

The adsorption of Crystal violet from aqueous solution by using potato peels (*Solanum tuberosum*): equilibrium and kinetic studies

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Abstract

The present study shows that the potato peel (PP) can be used as a potential agricultural by-product adsorbent for the removal of Crystal Violet (basic dye) from aqueous solution. The potato peel (PP) without any modification, was characterized by Fourier transform infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) coupled by EDX. In batch optimization studies, the maximum removal efficiency was obtained at the optimum levels of contact time (20 min), adsorbent dose (0.5 g.L⁻¹), initial dye concentration 20 mg.L⁻¹ and pH 6. The equilibrium adsorption data was found to follow the Langmuir isotherm model and maximum monolayer capacity was found to be 555 mg.g⁻¹. Also the Langmuir and Freundlich isotherm models constants R_L and n suggesting favorable adsorption of CV on the PP. Kinetic analysis revealed that adsorption experimental data was best fitted by pseudo-second order model ($R^2=0.996$). The results suggest that potato peel would be suitable for removing Crystal violet from aqueous solution.

1. Introduction

The dye wastewater has long been a major environmental problem all over the world. The problem is further aggravated by rapid industrialization, population growth and unskilled utilization of natural water resources [1]. The discharge of effluent containing synthetic dyes from several industries like textile, printing, leather and cosmetics into the hydrosphere have adverse effect in environment. The presence of very small amounts of dyes in water (less than 1 ppm for some dyes) is highly visible and undesirable [2]. It is already demonstrated that dyes damage the aquatic ecosystem by varying the path of sunlight which largely affect the photosynthetic activity (lowers down the dissolve oxygen) [3]. Moreover, many dyes are toxic and even carcinogenic thus affecting the human health [4]. As a representative of synthetic dyes, Crystal violet (CV) is a cationic dye that can easily interact with negatively charged cells membrane surfaces, enter into cells and can concentrate in cytoplasm [5]. CV is a member of triphenylmethane group. Even though the dye is carcinogenic it is still extensively used as a biological stain for identifying the bloody fingerprints being a protein dye, as a dermatological agent and is used in various commercial textile operations [6]. It is also used in Gram's stain for the demonstration and primary classification of dyes [7].

Many researchers have reported that Crystal violet could induce carcinogenic effects, is toxic to mammalian cells and also a mutagen, mitotic poison [8]. It can cause skin and digestive tract irritation. It is responsible for moderate eye irritation, causing painful sensitization to light. It can cause permanent injury to the cornea and conjunctiva [9], which is also non-biodegradable and persists in the environment for longer time [10].

Therefore, wastewater containing CV must be purified before release in the environment to protect the ecosystem and human health. For this purpose, several methods are commonly used. These treatment options include biological, physio-chemical, membrane filtration, ozonation, advanced oxidation and integrated

treatment processes [2]. However, major drawbacks of these technologies are long operation time, high sludge production, high cost, not eco-friendly etc [11]. While adsorption process is alternative due to its performance and ease of operation as well as possibility of regeneration. Utilization of activated carbon as an adsorbent has been found to be most effective in dye removal, but its high cost and its loss during the regeneration restricts its application [12]. Major advantage of adsorption is use of low cost adsorbents especially agricultural wastes. Production of adsorbent from different wastes not only achieves the removal of pollutants in harmless forms but also has environmental effects of reducing waste [13].

The present work was undertaken to evaluate the application potential of potato peel as a low-cost adsorbent for the removal of Crystal violet (CV) from aqueous solutions. The effect of different adsorption factors such as solution pH, adsorbent dose, initial dye concentration and contact time was studied. Kinetic and isothermal studies of the adsorption process were carried out to understand the equilibrium and mechanism of adsorption process.

2. Materials and methods

2.1. Preparation of adsorbent

The waste potato peel (PP) were washed with distilled water to remove dust and other inorganic impurities, filtered and oven-dried for 24 h at 343 K to reduce the moisture content. Dried biomass was grind and sieved to get the particle ranges from 75 to 150 μm .

2.2. Adsorbate solution

The adsorbate, Crystal Violet (CV) dye (chemical formula = $\text{C}_{25}\text{H}_{30}\text{N}_3\text{Cl}$, Molecular weight = $407.98 \text{ g}\cdot\text{mol}^{-1}$, solubility in water $16 \text{ g}\cdot\text{L}^{-1}$ at 25°C) was obtained from Sigma Aldrich (purity = 99%). The structure of Crystal violet is illustrated in Fig.1. Stock solution was prepared by dissolving accurately weighed quantity of dye in $1000 \text{ mg}\cdot\text{L}^{-1}$ of distilled water. The experimental solutions of desired concentration were prepared accordingly by diluting the stock solution with distilled water. The concentration of CV dye was measured at $\lambda_{\text{max}} = 509 \text{ nm}$ using UV-Visible spectrophotometer.

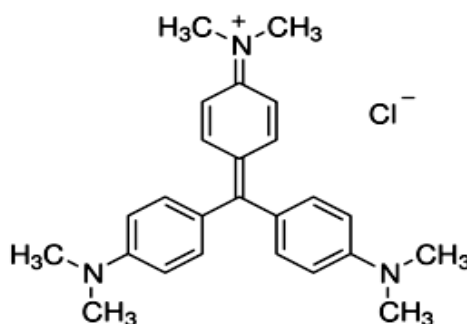


Fig 1. Chemical structure of Crystal violet.

2.3. Adsorbent characterization

The morphology of PP was analyzed by Scanning Electron Microscopy (SEM) coupled by Energy Dispersive X-Ray analysis (EDX) for microanalysis of surfaces (Quanta 200 FEI).

The functional groups that are involve in adsorption of CV on the PP were analyzed by Fourier Transform Infrared Spectroscopy (FTIR) using a range sweeping wavelength $400\text{-}4000 \text{ cm}^{-1}$ (BRUKsER Vertex70).

2.4. Adsorption experiments

The removal efficiency of the potato peel was studied by batch adsorption experiments (all experiments were run in duplicate). Batch studies for CV were carried out to investigate the contact time, adsorbent dose, initial concentration of CV and initial pH of solution.

Since the adsorption process of CV is fast, the time required for the solid phase in contact with the solution phase system to achieve equilibrium was determined by preliminary experiments. Approximately, 2 g of the adsorbent was added to 200 mL of CV solution (20 mg.L⁻¹) and the resulting suspension was shaken at ambient temperature (30 °C), at pre determined time intervals (2-60 min) to ensure establishment of equilibrium. The PP was separated by filtration through a 0.45 µm pore size of cellulose membrane. Residual concentration of crystal violet was determined using a UV/vis spectrophotometer at 509 nm.

Then the dose effect of potato peel powder was studied with different interval (0.25-1.5 g.L⁻¹), each mass of adsorbent were added to V = 200 mL of CV solution (20 mg.L⁻¹) in Erlenmeyer flask at equilibrium time.

The effect of initial dye concentration on equilibrium was observed by mixing 0.5 g.L⁻¹ (optimum mass found) of adsorbent with 200 mL of CV solutions with different initial concentrations ranges from 10 to 50 mg. In order to investigate the effect of initial solution pH on the adsorption property of CV, solutions of 30 mg.L⁻¹ (the optimum value according to concentrations-effect tests) with the same amount of adsorbent (0.5 g.L⁻¹) were adjusted with a pH-meter to different values by adding drops of HCl and NaOH (0.1 M) solutions and shaken at 80 rpm for 25 min at ambient temperature

The percent removal of CV from solution was calculated by the following equation:

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

Where C₀ is the initial concentration of dye and C_e is the concentration of dye at equilibrium.

The adsorption capacity q_e (mg.g⁻¹) after equilibrium was calculated by mass balance relationship equation as follows:

$$Q_e = \frac{C_0 - C_e}{W} \times V \quad (2)$$

Where V is the volume of the solution (l) and W is the mass of adsorbate (g).

2.5. Theory

2.5.1. Adsorption isotherm

The adsorption isotherm models generally used to design and to understand the mechanism of interaction existing between adsorbate and the adsorbent at equilibrium [14]. In this work, two isotherm models (Langmuir and Freundlich) were applied to describe the distribution of dye ions between the liquid phase and the solid phase at constant temperature.

The Langmuir model assumes monolayer adsorption on a structurally homogeneous adsorbent, where all the adsorption sites are energetically uniform. Is based on the assumptions that each active site can only react with one dye molecule, no further Adsorption can take place at that site [15]. The Freundlich model assumes heterogeneous energetic distribution of active sites, accompanied by interactions between adsorbed molecules [16].

The linear form of the Langmuir and Freundlich isotherm models are given by Eqs. (3) and (4) respectively.

Langmuir equation:
$$\frac{1}{q_e} = \frac{1}{q_{\max}} + \frac{1}{(K_L \times q_{\max})} \times \frac{1}{C_e} \quad (3)$$

Freundlich equation:
$$\text{Log}(q_e) = \text{log}(K_F) + \frac{1}{n} \text{Log}C_e \quad (4)$$

Where q_e (mg.g⁻¹) and C_e (mg.L⁻¹) are the adsorption capacity and the concentration of the dye at equilibrium, q_m (mg.g⁻¹) is the maximum adsorption capacity, K_L (L.mg⁻¹) is the Langmuir constant related to the free energy of adsorption. K_F (mg.g⁻¹) (L.mg⁻¹)^{1/n} is the Freundlich isotherm constant indicative of the adsorption capacity, 1/n is the adsorption intensity which should have a value in between 0.1 and 1.0 for favorable adsorption.

The essential characteristics of Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor or equilibrium parameter. R_L is defined as [17]:

$$R_L = \frac{1}{1 + K_L C_0} \quad (5)$$

Table 1. The parameter R_L indicates the shape of isotherm.

R_L value	Type of isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

2.5.2. Adsorption kinetics

Kinetic studies are carried out to provide information about the mechanism of adsorption, two classical kinetics models, a pseudo-first order kinetic model of Lagergren (Eq. (6)) and a pseudo-second order kinetic model of Ho (Eq. (7)) were employed to test the experimental data of dye adsorption by potato peel.

Pseudo-first-order [18]:
$$\text{Log}(q_e - q_t) = \text{Log}q_e - \frac{K_1}{2.303}t \quad (6)$$

Pseudo-second-order [19]:
$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e}t \quad (7)$$

Where q_t (mg.g^{-1}) and q_e (mg.g^{-1}) are the adsorption capacity of dye at time t and at equilibrium, k_1 (min^{-1}) and k_2 ($\text{g.mg}^{-1}.\text{min}$) are the rate constants of pseudo-first order and pseudo-second order models respectively. The best-fit model was chosen based on the values of the regression coefficient (R^2) of the linear plots of pseudo-first order and pseudo-second order kinetic model equations.

3. Results and discussion

3.1. Batch optimization studies

3.1.1. Effect of contact time on CV adsorption by pp

The performance of adsorption process was highly influenced by the contact time. It assists to find out the time required for equilibrium attainment for CV adsorption on to PP. The study of this parameter can save money and energy in the case of adsorption process on an industrial scale. The contact time in the range of 2-60 min was investigated. Other variables i.e., dye initial concentration (30 mg.L^{-1}), pH; 6, adsorbent dose (2 g) and 30°C were constant and results thus obtained are represented in Fig.2. The adsorption capacity of PP increases with time and attains saturation in about 20 min. Initially, the removal of Crystal violet is rapid, but it gradually decreases with time until it reaches equilibrium. After attaining the equilibrium, further adsorption was insignificant as a function of contact time

The rate of adsorption capacity is higher in the beginning due to the availability of active sites on the PP adsorbent; there are a number of accessible active sites on the adsorbent surface [26]. Whereas the slow CV adsorption was due to the saturation of binding sites and dye ions occupy the remaining vacant sites slowly due to repulsive forces between the dye ions and ions already adsorbed on the adsorbent [27]. Saeed et al. [28] also

found that the most of CV dye adsorption took place within <5 min. The rapid adsorption phase spanned for <20 min resulting in > 80 % dye sorption.

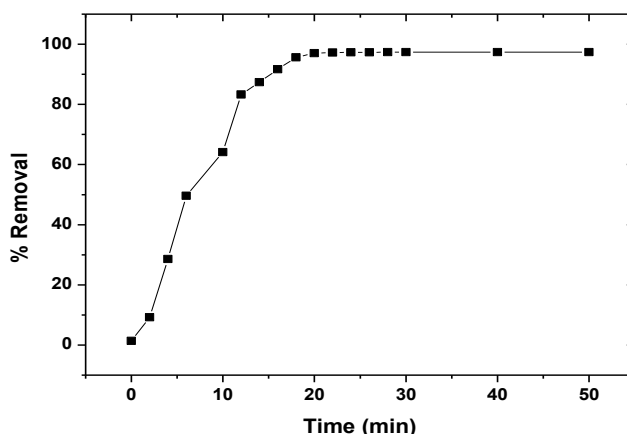


Fig 2. Effect of contact time on adsorption of CV on PP (pH_i 6, Co: 50 mg.L⁻¹, T: 30°C, Co: 30 mg.L⁻¹, PP dose: 2 g)

3.1.2. Effect of adsorbent dose

It is well recognized that the amount of adsorbent is an important parameter that affects adsorption capacity. In order to observe the minimum possible amount which shows maximum adsorption, the effect of PP dose on CV removal was studied by keeping all other experimental conditions constant.

The effect of dose is examined by varying the amount of the adsorbent from 0.25 to 1.5 g in 200ml of dye solution (30 mg.L⁻¹). The results revealed that removal efficiency and adsorption capacity showed inverse relationship with adsorbent dose. It is clear from fig. 6 that with increase in the amount of the adsorbent, the % removal increases from 87.89 % to 93.45 % this can be attributed to increased surface area and the adsorption sites, as already reported [29]. Conversely, there is a decrease in the adsorption capacity. However, the dye removal remains constant, whereas the adsorption capacity decreases at PP dose >1.2 g.L⁻¹. This decreasing adsorption capacity at higher adsorbent dose was due to low surface area may be attributed to saturation of adsorption sites due to particulate interaction such as aggregation [21, 24]. The results of this experiment indicate that 0.5 g of adsorbent is considered optimal for maximum CV removal (The cross point of the two curves, fig.3).

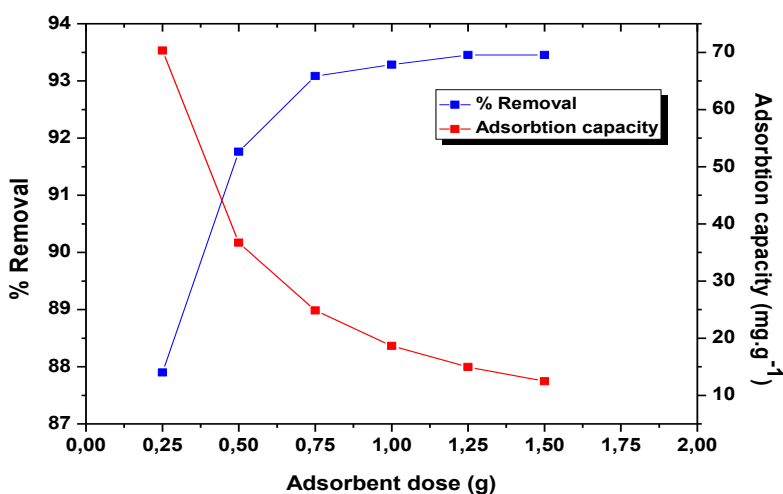


Fig 3. Effect of adsorbent dose on the adsorption of CV on PP. (pH_i 6, T: 30 °C, Co: 30 mg.L⁻¹, Contact time: 20 min)

3.1.3. Effect of initial dye concentration

To reach the maximum values of adsorption capacity representing the saturation of all available active sites on the surface of the adsorbent, the effect of initial CV concentration (10-50 mg.L⁻¹) on the adsorption efficiency and adsorption capacity of PP was investigated and the results are given in Fig.7. The results revealed that adsorption efficiency and adsorption capacity have opposite correlation against an increase in initial CV concentration. Adsorption capacity of PP increased from 18.36 to 99.35 mg.g⁻¹, whereas the removal efficiency decreased from 98% to 89% against an increase in CV concentration from 10 to 50 mg.L⁻¹, respectively. Similar results have been reported by other researchers previously for different adsorbents [6, 26, 27, 30].

As may be noted from Fig. 4, the maximum amount of dye adsorbed on potato peel was 99.35 mg.g⁻¹. The value is much higher than the other bio-waste materials previously reported as adsorbents for the removal of Crystal violet. Atmani et al [31] showed that adsorption capacity of CV on skin almond waste increased as a function of initial dye concentration. However, the maximum adsorption capacity presented in their study was 85.47 mg.g⁻¹. In another study, Senthilkumar et al. [6] found that the maximum adsorption capacity is 60.42 mg.g⁻¹ for the adsorption of CV by activated carbon from male flowers of coconut tree.

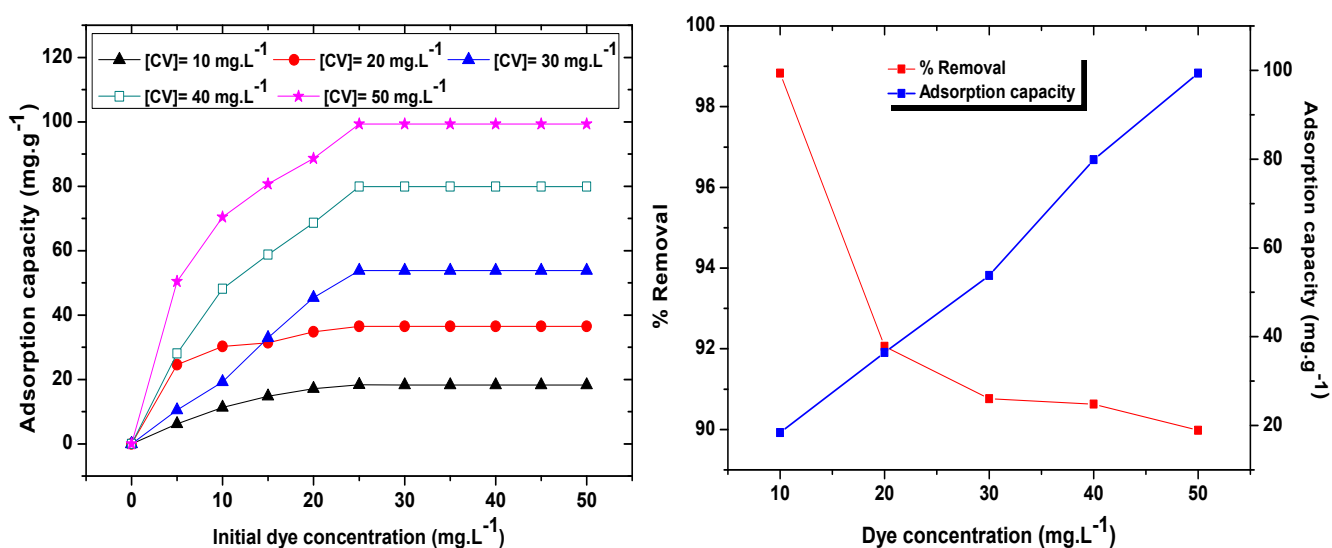


Fig 4. Effect of initial ion concentration on adsorption of CV on PP. (pH_i 6, T: 30°C, PP dose: 0.5 g, Contact time: 20 min)

Auta and Hameed [32] argue that the adsorbent surface contains fixed number of available sites per unit mass of the adsorbent. The higher adsorption capacity observed at lower concentration was attributed to probable proportionate number of vacant sites to the molecules of the adsorbate at those lower concentrations. However, the lower adsorption yield at high concentration of CV may be attributed to saturation of surface active sites as well as possible repulsive interactions between the dye molecules on the solid and bulk phases [33].

3.1.4. Effect of pH

The pH of an aqueous solution influence on the surface properties of the adsorbent and ionization/ dissociation of functional groups on the adsorbate molecule [28], which play important role in the whole adsorption process. The effect of solution pH was studied for adsorption of Crystal violet on potato peel at temperature 30 °C, initial concentration (20 mg.L⁻¹) and 0.5g amount of adsorbent. The range of solution pH was adjusted between 2 and 12. It was observed that the percent removal of CV increase with the increase in pH of the dye solution, appreciably up to pH 6.0 (Fig. 5). A further increase in dye adsorption between pH 6.0 and 12 was insignificant. Since the optimum pH for dye adsorption by PP was found to be 6.0, this pH was used for further studies. The percent removal of CV was found to increase from 84% at pH 2 to 93% at pH 12. This behavior can be explained on the basis of change in surface charge of the PP. In case of basic dyes (Crystal violet) at lower pH, the H⁺ ions will compete with the dye cations causing a decrease in percent value [26]. As the pH increases, the

adsorption of CV increases. So the surface of the adsorbent becomes positively charged, and this decreases the adsorption of the positively charged dye ions through electrostatic repulsion [34]. The insignificant removal of the dye, observed at pH between 6 and 12 can be attributed to a cationic exchange process between the dye molecule and the adsorbent with the release of protons (H^+), as suggested in an earlier study [35].

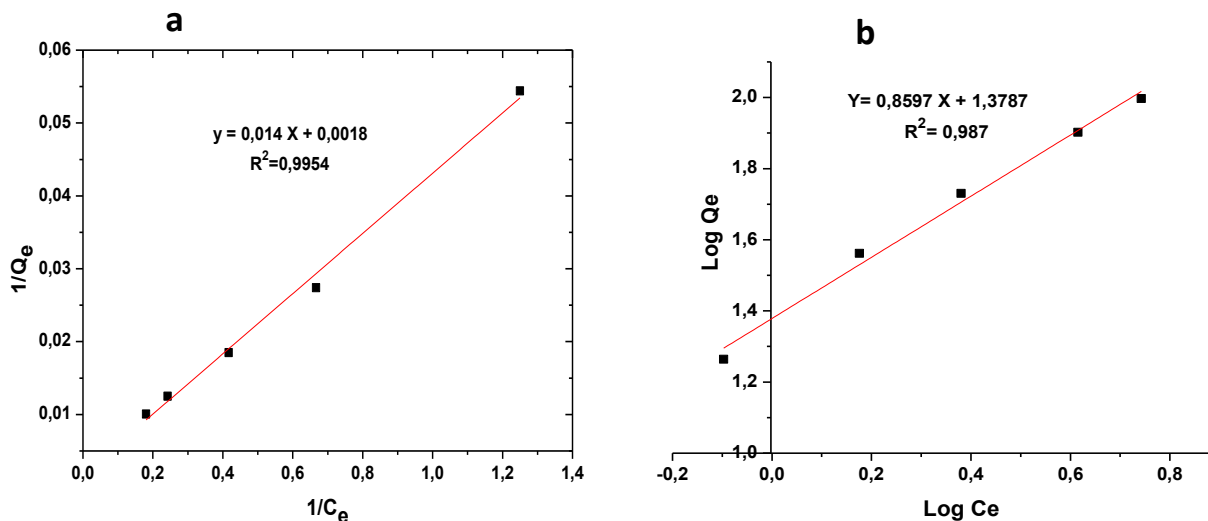


Fig 5. Effect of pH on the adsorption of CV on PP. (pH; 6, T: 30°C, PP dose: 0.5 g.L⁻¹, C₀ : 20 mg.L⁻¹, Contact time: 20 min)

3.2 Adsorption isotherm studies

In order to represent the adsorption system at equilibrium, two isotherm models (Langmuir and Freundlich) were applied in this work to test the obtained experimental results. Adsorption isotherm studies are performed at the concentration ranges from 10 to 50 mg.L⁻¹. A comparison of the Langmuir (Fig. 6a) and Freundlich (Fig. 6b) adsorption isotherms show that the adsorption characteristics of CV dye onto PP followed more closely the Langmuir isotherm equation (Eq. (3)) than the Freundlich isotherm equation (Eq. (4)). This observation is further supported by the higher value of R^2 (0.995), which regarded as a measure of the goodness fit of experimental data on the isotherm's models.

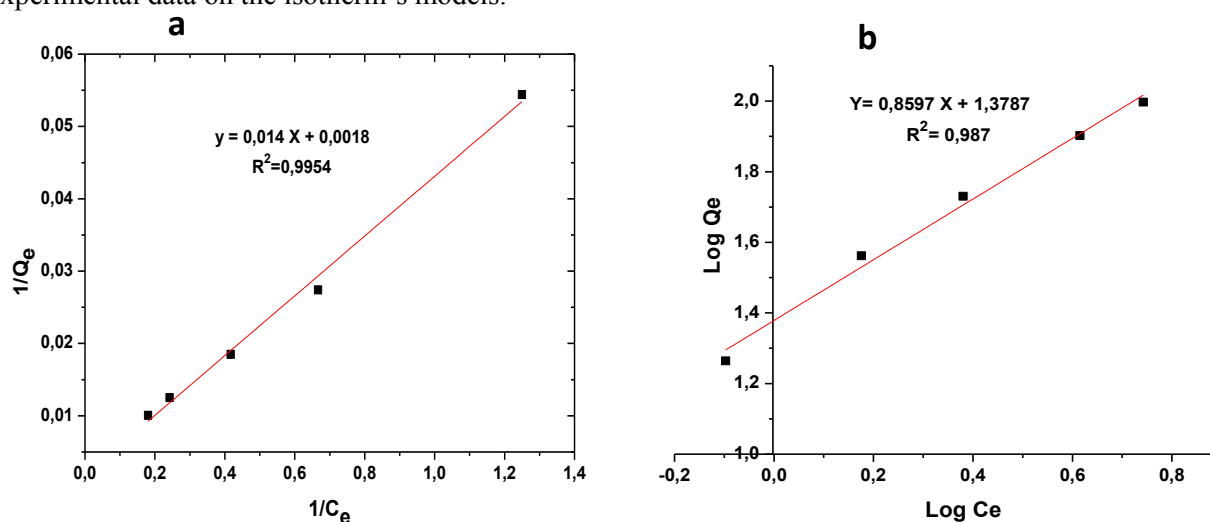


Fig 6. Langmuir (a) and Freundlich (b) model plots of crystal violet adsorption.

The estimated parameters for the different models are summarized in Table 2. As observed, the values of R_L and $1/n$ lie in between 0- 1 and 0.1- 1.0 respectively, it represents favorable adsorption process.

The maximum monolayer adsorption capacity (q_{max}) was estimated as 555 mg.g⁻¹. This value was compared to those of some adsorbents reported in literature as seen in Table 2.

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Table 2. Adsorption capacity (q_m) of various adsorbents for the crystal violet dye.

Adsorbent	Q_m (mg.g^{-1})	Reference
Tomato plant root	94.3	[36]
Sulphuric acid activated carbon (male flowers coconut tree)	85.8	[37]
skin almond waste	85.47	[31]
Treated ginger waste	64.9	[38]
Activated rice husk	64.87	[42]
Phosphoric acid activated carbon (male flowers coconut tree)	60.4	[6]
Pinus bark powder	32.8	[26]
Jute fiber carbon	27.99	[39]
Wood apple	19.8	[40]
De-oil soya	5.79	[7]
Sugarcane dust	3.8	[41]
Neem sawdust	3.8	[43]
Palm kernel fiber	78.9	[44]
Potato peel	555	This study

It is observed that the adsorbent in this study had q_{max} value which is better than some previously reported biomass residue adsorbents in literature, hence there is potential for the exploitation of the potato peel adsorbent for dye remediation of polluted aqueous systems.

Table 3. Adsorption isotherm constants for adsorption of CV onto potato peel.

Langmuir isotherm parameters			Freundlich isotherm parameters		
q_m (mg.g^{-1})	K_L (L.mg^{-1})	R^2	K_F (mg.g^{-1}) (L.mg^{-1}) ^{1/n}	n	R^2
555	0.128	0.995	23.916	1.163	0.987

The value of the Freundlich isotherm constant “n” which describes the adsorption intensity of the adsorbent also shown in Table 3. This value was 1.163 and indicates that the adsorption of Crystal violet by the PP adsorbent was favorable. According to Sarada et al. [44] a Freundlich isotherm constant “n” value between 1 and 10 indicates that the adsorption intensity is favorable. Furthermore, the value of the Freundlich constant “ K_F ” which relates to the adsorption capacity for a multilayer system is also presented in Table 2. The value of K_F obtained for the PP adsorbent was $23.91 (\text{mg.g}^{-1}) (\text{L.mg}^{-1})^{1/n}$, thus indicating that the multilayer loading capacity of the PP adsorbent was low.

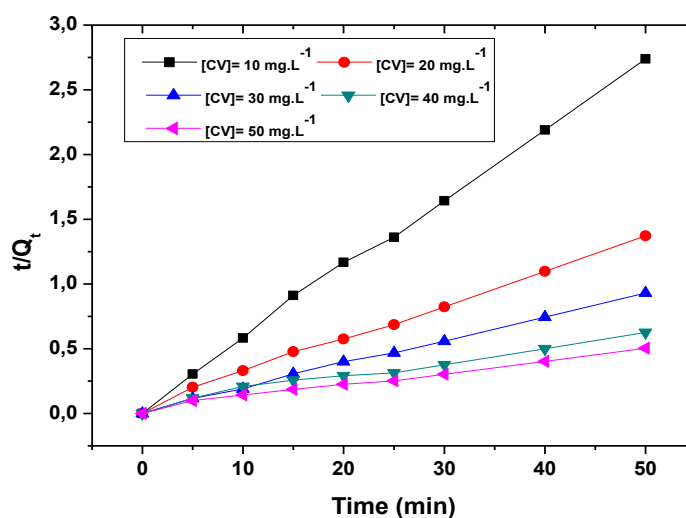
3.4 Adsorption kinetics

Adsorption kinetics of Crystal violet onto PP was modelled by pseudo-first order (Eq. (6)) and pseudo-second order (Eq. (7)) and the experiments were conducted at initial CV concentrations ranges from 10 to 50 mg.L^{-1} by varying contact time from 5 to 60 min. The values of pseudo-first-order rate constants, k_1 and q_e were calculated from slopes and intercepts of the plots of $\log(q_e - q_t)$ versus t and are listed in Table 4.

Table 4. Kinetic parameters for adsorption of CV onto potato peel.

$q_{e, \text{exp}} (\text{mg} \cdot \text{g}^{-1})$	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
	$q_{e, \text{cal}} (\text{mg} \cdot \text{g}^{-1})$	$k_1 (\text{min}^{-1})$	R^2	$q_{e, \text{cal}} (\text{mg} \cdot \text{g}^{-1})$	$K_2 (\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1})$	R^2
36.451	30.157	0.139	0.954	37.87	0.014	0.996

The experimental data for the removal of CV when analyzed on the pseudo-first-order equation did not result in a perfect straight line when a plot was drawn between $\ln(q_e - q_t)$ and t indicating a non-fit on the model. The data showed a better fit to Pseudo-second-order model R^2 (0.9976) which can be further confirmed by the excellent accordance between the calculated q_e value and the experimental results.

**Fig 7.** Pseudo-first order and pseudo-second order kinetic plot for the adsorption of the CV on PP.

The best correlation for the system provided by the pseudo-second-order model suggests that chemical adsorption involving valency forces through sharing or exchange of electrons between adsorbent and adsorbate might be significant [10].

Saeed et al. [28], El Sayed [44], Ahmad [26] reported that CV dye adsorption on grapefruit peel, palm kernel fiber and coniferous pinus bark powder fitted well to pseudo-second order kinetic model, and the process was thought to be chemisorption controlled.

4. Conclusions

In this work, an experimental study on the utilization of potato peel (PP), an agricultural residue for the removal of Crystal violet from aqueous solution was investigated. The experimental results showed that operating parameters such as contact time, pH, PP dose and initial CV concentration greatly affected the adsorption capacity of CV on the PP. The adsorption capacity of CV ($\text{mg} \cdot \text{g}^{-1}$) increased with increase in initial dye concentration, time and pH and the optimized pH for adsorption was 6. The kinetics of CV removal indicated an optimum contact time of 20 min via a two stage of adsorption kinetic profile (initial fast and subsequent slow equilibrium). Adsorption isotherm data was best fitted with Langmuir model, this indicates that the monolayer Adsorption capacity of the PP adsorbent was $555 (\text{mg} \cdot \text{g}^{-1})$, and a Langmuir constant of $0.128 (\text{L} \cdot \text{mg}^{-1})$ that the adsorbent was effective for CV removal from an aqueous system. Adsorption kinetics of CV removal by PP follows pseudo-second order model. Results of this work suggest that the potato peel (*Solanum tuberosum*) may be promising low cost adsorbent for the removal of CV from wastewater. Therefore, it can be further examined and considered as a cheaper alternative to commercial adsorbents.

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