of crystal violet dye

#### 3-2 Calibration curve

The calibration curve for crystal violet dye used 10 ml solutions at various concentrations between 10-20 ppm of its previously prepared standard solution. Researchers obtained the absorbance measurements for these solutions when utilizing a maximum wavelength of (590) nm for each dye at its point of maximum absorption through operation against Planck's solution which represents water as the reference solution. The determined calibration curves generated from the absorbance and concentration data of aqueous solutions appeared as a straight line according to the preceding illustration.



Figure 3: Calibration curve of crystal violet dye at maximum wavelength  $\lambda$ max (905) nm

#### **3-3 FTIR Spectroscopy**

The active compounds on the surface of the selected caper stem powder were identified using

infrared spectroscopy. The powder spectrum was shown as shown in Figure (4), and the results were shown as mentioned in Table (2).



Figure 4: Infrared (IR) spectrum of the caper stem

Tuble 21 Interpretation of the T TIRt speetrum							
Ref	Possible compound:	Functional	Peak	Peak number			
		group	Intensity	(cm-1)			
[20]	Phenols, flavonoids, amines, or carboxylic acids	O-H or N-H	Wide	3400			
[20]	Alkanes	C-H	Strong	2900			
[20]	Ketones, aldehydes, or carboxylic acids	C=O	Strong	1700			
[21]	Alkenes or aromatic rings (phenols or flavonoids)	C=C	Medium	1500-1600			
[20]	Alkanes	CH3, CH2	Weak	1450			
[20]	Alkanes	CH3	Weak	1375			
[21]	Alcohols, ethers, or esters	C-0	Medium	1000-1200			

Table 2: Interpretation of the FTIR spectrum

# 3-4 Factors affecting the comparison of crystal violet dye adsorption efficiency in the presence and absence of a light source.

#### 3-4-1 Adsorbent Mass

The effect of adsorbent weight on the percentage of crystal violet dye adsorption was studied by taking different quantities (0.25-2 g) of caper stem powder, at a constant dye concentration and volume, and at laboratory temperature, both with and without light. The dye removal percentage from the powder surface

increases when the material weight goes up in photodegradation conditions. The presence of active chemical compounds in the powder enhances chemical reactions due to their response with light creating dye breakdown and promoting photodegradation while enhancing adsorption and neutralizing ultraviolet radiation-generated free radicals [22]. The number of active sites on the surface raises because of light exposure thus increasing the adsorbent surface area [23].



Figure (5) The percentage of dye removal when the mass of the absorbent material changes with and without the presence of light rays.

#### 3-4-2 Contact Time

A time span from 15 to 90 minutes at laboratory temperature under light and dark conditions served to evaluate crystal violet dye adsorption processes. The experiments established that dye removal percentages rose in direct correlation with the duration the solution remained in contact. The decomposition of dye happens more efficiently when light interacts with ultraviolet rays through extended contact between caper powder and the reaction length. This improved chemical response makes more free radicals to form and decompose the dye. The degradation process builds up as time passes resulting in greater efficiency of crystal violet dye removal [24]. The availability of active sites combined with adsorption time allows higher contact opportunities between the materials to occur in the dark environment. After an initial rise the process became stable until all active sites became filled [25].



Figure (6) Removal percentage of crystal violet dye with varying time, both with and without light.

#### 3-4-3 Initial Concentration of the Adsorbent

Multiple initial crystal violet dye concentrations from 25 to 100 ppm were employed in combination with a certain amount of caper plant (stem) powder to measure percentage removal under light and dark conditions. An elevated initial concentration of dye solution across the adsorbent surface resulted in decreased percentage of dye removal. The method of free radical production and effective powder-dye contact during oxidation in the solution becomes restricted due to two factors: surface saturation and chemical reaction blocking effects and light absorption processes which hinders the required free radical production for oxidation. The enhancement of chemical compounds within the system tends to deteriorate reaction efficiency and leads to decreased dye removal percentage [26]. Light exposure diminishes the number of accessible active sites located on the adsorbent surface surface as initial concentrations grow thus decreasing dye adsorption levels [27].



Figure (7) Removal rate of crystal violet dye with and without light rays.

The experimental results indicated that elevated values of weight capacity to remove dye from the surface correspond to rising initial dye concentration since many competing dye molecules target adsorbent sites leading to greater removal capacity. Low initial dye concentration leads to inadequate dye-site ratios on the surface which brings about fast adsorption rates [28].



Figure (8) Adsorption capacity and removal rate of crystal violet dye with and without light.

#### 3-4-4 Effect of pH

The research evaluated pH effects on crystal violet dye removal by testing different pH solution variants with fixed dye solution concentration and volume while using laboratory temperature light-dim conditions with a specific quantity of caper plant (stem) powder. Ultraviolet radiation together with basic conditions led to increased crystal violet dye removal percentages on the caper plant powder surface. Hydroxide ions in greater numbers act as oxidation catalysts responsible for this observation. A basic environment enables the occurrence of photolysis reactions. The separation of electrostatic bonds between dye and powder enables active substances in the caper

plant powder to bond more strongly with the dye molecule. The chemical bonds in the dye show increased susceptibility to degradation and the dye-powder reaction is stimulated by ultraviolet radiation when operated in a basic solution environment [29]. The surface obtains negative charge because of hydroxide ion excess and this positive charge draws the cationic dye toward the surface. The removal rate of the powder increases significantly as acidity levels rise until pH reaches a value of 11. The removalrate of the dye increases because hydrogen ions from the acidic environment bind to the available active sites on the surface [30].



Figure (9) Removal rate of crystal violet dye when the pH value changes with and without light.

#### 3-4-5 Temperature

Scientists conducted experiments on crystal violet dye removal under different temperature

conditions using a particular amount of caper plant stem powder under light and dark conditions. A decrease in crystal violet dye removal percentage occurred on the powder surface along with rising temperature. Ultraviolet radiation exists as the main factor behind this result. Too much temperature can degrade chemical compounds of both the powder and dye which in turn diminishes the capacity of dye to absorb photons needed for removal. High temperatures affect the composition of active substances within the powder and trigger adverse chemical reactions and cause powder agglomeration that results in lower crystal violet dye removal percentages [29]. An absence of light exposure reveals that the diffusing rate of spread molecules across the adsorbent surface becomes faster. The weakening of molecular attraction between the surface and its adjacent adsorbed molecule occurs as a result. At lower temperatures the bonds separating from each other become stronger so the crystal violet dye adsorbs better onto stem surfaces due to surface dye dissociation from temperature elevation [31].



Figure (10) Removal rate of the crystal violet dye when the temperature changes with and without light.

**4** - Adsorption Isotherms: From Figures (11, 12, 13, and 14) of the linear Freundlich and Langmuir equations, we note that the results of the dye isotherm have a high correlation coefficient with the linear Freundlich

equation both with and without light. This indicates that the adsorption of the dye onto the surface (stem) of the caper plant can be described as physical adsorption.







Figure (12) Langmuir equation for dye adsorption on the powder surface without light



Figure (13) Freundlich equation for dye adsorption on the powder surface in the presence of light





Isotherm	The sloping	الثوابت	CV	CV			
	surface		بالتشعيع	بدون			
Langmuir	stem powder	<b>R</b> <sup>2</sup>	0.9535	0.9394			
		b	0.62	0.16			
Freundlich	stem powder	<b>R</b> <sup>2</sup>	0.9901	0.9919			
		n	2.69	1.96			
		K <sub>f</sub>	6.106	3.317			

#### Thermodynamic studies of adsorption

Research was conducted to analyze how temperature influences the adsorption of crystal violet dye when using caper plant powder (stem) at four different temperatures from 30 to 60 degrees Celsius and a constant dye concentration of 100 ppm.

To describe the thermodynamics of the adsorption process, the thermodynamic functions, including the change in free energy ( $\Delta G$ ), the change in enthalpy ( $\Delta H$ ), and the change in entropy ( $\Delta S$ ), were calculated using the following equations [32].

∆G∘=-RT Ln K

Where

 $\Delta$ Go: the change in free energy in units of kJ mol-1

R: the universal gas constant (1 kJ k-1 mol -0.008314)

K: the chemical equilibrium constant for the equilibrium process.

In general, if the free energy value ( $\Delta G$ ) ranges between (-20) - (0) KJ mol-1, the adsorption is physical. However, if the values of ( $\Delta G$ ) range between (-400) - (-80) KJ mol-1, the adsorption is chemical. [33]

## The change in entropy and enthalpy can be calculated from the following equation:

 $lnK{=}\Delta S/R{-}\Delta H/RT$ 

The values of the change in enthalpy and the change in entropy are calculated from the slope and point of intersection of the graphical relationship below, Figure (15). The thermodynamic functions are calculated below in Table (4), indicating that the reaction is exothermic, there is a lack of randomness in the system during the adsorption process, and the process is non-spontaneous.







Figure (16) Effect of temperature on dye adsorption to the powder surface without light

Table 4: Thermodynamic constants with and without light rays							
Thermodynamic functions	$\Delta \mathbf{H^{o}}$	$\Delta S^{o}$	$\Delta \mathbf{G^o}$				
With light rays	-12.076085	-0.055332164	0.36119101				
Without light rays	-4.33550158	-0.018642482	1.354539232				

#### Table 4: Thermodynamic constants with and without light rays

#### From the results obtained in Table (3), we note that:

- 1. The positive free energy change ( $\Delta G$ ) values establish that the powder surface adsorption of crystal violet dye occurs through a non-spontaneous reaction [34].
- 2. The crystal violet dye shows negative enthalpy ( $\Delta H$ ) values for adsorption onto powder surfaces because the process occurs exothermically. The adsorbed molecules on the adsorbent surface gain extra kinetic energy when the temperature rises and they return to dissolve in solution.
- 3. The negative values of entropy change ( $\Delta$ S) when crystal violet dye adsorbs onto powder surfaces show that adsorbed molecules arrange themselves more orderly on the adsorbent surface than in liquid phase where they occupy specific surface locations with a particular spatial pattern [35].

#### 4. CONCLUSION

This research proved that the aqueous solution of caper plant extracts functions as a cost-effective material to eliminate crystal violet dye from liquid solutions. Aqueous plant extract size together with irradiation duration equally influences the removal rates under light and dark conditions. Dye adsorption onto this material depends on solution pH values together with adsorbent amount and contact duration and initial dye concentration. The adsorption isotherm follows the Freundlich model while showing no agreement with the Langmuir model. Dye adsorption onto the plant surface has been identified to be an exothermic process of spontaneous nature with lower disorder.

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