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ADSORPTION OF CRYSTAL VIOLET DYE FROM WASTE WATER BY USING ORANGE PEEL (*CITRUS SINENSIS*) AS AN ECO-FRIENDLY ADSORBENT

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ABSTRACT

Crystal Violet (CV) is a cationic dye commonly used in the textile industry and is considered a model pollutant due to its potential health hazards and environmental impact. To mitigate the harmful effects of CV in wastewater, adsorption is widely employed as an effective treatment method. This study aims to remove CV from aqueous solutions using activated orange peel as an adsorbent. In this study, batch adsorption experiments were conducted to investigate the effects of contact time, pH, adsorbent dose, and dye concentration on dye removal. Additionally, adsorption isotherms were analyzed to evaluate the performance of activated orange peel as an adsorbent. The maximum percentage removal of the dye using an adsorbent dose of 0.2 g was observed in a 2 $mg\ l^{-1}$ solution. The highest percentage removal was achieved at 98% in a sodium hydroxide (NaOH) medium and 97% in a sulfuric acid (H_2SO_4) medium. The dye removal per unit mass of orange peel increased with contact time, reaching a maximum percentage removal of 84% within 50 minutes. At ambient temperature, an adsorption efficiency of up to 91% was attained within 180 minutes. Equilibrium adsorption data were analyzed using Langmuir and Freundlich isotherm models. The data showed an excellent fit to the Langmuir isotherm model, with a high correlation coefficient (R^2). The results suggest that orange peel is a cost-effective, eco-friendly, and efficient adsorbent for the treatment of industrial wastewater.
Keywords **:** Adsorption, batch, crystal violet dye, orange peel, wastewater.

Introduction

According to the U.S. Geological survey, only a little more than 1.2% of freshwater is available as a surface water, which serves most of life's needs (Gleick, 1993). In the present scenario, burgeoning population growth, flourished industrialization and unsustainable usage of water resource cause water pollution in the world (Gul *et al.,* 2022). The use of dyes in textile industry is one of the factors that contributes the water pollution. Numerous kinds of industries use different dyes to beautify their products and discharge the waste water without proper treatment. Therefore, it is important to remove or reduce its concentration before releasing it into aquatic streams, as well as to control the cause of several diseases. Dyes is a coloured, mostly synthetic and complex aromatic structures, hence they are stable and non-biodegradable (Seshadri *et al.,* 2022). In this climate change era, many industries such as textile, carpet, garment, paper and pulp, plastics, printing ink, cosmetics and foodstuff industries, etc., use different dyes and they generate a considerable quantity of coloured effluent.

Crystal violet (CV) is a mixture of methyl pararosaniline stable dye and it has antibacterial and antifungal properties. Its high visibility helps in easy detection and also representative chemical for cationic dyes. CV dye is largely employed in veterinary and animal medicine as a biological stain (Sulthana *et al.*, 2022). Toxicological investigations have revealed that CV has carcinogenic and mutagenic effects in rodents. It may cause eye, skin, digestive tract irritation, permanent injury to cornea (Homagai *et al.,* 2022). In extreme condition, it may lead to respiratory and kidney failure and permanent blindness (Oguanobi *et al.*, 2019).

Various treatment methods are available for the removal of dyes from the wastewater. It includes chemical methods such as oxidative processes, H_2O_2-Fe (II) salts (Fenton's reagent), ozonation, photochemical and electrochemical destruction (Mittal *et al.,* 2010). Physical methods are adsorption, activated carbon, membrane filtration, ion exchange resins, irradiation, electro kinetic coagulation (Manenti *et al.,* 2010). Biological methods include aerobic and anaerobic degradation etc. but this treatment is not efficient because majority of dyes are non-biodegradable (Dutta and Bhattacharjee, 2022). In the present study, CV is used as model pollutant through adsorption study. Adsorption is the deposition of substance on the surface. It is a spontaneous and non-uniform. Adsorptive removal of pollutants is an economically feasible and effective technique for treatment of dye-contaminated wastewater (Al-Momani *et al.,* 2002; Okareh and Adeolu, 2015). This treatment reduces the chances of environmental pollution caused by chemicals (Alkherraz *et al*., 2020; Fosso-Kankeu, 2019; Zaidan, 2013). It is hypothesized that orange peel (*Citrus sinensis*) can serve as an effective, eco-friendly, and low-cost adsorbent for the removal of Crystal Violet dye from wastewater, demonstrating high adsorption efficiency under optimized conditions. With this background, the broad objective of the study is (i)To understand the effects of adsorption temperature, pH and dose on activated orange peel (*Citrus sinensis*). (ii)To determine the adsorption isotherm models of crystal violet dye.

Materials and Methods

Preparation of Adsorbent: In the present study, the oranges gathered from near local market. Firstly the oranges washed with distilled water in several times to eliminate all the contaminants. After that the endocarp removed from the orange peel, then the peel was allowed for sundried for 10 days. Then the dried material was cut down in small pieces upto 0.5 to 2.5 cm. It is used for the preparation of activated orange peel. This adsorbent was stored in airtight container for further analysis.

Preparation of Adsorbate: CV dye was used as the adsorbate. A stock solution of CV (1 g/L) was prepared by dissolving 1 g of the CV dye in 1000 ml of double distilled water to form a 1000 $mg\ l^{-1}$ solution. The basic properties of CV dye have been shown in Table 1.

Proximate Analysis: In proximate analysis moisture content, volatile matter, ash content and fixed carbon content was observed (Oikeh *et al*., 2013). Proximate analysis of the adsorbent (orange peel) was carried out using the procedure given in IS: 9235-1979.

Batch Adsorption: In this study to analyzed the different parameter consists of effect of dose, pH, time and temperature. All the batch studies were performed at the shaking rate of 180 ± rotation per minute (rpm). The pH values ranged from 2.0 to 12.0 for adjustment of ph. 0.1 N NaOH and 0.1 N H_2SO_4 solution were used.

Effect of Dose: Take 6 Nessler's tube, added the 0.2 g of adsorbent (Orange peel) in each one of them and added into the 2, 4, 6, 8, 10, 12 $mg\ l^{-1}$ adsorbate respectively, wait till 5 min and then filtered using the filter paper 42 and absorbance was measured in spectrophotometer at 470 nm.

Effect of pH: A series of 6 Nessler's tube having a capacity of 100 ml were taken. Added into the 12 $mg\ l^{-1}$ stock solution and 0.2 g of adsorbent (orange peel) in each Nessler tube. Out of 6 take 3 Nessler's added 2 ml, 4 ml, 6 ml H_2SO_4 in it and remaining tube was added 2 ml, 4 ml, 6 ml NaOH. Wait for 5 min, filtrate the solution and measured pH by pH meter. After that measure the absorbance in spectrophotometer at 470 nm.

Effect of Time (Kinetics): Take a conical flask and added into the 0.2 g of adsorbent (orange peel) in 12 $mg\ l^{-1}$ solution, diluted with 50 ml distilled water, after that conical flask Put into the shaker for shaking the sample at different time intervals starting from 2 to 180 minutes (2, 5, 10, 20, 30, 45, 60, 75, 90, 120, 150 and 180 min) at 180 rpm. Samples were taken out of shaker at appropriate time and were filtered. Absorbance measured of residual crystal violet dye at spectrophotometer at 470 nm.

Effect of Temperature: The effect of temperature on the adsorption characteristics was investigated by determining the adsorption isotherm. 12 $mg\ l^{-1}$ solution were taken in series of conical flasks and temperature of the solution was adjusted to 30 ^{0}C, 40 ^{o}C and 50 ^{o}C. The flask was shaken on mini rotary shaker for 2 min at 180 rpm, then filtered by filter paper. The concentration of dye in the supernatant liquid for all three concentrations were determined spectrophotometrically at 470 nm.

Adsorption Equilibrium: Experiments were carried out at the optimum pH by contacting a fixed amount of adsorbent (0.2 gm) respective adsorbate solution having C_o i.e., 12 $mg\ l^{-1}$. After contact time of 2 min the mixture was filtered and the filtrate was analyzed for finding out the residual adsorbate concentration (Ce) (Nuhu, 2018).

$$\%\,\text{Removal} = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

Freundlich isotherm: Freundlich adsorption isotherm is based upon the multilayer adsorption on heterogeneous sites of adsorption of adsorbate molecules on the adsorbent surface having unequal binding sites with different adsorption energies (Mittal *et al.*, 2009). The mathematical model can be shown as,

$$q_e = KC_e^{1/n} \quad (2)$$

Taking log on both sides, the linearized form can be written as,

$$\log_{qe} = \log K + \frac{1}{n}\log C_e \quad (3)$$

where,

q_e(mg/g) = The equilibrium capacity (amount of CV adsorbed by the orange peel at equilibrium)

C_e (mg/L) = The equilibrium adsorption concentration (CV solution)

K = The Freundlich constant (adsorption capacity)

A plot of log q_e verses log C_e produces a straight line with a slope = $\frac{1}{n}$ and intercept = log K.

Langmuir isotherm: Adsorption is a homogeneous process. It is an empirical model based on kinetic principles. That is the surface rates of adsorption and desorption are equal with zero accumulation at equilibrium conditions (Langmuir, 1916). This isotherm is based on the following assumptions monolayer adsorption, homogeneous sites, constant adsorption energy, and no lateral interaction between the adsorbed molecules. The Langmuir isotherm can be written as,

$$q_e = \frac{q_0 K C_e}{1 + KC_e} \quad (4)$$

where,

q_e (mg/g) = The equilibrium adsorption capacity (quantity of dye adsorbed per specific unit mass of adsorbent);

q_o (mg/g) = The maximum adsorption capacity (amount of adsorbed surfactant);

C_e (mg/L) = The equilibrium adsorption concentration;

K = The Langmuir constant in (L/mg).

The reversibility of the adsorption and presence of adsorption sites of fixed numbers describes the nature of the model. Linear form of the Langmuir model (Banerjee and Chattopadhyaya, 2017). A plot between $\frac{C_e}{q_e}$ versus C_e will generate a straight line with a slope of $\frac{1}{n}$ and an intercept is logK.

$$\frac{1}{q_e} = \frac{1}{Kq_0C_e} + \frac{1}{q_0} \quad (5)$$

$$\frac{C_e}{q_e} = \frac{1}{Kq_0} + \frac{C_e}{q_0} \quad (6)$$

Results and Discussion

In the present study, proximate analysis results indicate that orange peels are generally suitable for waste water remediation purposes (Table 2). The use of UV-Visible Spectrophotometer, the concentrations of CV dye in the solutions were determined. The range of calibration curve of dye prepared from the stock solution (1000 *mg* l^{-1}) varies between 2-12 *mg* l^{-1}, as shown in Fig. 1 and the linear regression equation was y = 0.0462x with a high correlation coefficient (R^2= 0.99). It exhibits the amount of CV adsorbed increases with increasing concentration. The highest concentrations of crystal violet in the solutions were determined at 12 *mg* l^{-1} concentration. The Influence of adsorbent dosage on the removal of CV with different concentrations ranging from 2 to 12 *mg* l^{-1}. The adsorption capacity of adsorbent was determined by adding same quantities of adsorbent dose i.e., 0.2 g, Fig. 2 shows that the dosage of adsorbent significantly influences the amount of crystal violet adsorbed. Increasing the amount of adsorbate from 2 to 12 *mg* l^{-1}, led to a decrease in adsorbent capacity because with increasing adsorbate dose, unsaturated residual adsorption sites reduce adsorption capacity during the adsorption process (Clavete *et al.,* 2010). The maximum percent removal in orange peel was found to be in 2 *mg* l^{-1} solution (Fig. 3). The percent removal of CV decreases with increases in adsorbate dosages because of no more availability of more adsorbent sites.

The adsorption of dyes at the surface of adsorbent is also influenced by solution pH, because change in pH affects the surface charge on the adsorbent. Batch adsorption experiment was brought out by changing pH of solution from 2 to 10 by keeping other experimental conditions constant at adsorbent dose 0.2 g, with initial concentration is 12 *mg* l^{-1} solution of CV dye and adding acidic and alkaline solution in each tube at the range from 2, 4, 6 ml. The maximum percent removal of NaOH was found to be 98% at 8 pH and H_2SO_4 is 98% at 4 pH. (Fig. 3) In acidic medium, the percent removal of CV dye is highest at pH 2 and then, decreases slightly with increasing pH, after which it remains almost constant. This might be due to change in adsorbate structure, and the functional groups present at the surface of adsorbent may change with change in pH and can be protonated or deprotonated to generate different surface charges at different pH in the solutions, leading to an electrostatic attraction/repulsion between the charge's adsorbents and adsorbates (Alshabanat *et al*., 2013; Cheruiyot *et al*., 2019).

The adsorption study was performed at different time intervals ranging from 2 to 180 min to determine the equilibrium contact time for the removal of CV dye from the aqueous solutions. Other experimental conditions were kept constant. The experimental results are given in Fig. 4. As the contact time increased, the adsorption increased until it reached an equilibrium state at 45 minutes. The adsorbent surface performs fast adsorption protection in the initial period, with a gradual decrease in the adsorption speed due to the blockage of the adsorption sites. The decline continues until it reaches an equilibrium state. This phenomenon explains that adsorption sites are initially active sites but are gradually filled with time, which leads to blockage of absorption sites and a noticeable decrease or lack of availability of the free site (El-Hashani *et al.,* 2018; Elsherif and Yaghi, 2017; Elsherif *et al.,* 2018a, b). Hence, the equilibrium contact time for the CV adsorption onto adsorbent was 180 min with 91% efficiency. The influence of temperature on the adsorption was investigated at three different temperatures (30°C, 40°C, 50°C). The graph shows that the adsorption of CV is slightly decrease and then gradually increase in temperature (Fig 5). Adsorption increases with increase in temperature, which indicates the adsorption process is endothermic.

The variation in the amount of adsorbate adsorbed on the surface of adsorbent with the change in pressure at constant temperature can be expressed by mean of a curve termed as called Adsorption isotherm (Kalam *et al*., 2021). In the present work, two adsorption isotherms Freundlich and Langmuir have been explored. The Freundlich isotherm describes multilayer adsorption and assumes exponential decay in the energy distribution of adsorbed sites. However, it is not valid for a large range of adsorption data (Al-Ghouti and Daana, 2020). Langmuir isotherm, the monolayer assumption requires identical adsorption sites, and only one molecule can be adsorbed at each site. There is no more adsorption in a site once a surfactant molecule has occupied it. This model converts to Henry's model at very low concentrations ($KC_e << 1$). The L-shape curve shows the Langmuir isotherm model having a single plateau (Table 3). As compare to both model of adsorption isotherm, langmuir model the value of R^2 is closer to the 1 as compared to the Freundlich isotherm model for the adsorption of CV dye. Hence, it is clearly noticeable that langmuir model is better fit in the adsorption isotherm model. The results indicates that the adsorption process is more likely to be monolayer.

In this study, orange peel (*Citrus sinensis*) was successfully used for the removal of CV dye from its aqueous solution. The technique of batch operations was used to explain the removal process. The batch adsorption investigations were done by optimizing various parameters such as adsorbent dosage, pH, contact time and temperature for CV dye removal onto adsorbent (orange peel). The adsorption was single layer adsorption as per the Langmuir model. Equilibrium adsorption data fitted well with the langmuir isotherm model ($R^2 = 0.97$). Thermodynamic analysis indicated the process to be spontaneous and endothermic. On the basis of R^2 for this study Langmuir isotherm is valid. These findings suggest that orange peel holds promise as a cost-effective, eco-friendly adsorbent for treating wastewater contaminated with organic crystal violet dye. Its potential as a good adsorbent can be explored for other toxic dyes as well as other hazardous pollutants. This can be concluded that orange peel which are waste materials and which generate abundance in our country can be used for the removal of CV dye from aqueous solution.

Table 1: Basic properties of Crystal Violet (CV) dye

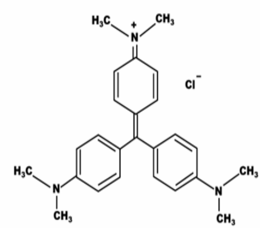
Property	Result	Chemical Structure
Another name	Gentian violet or basic violet 3	
Molecular formula	$C_{25}H_{30}N_3Cl$	
Molecular weight	407.98 g/mol	
Appearance	Purple solid	
Colour	Greenish to dark-green powder with metallic luster	
Dissolved in water	Blue-violet colour.	
Maximum absorbance	590 nm	

Table 2: Proximate analysis of Orange Peel

Parameter	% Removal
Moisture Content (MC)	10-12
Ash Content (AC)	0.3-0.5
Volatile Matter (VM)	78-80
Fixed Carbon Content (FCC)	10-12

Table 3: Langmuir and Freundlich adsorption model

Isotherm Model	1/n	logK	R^2
Freundlich	-0.15	-0.17	0.96
Langmuir	-0.49	1.91	0.97

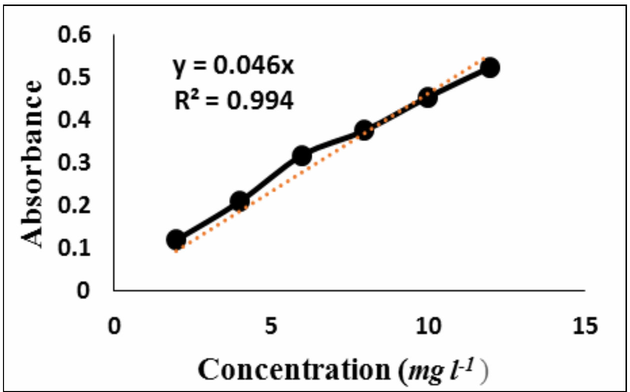


Fig. 1: Calibration curve of Crystal Violet (CV) at 470 nm

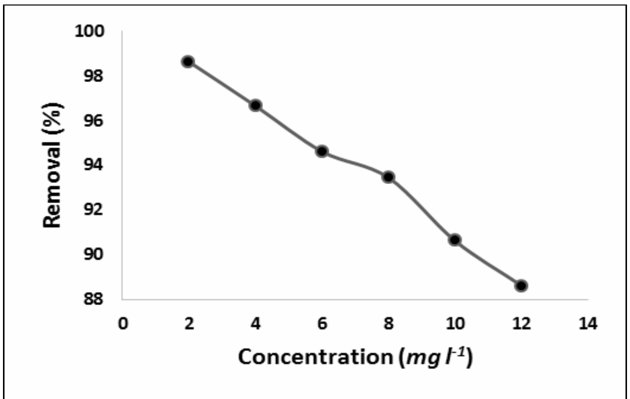


Fig. 2: Effect of concentration vs. % removal of CV dye

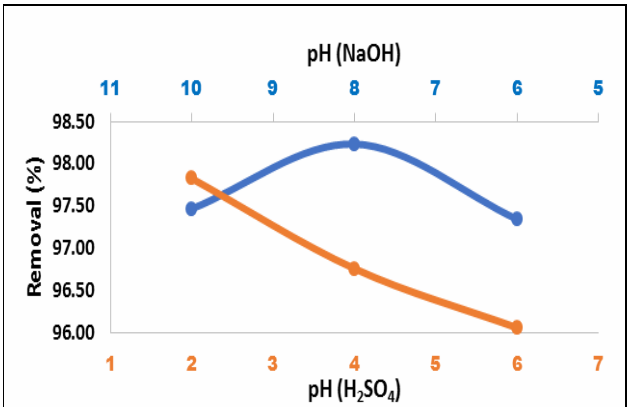


Fig. 3: Effect of initial pH on removal of CV dye

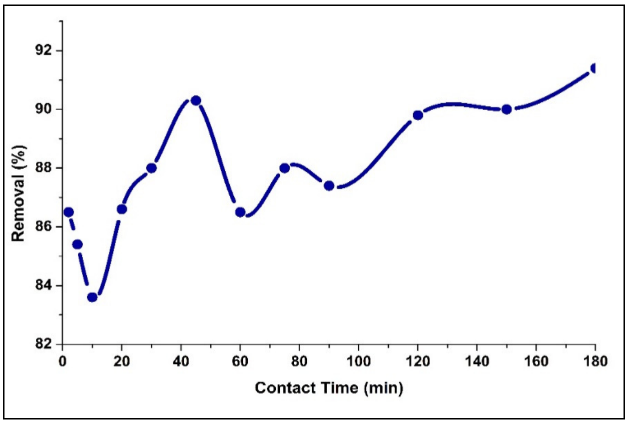


Fig. 4: Effect of contact time on removal of CV dye

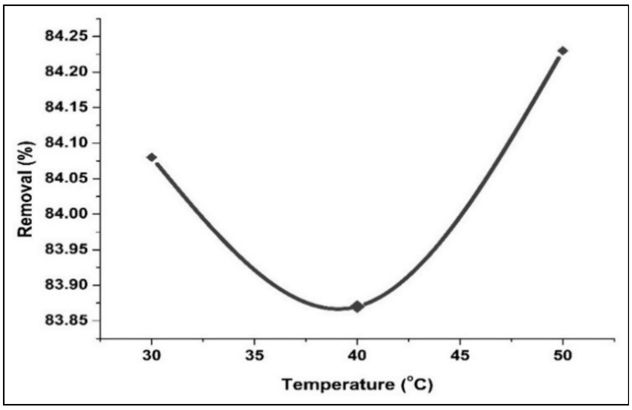


Fig. 5: Effect of temperature on removal of CV dye

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Data availability: On request, data will be available.

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