

Article

Efficient Decolourisation of Astrazon Pink Dye Using Biocarbon Derived from *Prosopis juliflora* Shells: Kinetics, Isotherms, and RSM-Based Optimization for Sustainable Wastewater Treatment

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Abstract

This study investigates the efficiency of biocarbon derived from *Prosopis juliflora* shells in removing Astrazon pink dye from aqueous solutions. The biocarbon obtained from the thermochemical process was characterised using FTIR Spectroscopy, SEM microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS), and XRD. Batch adsorption experiments were conducted to assess various factors, including the Potential of Hydrogen (pH), Dosage of biocarbon, Astrazon pink dye concentration, temperature, and Time of contact. Similarly, Adsorption isotherm models, including the Langmuir and the Freundlich isotherms, were used to evaluate the adsorption capacity. In contrast, pseudo-first-order and pseudo-second-order models were used to analyse the kinetics of dye adsorption. The interactive effects of selected variables on the removal of Astrazon Pink dye from synthetic water were determined using Response Surface Methodology (RSM). The maximum dye uptake, 98.54%, was achieved with a biochar dose of 8 g/L at 50 ppm dye concentration, pH 7.5, and 35 °C. The Freundlich adsorption isotherm model and the pseudo-second-order kinetic model are the better-fitting models for the dye adsorption process, with R² values of 0.99. Consequently, the thermodynamic parameters indicate that the process is endothermic and spontaneous.

Keywords: *Prosopis juliflora* shell-derived biocarbon; Astrazon pink; cationic dyes; adsorption; isotherms and kinetics; thermodynamics; response surface methodology

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1. Introduction

Water contamination is a significant environmental issue that affects both human health and the environment globally [1]. On the other hand, rapid industrialisation leads to massive discharge of hazardous chemicals into the ecosystem. Discharging organic and inorganic pollutants untreated into natural water streams creates serious environmental issues [2]. Several industries, such as paper and pulp, textiles, tannery, paint, edible products, pharmaceuticals, cosmetics, and others, utilise coloured dye compounds in diverse applications [3–5]. Almost all dye compounds are water-soluble and hazardous to humans, aquatic organisms, and crop plants [6]. The small amount of dye dissolved in the water stream can act as a barrier to solar radiation, preventing it from penetrating the

stream and reducing photosynthetic activity, which can damage aquatic life [7]. It also disrupts food chain activities, harming humans and other organisms by dissolving them without treatment, and producing carcinogenic, mutagenic, and teratogenic effects [8]. Removing dye residues from the water stream was achieved using techniques such as photo-oxidative degradation, phytoremediation, glomerular filtration, and adsorption [9–11]. Among the various techniques, adsorption is the most basic and economical method for removing dyes, thanks to the adsorbent's regeneration capacity, which allows repeated use. Additionally, the large volume of wastewater with no harmful residues will result in an efficient adsorption process [12]. In the past decades, various investigational research has been carried out for the degradation of colorants from waste streams using different adsorbent materials like Silicon dioxide [13], alkoxide [14], activated charcoal [15], horticultural charcoal [16,17], graphite oxide [18], montmorillonite clay [19], coal ash [20], etc. Recently, biochar has shown promise as an economical adsorbent compared to other commercial adsorbents. Biochar is a high-carbon, granular residue obtained from the pyrolysis process of various organic biomass materials [21,22]. Over the past few decades, the use of biochar for adsorption has increased significantly due to its economic benefits, high removal efficiency, and greater biomass availability [23–25]. The significant merit of biochar is that it contains a larger surface area and high functionalities, which play an essential role in dye removal efficiency. Functional groups and organic pollutants interact through physical adsorption, the hydrophobic effect, electrophilic interactions, hydrogen bonding, and pi-pi interactions [26], thereby separating them from the aqueous solution. Various reports suggest using biofuel, agricultural waste, and green biocarbon to eliminate colorants from waste streams. Renewable organic materials like plantains [27], corn [28], waste coconut shells [29], bamboo culms [30], turmeric leaf waste [31], mango leaves [32], macauba palm [33] fruit peel biochar [34], *Acacia leucophloea* wood sawdust [35], sugar-beet-pulp [36], oak wood biochar [37], coir [38], Karanja oil seed cakes [39], are used for dye removal in the water stream. Hence, numerous possibilities exist for studying the economic production and application of biochar in removing colors from sewage. *Prosopis juliflora* shell is a naturally available waste obtained from the pyrolysis process. The study reports the removal of the methyl orange colorant using *Prosopis juliflora* biochar, achieving 64% removal efficiency [40]. The biochar derived from *Prosopis juliflora* removes acid blue dye with a removal efficiency of 69% [41]. The report shows the decolorisation of Swiss blue and methyl violet from rice straw biochar, achieving removal efficiencies of 94.45% and 92.70%, respectively [42]. The primary objectives of the current research work are to focus on sustainable wastewater treatment and the efficient removal of Astrazon Pink using biocarbon derived from *Prosopis juliflora* shells: kinetic and isotherm analysis and RSM optimisation.

2. Materials and Methods

The chemicals used for the experimental analysis were purchased from Sigma-Aldrich Private Limited, Bengaluru, India. *Prosopis juliflora* seed shell residue was obtained from the local area in and around the Vellore district, Tamil Nadu. It is used as a biomass feedstock for biochar production. The biomass was cleaned using running water to remove dirt and dust, dried for 2 days at 70 °C, and crushed to less <100 mm [42]. The synthetic water was prepared by adding the required amount of Astrazon pink to deionised water and used as a synthetic solution [43].

2.1. Preparation of Biochar

The *Prosopis juliflora* biomass was converted into biochar (biocarbon) via a thermochemical process in a muffle furnace at 350, 400, 450, 500, and 550 °C, with a constant heating rate, maintained for 30 min [44–46]. The biochar was cooled, ground, sieved, and

placed in a desiccator for further processing, including adsorption and characterisation studies. The biochar quantity (%) after the thermochemical process was calculated from Equation (1).

$$\text{Biochar yield} = \frac{\text{Mass of Biochar}}{\text{Mass of Biomass}} \times 100\% \quad (1)$$

2.2. Batch Study

Initially, a synthetic stock solution of Astrazon pink (1 g/L) was prepared in deionised water and diluted to the desired concentration. In a 250 mL Titration flask containing the required dye concentration, a specific biochar dosage was added to determine the dye removal efficiency in a synthetic water sample. The flask is tightly packed and sealed, and it is kept in a rotatory shaker at 250 revolutions per minute. To optimise the removal efficacy of dye, different parameters are varied: Initial concentration of dye (10–100 mgL⁻¹), contact time (10–100 min), temperature (25–55 °C), and solution pH (2–8), Dosage of biochar (1–10 gL⁻¹). After adsorption, the prepared stock was centrifuged for 15 min at 4000 r/min. After centrifugation, the supernatant was used to determine the dye concentration by UV-visible spectroscopy at 254 nm. The adsorption capacity of the dye at equilibrium was determined using Equation (2).

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

where Q_e = adsorption of dye by adsorbent (mg g⁻¹), V = Volume of dye solution (L), C_o = Initial dye Concentration (mg L⁻¹), C_e = Final concentration of dye (mg L⁻¹), W = Weight of Biochar (g).

2.3. Instrumental Analysis

The porosity and specific surface area of the pre- and post-treated biochar were identified using Scanning Electron Microscopy with EDX (a Jeol 6390LA/OXFORD XMX N, JOEL, New Delhi, India, FT-IR analysis (Thermo Nicolet iS50, 4000 cm⁻¹ to 500 cm⁻¹), and X-ray Diffraction (Bruker D8 Advance brand). The zero-point charge (pzc) of biochar is determined by titration in a series of 100 mL conical flasks using a 0.1 mol L⁻¹ KNO₃ solution [46]. In addition, the zero-point charge of biochar (pzc) is determined using the salt titration method, conducted in a series of 100 mL conical flasks, with a 0.1 mol L⁻¹ KNO₃ solution [46].

2.4. Isotherm and Kinetics Adsorption Studies

Two distinct isotherm models, the Freundlich (Equation (3)) and Langmuir (Equation (4)) models, were analysed for the adsorption of dye isotherm data:

$$\text{Freundlich model: } \log q_e = \log k_f + \frac{1}{n} \log C_e \quad (3)$$

where q_e represents the adsorption of dye uptake (mg g⁻¹), K_F is the Freundlich constant, C_e is the Concentration of dye at the equilibrium state (mg L⁻¹), and $1/n$ is the heterogeneity factor calculated using the slope and intercept from the graph of $\log q_e$ vs. $\log C_e$.

$$\text{Langmuir model: } \frac{1}{q_e} = \frac{1}{K_L q_{\max}} \cdot \frac{1}{C_e} + \frac{1}{q_{\max}} \quad (4)$$

where q_e represents the equilibrium dye adsorption (mg/g), $q_{\max}$ represents the maximum dye adsorption (mg/g), C_e represents the sorbate absorption at equilibrium (mg/L), and K_L is the Langmuir constant. Both the Pseudo-1st-order Equation (5) and the pseudo-2nd-order model Equation (6) illustrate the sorption kinetics as shown below:

$$\text{Pseudo-1st-order kinetics } \ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

Pseudo-2nd-order kinetics

$$\frac{t}{q_e} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (6)$$

where q_t is the dye adsorption capacity at time t (mg g^{-1}), q_e is the equilibrium sorption dye capacity (mg g^{-1}), and k_1 (min^{-1}) and k_2 ($\text{g mg}^{-1} \text{min}$) are the rate constants of the Pseudo-1st-order and -2nd-order models, respectively.

2.5. Thermodynamic Study

A thermodynamic study was used to analyse the equilibrium data. Gibb's energy (ΔG°), Enthalpy reaction (ΔH°), and change in entropy (ΔS°) are found using Equations (7) and (8) [47,48]

$$\log \frac{q_e}{C_e} = \frac{\Delta S^\circ}{2 \cdot 303R} + \frac{\Delta H^\circ}{2 \cdot 303RT} \quad (7)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (8)$$

In K_L vs. $1/T$ linear plot, the Van't Hoff equation. Where q_e represents the Equilibrium concentration at the solid phase (mgL^{-1}), C_e is the Equilibrium concentration (mgL^{-1}), T represents the Temperature (symbol $^\circ \text{K}$, R is the Universal Gas Constant (8.314 J/mol K), ΔH° is the Enthalpy change, ΔS° is the change in entropy.

2.6. Full Factorial Design of Optimisation of Astrazon Pink (FG) Dye Removal

We conducted a statistical optimization of Astrazon pink using a CCD to eliminate dye and investigate the impact of predictor variables on the adsorption process. Four parameters were considered independent variables: pH (A), Contact time (B), Dye Concentration (C), and Biochar dose (D). These variables were used to determine the optimal conditions, as shown in Table 1.

Table 1. Range of independent variables for different adsorption parameters.

Parameters	Independent Variables	Units	Levels		
			Coded Low (−1)	Mean	Coded High (+1)
pH	A	-	2	7	13
Contact time	B	min	10	90	170
Dye Concentration	C	mg/L	10	50	90
Biochar dose	D	g/L	1	8	15

The Box–Wilson Central Composite design involves a factorial run of $2n$, an axial run of $2n$, and a center point with 6 runs. The center points indicate the produced date and possible experimental error. The axial points are selected to minimize model-prediction variance and ensure repeatability, and are constant at the points nearest to the center of the design [49]. The total runs conducted through the experiment are determined by the following Equation (9) [50].

$$N = 2^n + 2n + n_c \quad (9)$$

$$N = 2^4 + 2(4) + 6 = 30 \text{ runs}$$

where n is the number of input variables (Independent variables), n_c is the number of center points, and N indicates the total number of runs for the analysis of experiments. This suggests that 30 runs, comprising 16 factorial runs, 8 axial runs, and 6 center runs, were required for the modelling and optimisation process. The experimental data

obtained were studied using State Ease 360 software version 23.1.8. The various parameters and their coded values for Astrazon Pink (FG) adsorption capacity are listed in Table 1. To explore the optimum conditions for the adsorption of Astrazon Pink (FG) from wastewater using an unaltered *Prosopis juliflora* biochar seed shell. The Independent variable (response) and the uptake capacity of Astrazon Pink (FG) are determined through batch experiments using a statistical model tool. The ideal pattern illustrates the predicted adsorption percentage response (Y) to the four independent variables, as represented by a 2nd-order polynomial expression in Equation (10) [51,52].

$$Y = x_0 + \sum_{i=1}^n x_i x_i + \sum_{i=1}^n x_{ii} x_i^2 + \sum_{i=1}^n \sum_{j+1}^n x_{ij} x_i x_j + \epsilon \quad (30)$$

where Y is the adsorption percentage predicted response; x_0 = constant; x_i = linear coefficient; x_{ii} = Squared coefficient; x_{ij} = cross product coefficient; x_i and x_j = independent factor; ϵ = random variation.

3. Results

3.1. Instrumental Analysis

3.1.1. SEM with EDX

A scanning electron microscope identified the morphological surface of the *Prosopis juliflora* shell biochar pre- and post-treated adsorption. Figure 1a,b. implies the image of biochar before and after adsorption. We crushed the raw *Prosopis juliflora* shell and thermally degraded it via thermochemical process to study wear and tear, which broke down the raw shell and created several pores, as seen in Figure 1a. Figure 1b represents the desirable variations that have occurred. It results from the bonding of anions to the biochar surface, which becomes smooth after adsorption. The SEM images after sorption show a heterogeneous, smooth surface, with the dye present in aqueous solution, which facilitates the adsorption of Astrazon pink. We also conducted Energy-Dispersive X-ray Spectroscopy (EDX) to determine the chemical composition of biocarbon before and after treatment. Figure 2a,b show the EDS spectra of the biochar before and after adsorption. The result revealed that the raw black carbon composition consists of the major constituents carbon (C, 68.6%) and oxygen (O₂, 27.3%). The dehydration of hydroxy and aliphatic groups induces decarboxylation, thereby accelerating thermal decomposition. The higher carbon content (72.7%) after adsorption shows the adsorption of Astrazon pink dye.

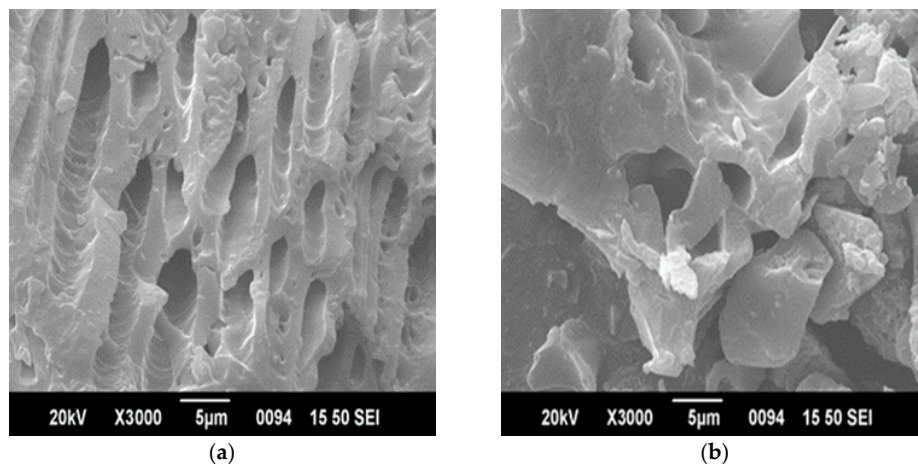


Figure 1. Scanning Electron Micrograph images of raw *Prosopis juliflora* biochar shell pre-adsorption (a) and post-adsorption (b).

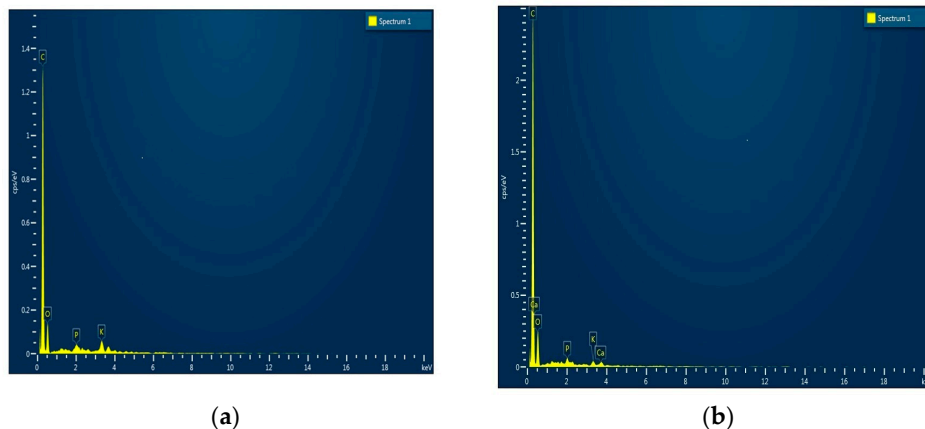


Figure 2. EDX of *Prosopis juliflora* black carbon shell pre-treated (a) and post-treated (b).

3.1.2. FT- IR Spectrum

FT-IR spectrum of a *Prosopis juliflora* biochar before and after treatment, as shown in Figure 3. The biochar showed a strong band of 729 (=Carbon-Hydrogen bond bend) designated alkene group bend, 1171 (C-H bend) designated primary alcohol stretch bond, 1592 (C=C Stretch, N-H bend) reflects the skeletal stretching indicates the (-OH), i.e., hydroxyl functional groups as it comes to dye bond, 2162 (C=C stretch) reflects alkynes stretching bond, 2930 (C-H stretch) alkanes and alkyls of stretching of a new bond between the dye molecule and the biochar, respectively, 3383 (O-H, N-H) shows the stretch bond of -OH groups. This variation indicates the absorption of Astrazon pink dye. Thus, the FTIR spectra of *Prosopis juliflora* shell pre-adsorption suggest that the dye exterior surface contains different functional groups due to electrostatic interactions [53,54].

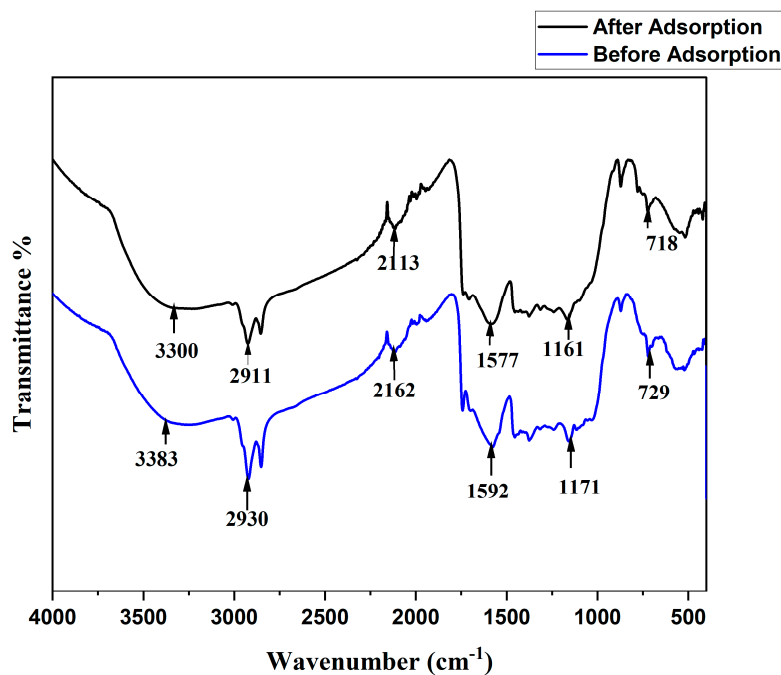


Figure 3. FT-IR of *Prosopis juliflora* biochar pre-Astrazon pink adsorption and post-Astrazon pink adsorption.

3.1.3. X-Ray Diffraction Analysis

The X-ray diffraction patterns of the sample are presented in Figure 4. The results reveal that these *Prosopis juliflora* shell biochars contained amorphous silica with its featureless diffractograms at a diffuse appearance at $2\theta = 20.332^\circ$ before adsorption and 24.243° after adsorption, indicating the existence of amorphous SiO_2 in it. It detected that the measurement range of (0 to 23°) of the 2θ scale of this amorphous phase suggests the occurrence of a halo [55]. The analysis reveals that the *Prosopis juliflora* shell biochar sample comprises a mixture of crystalline (002) and amorphous (100) phases. We examined the crystalline size and structure by detecting sharp curves as shown in Figure 4. The unidentified peak in the diffraction pattern indicates that a small amount of other minerals is present in the biochar formed during slow pyrolysis at low temperature [56]. Time changes, and reaction time increases. We observed remarkable changes in the frequency of the X-ray peaks, with the diffraction peak becoming stronger. The sharp and strong X-ray diffraction phase ($20\text{--}34^\circ$) of the 2θ scale.

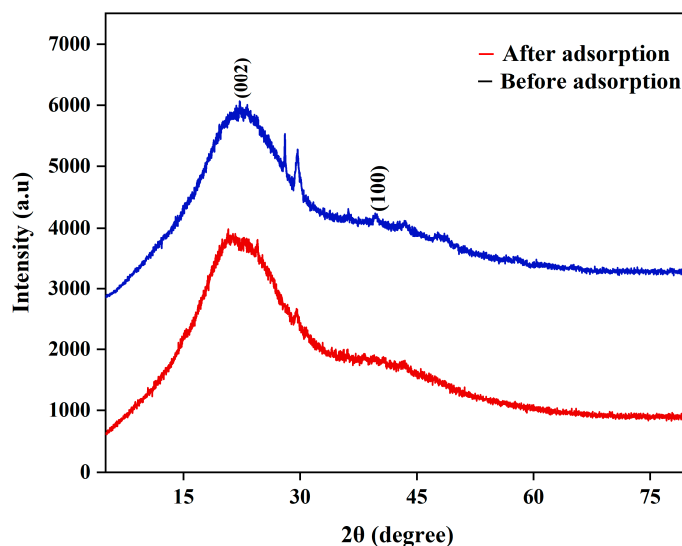


Figure 4. XRD spectra of *Prosopis juliflora* biochar before and after adsorption of Astrazon pink.

3.2. Batch Adsorption Study

3.2.1. Effect of Dosage of Biochar

The dosage of biochar is a crucial factor in influencing the dye adsorption capacity. Figure 5 illustrates the impact of biochar dosage on the percentage of dye removal. We examined the adsorption of Astrazon pink at different biochar dosages (1–10 g/L) and a dye concentration of 50 mg/L, at 35 °C. The study shows that increasing the dose of *Prosopis juliflora* biochar increases Astrazon pink removal percentage from 35.02% to 98.54%. The adsorption capacity differed from the removal rate: it first increased and then decreased with increasing adsorbent dosage from 1 to 10 g/L, reaching a maximum. This adsorption is attributed to the greater accessibility of biochar's more active sites. Based on the sorbent dosage, the dye removal efficiency was enhanced due to the availability of active sites [57]. A previous study reported that methyl violet 10B was removed to 86.4% by biochar derived from Oil Palm Kernel Shells [58]. Furthermore, increasing the addition of *Prosopis juliflora* biochar beyond an optimal contact time of 90 min would concurrently reduce sorbent capacity by decreasing the total surface area and increasing the diffusive pathway length [59]. The optimum *Prosopis juliflora* biochar dose is 8 g L^{−1}, as determined by the investigation of cationic dyes with a pH of 7.5 to dye concentration of 50 mg/L.

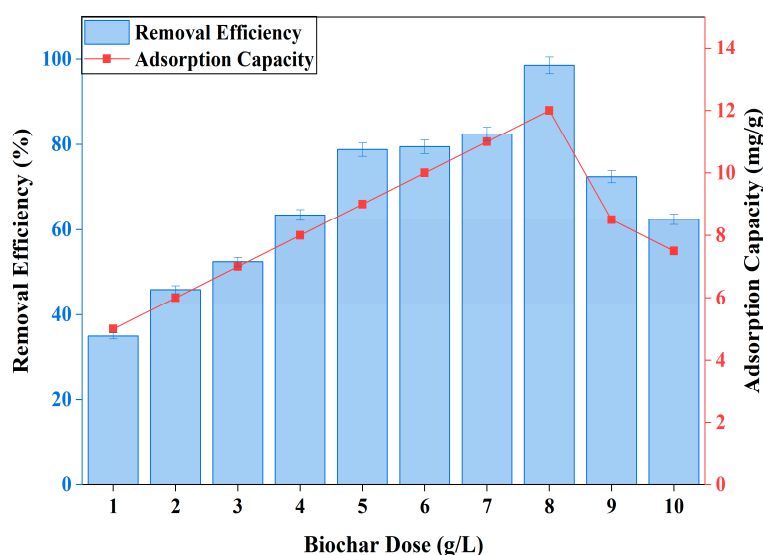


Figure 5. Effect of Biochar dose on the Removal of Astrazon pink.

3.2.2. Effect of Concentration of Dye

Figure 6 shows that the dyestuff concentration plays a crucial role in eliminating colourants, as demonstrated in batch studies. The dye dosage for the batch studies was varied from 10 to 100 mg L⁻¹ under sustained conditions: pH 7.5, temperature 35 °C, biochar dosage 8 g L⁻¹, and Contact time 90 min. The biochar resulted in a higher dye removal percentage (98.54%) compared to the results. The bar graph showed that the number of active sites on the adsorbent increased gradually with increasing initial dye concentration. The adsorption capacity differed from the removal rate: it first increased and then decreased with increasing dye concentration from 10 to 100 mg/L, reaching a maximum. The accumulation of dye molecules decreases the removal percentage at those active sites. After using 50 mg L⁻¹, the removal efficiency decreased considerably; therefore, we selected this concentration as optimal for Astrazon pink on *Prosopis juliflora* Biochar.

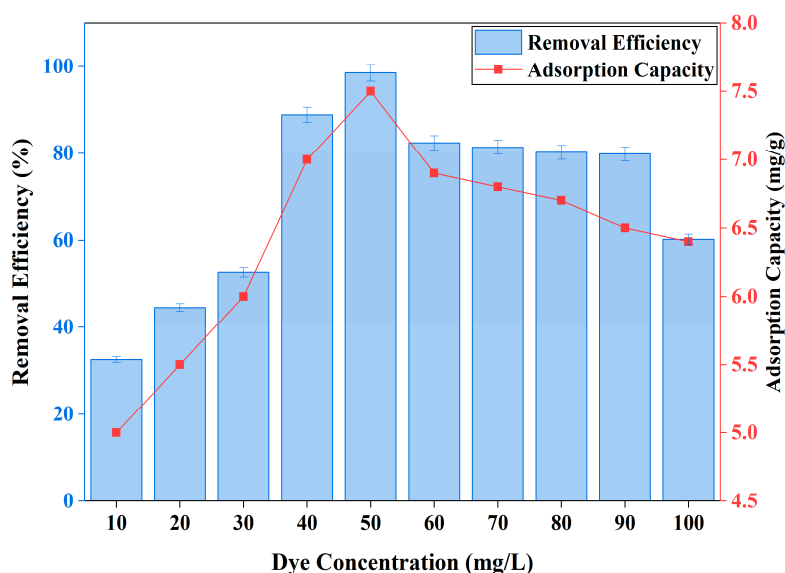


Figure 6. Effect of Initial dye concentration on the Removal of Astrazon pink.

3.2.3. Effect of Time of Contact

The effect of contact time on colorant removal efficiency was investigated in a series of batch studies under sustained conditions: 35 °C, 8 gL⁻¹ Biochar, pH 7.5, and an initial dye concentration of 50 mgL⁻¹. The contact time graph is shown in Figure 7. The results indicate that increasing the dye exposure time to the sorbent increases the percentage of dye removal. The maximum removal is 98.54% at 90 min for *Prosopis juliflora* biochar. After 90 min, there has been no significant increase in colorant removal efficiency. The adsorption capacity differed from the removal rate: it first increased and then decreased with increasing adsorbent dosage from 10 to 100 min, reaching a maximum. At 100 min, the removal efficiency decreases to half the earlier value due to the unattainability of active sites and prolonged exposure to the sorbent and colorant.

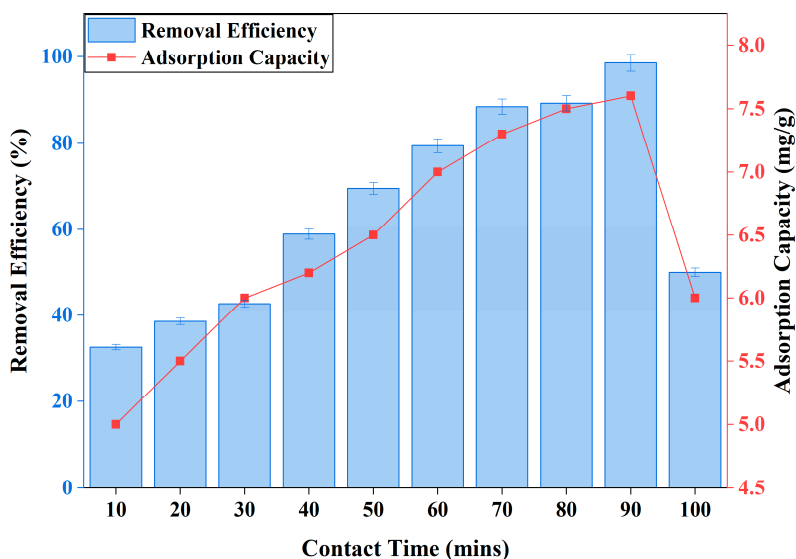


Figure 7. Effect of Time of Contact on the Removal of Astrazon Pink.

3.2.4. Effect of pH

The probable role of the functional groups in dye solution adsorption determines the equilibrium pH. Batch studies were performed to examine the pH sensitivity of biocarbon for the elimination of dye under conditions of 35 °C, 8 g/L Biochar dose, 90 min contact time, and 50 mg/L initial dye concentration. The effects demonstrate that the elimination of colorant at varying pH concentrations (4 to 8) is shown in Figure 8. The graph shows that the maximum elimination of the dye occurred between pH 7 and 7.5. The acid condition (pH 4) and the dye removal efficiency by biochar were 59.54% and 59.54%, respectively. The dye removal efficiency increased from 59.54% to 98.54% at pH 7.5. However, under alkaline conditions, the removal efficiency decreased slightly to 75% and 58%, respectively. This study's alkaline condition decreased Astrazon pink dye removal efficiency to 62.56% from 98.54%. The outcomes indicate that the elimination of dye at lower pH is attributed to its electrostatic attraction between the adsorbent and the dye molecule. (Positively charged ions and cations). The biochar is protonated with excess hydrogen ions, which are positively charged due to its lignocellulosic nature, comprising carboxylic acids, SO₂, and organic amide groups at low pH. The H⁺ ions will attract cations, including the reactive dye molecule, enhancing the biochar's adsorption capacity [60]. The adsorption capacity differed from the removal rate: it first increased and then decreased with pH from 2 to 8, reaching a maximum.

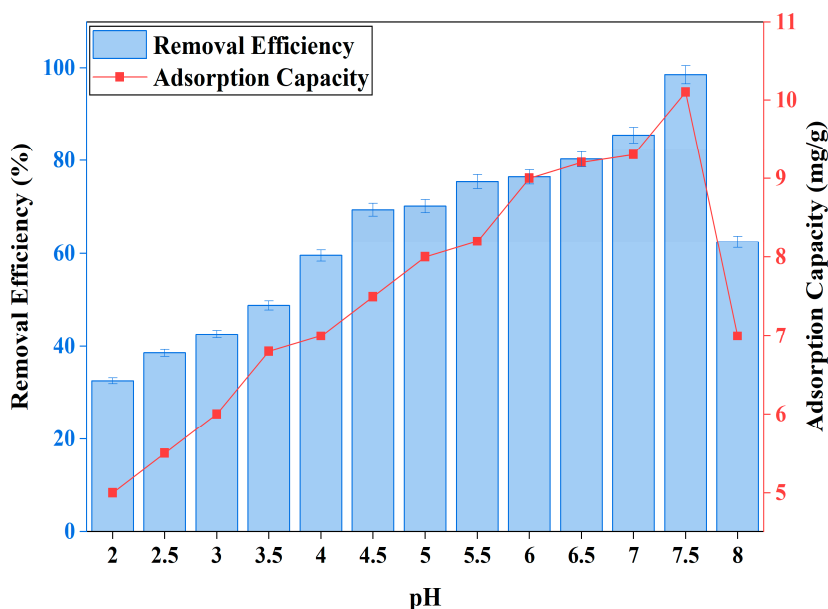


Figure 8. Effect of pH on the Removal of Astrazon Pink.

The solution's pH is greater than 7.5. The protonated H^+ bound to the biochar will significantly reduce the attraction of cations, resulting in a decrease in biochar adsorption capacity [61]. Hence, the optimal pH is 7.5. The zero-point charge (ZPC) of biochar is another reason to examine the adsorption behaviour of a cationic dye. The pHZPC of the biochar is 7.0. These results reveal that when the pH of the dye solution exceeds the pHZPC, the adsorbent surface becomes negatively charged, which favours the adsorption of Astrazon pink dye via electrostatic attraction. This is for the removal of Astrazon pink dye onto the *Prosopis juliflora* biochar.

3.2.4. Effect of Temperature

The endothermic reaction can occur as the adsorption capacity increases gradually with increasing temperature, favored by chemisorption. Likewise, as the temperature rises, the interatomic forces between the solvent and the adsorbent are high, and their nature is favourable to dye adsorption [62]. Simultaneously, as temperature increases, the biochar's adsorption capacity decreases. It acts as an adsorption and exothermic reaction, which is favored by physisorption [63]. In the batch studies, three factors vary, while the remaining factors are held constant to estimate optimal parameters, including biochar dosage (8 g/L), contact time (90 min), pH (7.5), and initial dye concentration (50 mg/L). The highest elimination percentage was 98.54% at an optimum temperature of 35 °C for *Prosopis juliflora* biochar, as represented in Figure 9. Beyond 35 °C, there is no significant adsorption of the dye, and the percentage is reduced to 82.45%. This is due to the bond between the biochar and the dye solvent breaking under high temperatures. The results indicate that biochar is ineffective at higher movement rates due to its electrostatic interactions with molecules. Hence, the results show an optimum temperature of 35 °C. The adsorption capacity differed from the removal rate: it first increased and then decreased with temperature ranging from 25 to 55 °C, reaching a maximum.

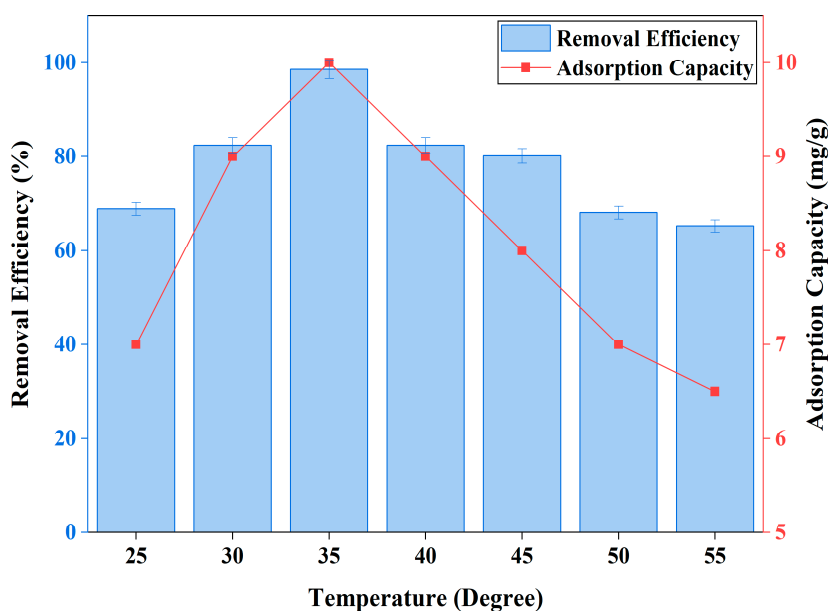


Figure 9. Effect of temperature on the removal of Astrazon pink dye.

3.3. Adsorption Kinetics

We determined the impact of contact time on the adsorption of Astrazon pink dye for the first few hours using *Prosopis juliflora* biochar. Observations showed rapid uptake at the exposure rate, followed by steady achievement in balance. During the rapid uptake period, approximately 80% of Astrazon pink adsorption capacity in *Prosopis juliflora* biochar is at equilibrium. The adsorption illustration was conducted over a short period, likely during the initial phase. Simultaneously in the rapid stage of adsorption, slower achievements were made at this stage the concentration of dye should not improve substantially because the dye molecules had already been used at the accessible sites, and this was additionally probed as Pseudo-first-order (Equation (6)) and Pseudo-second-order (Equation (7)) model was determinate to identify the kinetics of Astrazon pink adsorption (Table 2). The figure of Kinetic Curves shows the maximum biochar adsorption for Astrazon pink at 23.7473 mg/g, as shown in Figures 10 and 11. The kinetic data clearly indicate that the dyes provide a better estimate, with the lowest error percentage and a correlation coefficient above 0.99. The standard error of regression is 8.05312×10^{-4} . While analysing both models, the pseudo-second-order model fits well for comparing the experiment's predicted and uptake values.

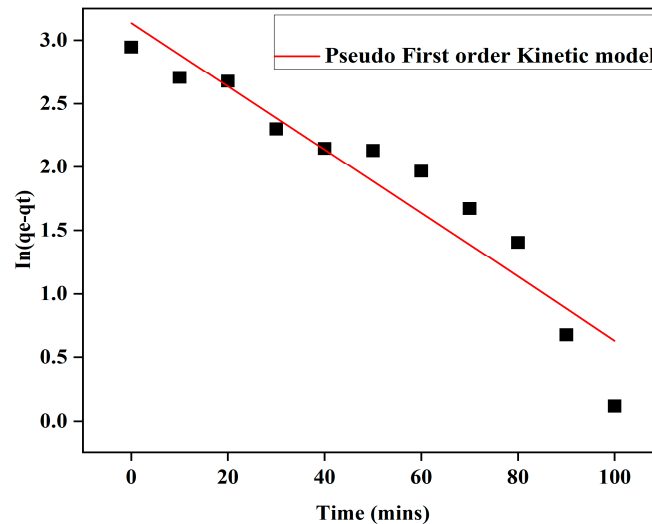


Figure 10. Pseudo-first-order kinetics of Astrazon pink onto *Prosopis juliflora* Biochar.

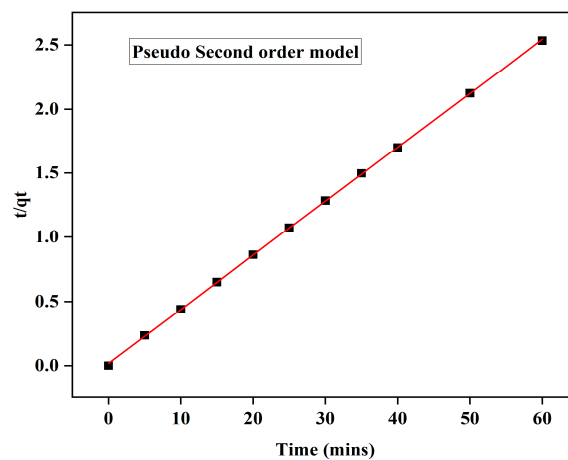


Figure 11. Pseudo-2nd-order kinetics of Astrazon pink onto *Prosopis juliflora* shell biochar.

Table 2. Kinetic values calculated for pseudo-1st- and 2nd-order models during the adsorption of Astrazon pink on *Prosopis juliflora*.

Model	Parameters			
Pseudo-first order	q_e (mg/g)	k_1 (min^{-1})	R^2	Standard Errors of Regression
	23.0245	0.025	0.8964	
Pseudo-second order	q_e (mg/g)	k_2 (g/mg) min^{-1}	R^2	
	23.7473	0.1008	0.9999	
				8.05312×10^{-4}

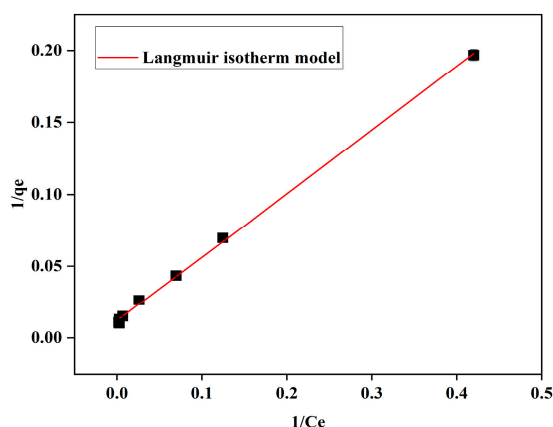
3.4. Isotherm of Adsorption

Astrazon pink dye was analyzed with *Prosopis juliflora* biochar using an initial dye concentration solution. The adsorption and elimination of the dye are significantly increased as the initial dye concentration increases from 10 to 50 mg/L. The adsorption of the dye showed a linear correlation. With a further increase in the dye concentration, the adsorption rate decreased. The improved sorption potential with a high initial concentration may be due to a greater driving force between the liquid and solid phases of the dye and that of the *Prosopis juliflora* shell biochar, as shown in Figure 12a,b. The adsorption

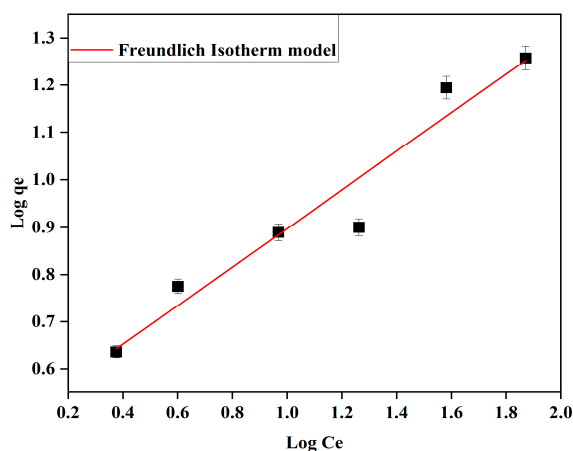
data are analyzed using two isotherm models, namely the Freundlich and Langmuir models, for the adsorption of the dye solution onto the biochar. Equations (3) and (4) denote the Langmuir and the Freundlich models, which were selected to fit the isotherms, as shown in Figure 12. The Langmuir model exhibits a correlation coefficient (R^2) greater than 0.9987, compared to the Freundlich model of Astrazon pink adsorption onto *Prosopis juliflora* shell Biochar. This happens primarily due to the monolayer adsorption of homogeneous materials [64], and the maximum adsorption uptake of *Prosopis juliflora* shell biocarbon monolayers ($Q_{\max}$) was 84.175 mg/g, as shown in Table 3. This result indicates that the condition is highly favourable for dye adsorption, making the adsorbent highly effective at the temperature during the pyrolysis process for biochar preparation.

Table 3. Adsorption Capacities of different adsorbents and *Prosopis juliflora*.

Dye	Adsorbent	$Q_{\max}$ (mg g ⁻¹)	References
Astrazon Pink	<i>Prosopis juliflora</i>	84.175	This study
Methylene blue	<i>Prosopis juliflora</i> activated with citric acid	37.6	[65]
Methylene orange	<i>Prosopis juliflora</i>	8.31	[66]
Maxilon Blue dye	<i>Prosopis juliflora</i> fruit seeds	84.54	[67]
Cu (II)	Date palm leaf waste	70	[68]



(a)



(b)

Figure 12. Experimental and Predicted uptake of Langmuir (a) and Freundlich (b) models for Astrazon pink onto *Prosopis juliflora* Biochar.

3.5. Thermodynamics Study

The thermodynamic parameters were analyzed using the free enthalpy (ΔG° , kJ mol⁻¹), reaction enthalpy (ΔH° , kJ mol⁻¹), and changes in entropy (ΔS° , J mol⁻¹ K⁻¹), which were calculated using the following equations. The combined Equations (8) and (9) are used to determine the value of ΔG . The thermodynamic results are shown in Table 4 and in Figure 13. The R² value of 0.99, with a standard regression error of 2.10869×10^{-5} , indicates a linear fit, suggesting that the ΔH° and ΔS° values calculated for biochar are reliable. Furthermore, the magnitude was consistent with that expected for physical adsorption of biochar [69]. The magnitude of the enthalpy change can be classified according to the type of interaction. The Gibbs energy is negative, indicating that the adsorption process will proceed spontaneously and is exergonic. The positive enthalpy change confirmed the endergonic nature of adsorption. The positive entropy value suggests an increase in randomness at the solid-solution interfaces during the adsorption of adsorbates on the active sites of sorbents.

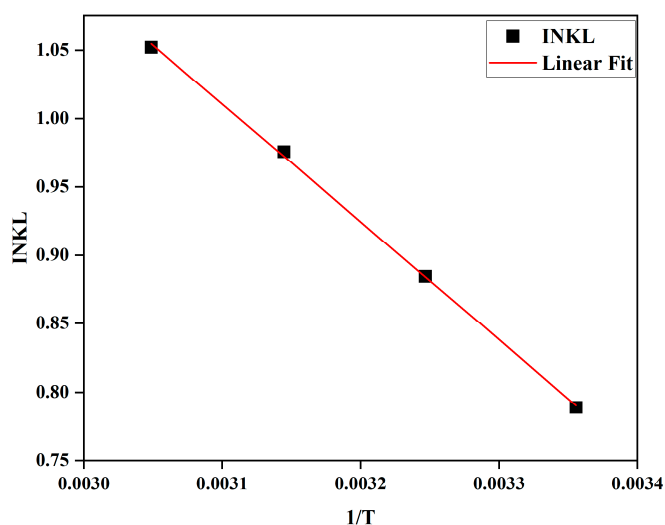


Figure 13. Thermodynamic effect of *Prosopis juliflora* Biochar on Astrazon Pink.

Table 4. Thermodynamic value for *Prosopis juliflora* Biochar onto Astrazon Pink dye.

Adsorbent	Temp (k)	KL	Regression of Standard Errors	ΔG° (kJmol ⁻¹)	ΔH° (kJmol ⁻¹)	ΔS° (Jk ⁻¹ mol ⁻¹)	R ²
<i>Prosopis juliflora</i> shell biochar	298	2.201	2.10869×10^{-5}	-1.9545	7.1651	30.6142	0.999
	308	2.422		-2.2651			
	318	2.653		-2.2579			
	328	2.864		-2.8693			

3.6. Optimisation of Astrazon Pink Dye Removal Using *Prosopis juliflora* Biochar Using Central Composite Design

The design matrix for a 4-factor experiment generated by RSM software version 3.0.0, using batch adsorption studies of Astrazon Pink on *Prosopis juliflora* biochar, is summarised in Table 5. A model that relates the adsorption capacity to the variables selected by Central Composite Design is termed a quadratic model (Tables 5 and 6) and is expressed in terms of the coding parameters by a 2nd-order polynomial equation, as shown in Equation (11):

$$Y = 1.33 - 1.73A - 0.1685B - 0.3474C + 1.39D + 0.6978AB + 1.22AC \\ - 0.8066AD + 0.5853BC - 0.4997BD - 0.4978CD - 6.96A^2 \quad (11) \\ - 4.18B^2 - 6.00C^2 - 5.54D^2$$

Table 5. Summary statistics Model for adsorption of Astrazon pink onto *Prosopis juliflora* biochar.

Source	Std. Dev.	R ²	Adjusted R ²	Predicted R ²	PRESS	
Linear	10.45	0.1701	0.0374	−0.1048	3631.70	
2FI	11.42	0.2468	−0.1496	−0.9922	6549.10	
Quadratic	6.50	0.8073	0.6275	−0.1099	3648.53	Suggested
Cubic	6.51	0.9097	0.6257	−12.0090	42,764.94	Aliased

Table 6. The experimental and predicted significant variables are used in the design matrix.

Run	Factor 1: A pH	Factor 2: B Contact Time Minutes	Factor 3: C Dye Concentration mg/L	Factor 4: D Biochar Dose g/L	Response (%) Experimental Values	Response (%) Predicted Values
1	7.5	70	50	8	92.78	91.78
2	3.5	90	50	8	85.54	86.54
3	7.5	90	50	8	97.44	98.54
4	2	10	90	1	82.31	81.31
5	13	170	90	1	72.78	71.78
6	2	10	10	1	75.45	74.45
7	8.5	90	50	8	62.454	63.75
8	2	10	90	15	90.89	91.08
9	7.5	90	50	6	86.76	85.78
10	7.5	90	50	8	98.54	98.54
11	7.5	150	50	8	75.67	76.67
12	13	170	10	1	82.34	83.34
13	2	170	10	15	88.78	89.78
14	13	10	90	1	83.56	84.56
15	7.5	90	50	8	98.54	98.54
16	2	170	90	1	80.89	80.15
17	2	170	90	15	79.89	80.89
18	7.5	90	50	8	97.54	98.54
19	13	170	10	15	64.56	65.56
20	7.5	90	50	8	96.34	98.54
21	7.5	90	50	12	90.87	90.86
22	2	10	10	15	83.45	83.31
23	13	170	90	15	82.45	82.45
24	13	10	10	1	80.12	81.12
25	7.5	90	30	8	76.54	75.65
26	12	170	10	1	70.56	70.58
27	13	10	10	15	73.45	74.54
28	7.5	90	50	8	96.44	98.54
29	7.5	90	30	8	93.45	92.45
30	13	10	90	15	65.89	66.59

Table 5 presents the forecast Astrazon pink adsorption capacity (mg/g) obtained using Equation (11). We found that the obtained value and the experimental values (actual) were moderate, indicating that both the forecast and experimental values showed good agreement [70]. Using the experimental (actual) values and the expected response, the model determined the correlation coefficient (R²) to be 0.8073. The adsorption capacity of the process parameters accounts for 98.54% of the variability, and this model explains only 1.00% of the response variation [71].

Additionally, the model's adequacy was assessed using an ANOVA (Analysis of Variance). Table 7 presents the fitting model in ANOVA and quadratic response surface forms. As mentioned in [72], ANOVA partitions the total variation into two components: one related to the model and the other to the experimental error, thereby determining whether the model is significant. It is calculated using the F-value, expressed as the square of the ratio of the mean model residual error. If the tabulated F-value exceeds the estimated value, the model is a strong predictor of the experimental data [73]. In the present study, the F-value of 4.49 suggests the fitness of the response surface model. The significance of the model was assessed using the probability of error (Prob > F) of 0.0033, indicating that the model is statistically significant [74]. The table clearly shows that the model is significant for the adsorption capacity of Astrazon pink onto *Prosopis juliflora* biochar. The F-value of 4.49 indicates that the model is statistically significant. In this study, *p*-values less than 0.0033 indicate that the model is significant. The ANOVA table shows that A², C², and D² are significant model terms. The other terms in the ANOVA table with values greater than 0.1000 indicate that the model terms are not significant. Many models are insignificant (not included to support the hierarchy) and therefore require improvement. Lack of Fit is substantial, as indicated by an F-value of 4.49. Figures 14 and 15 indicate the 3D surface plot of Astrazon pink adsorption capacity at pH(A), Contact time (min) (B), Dye Concentration (mg/L) (C), and Biochar dose g/L (D). However, increasing contact time will reduce adsorption capacity until the optimal elimination of Astrazon Pink dye by *Prosopis juliflora* biochar is achieved.

Table 7. ANOVA for the adsorption of Astrazon pink onto *Prosopis juliflora* biochar using a quadratic model.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	
Model	2653.92	14	189.57	4.49	0.0033	significant
A-pH	367.54	1	367.54	8.70	0.0099	
B-Contact time	89.78	1	89.78	2.132	0.1654	
C-Dye Concentration	98.66	1	98.66	2.34	0.1472	
D-Biochar Dose	3.30	1	3.30	0.0782	0.7836	
AB	2.24	1	2.24	0.0529	0.8212	
AC	7.37	1	7.37	0.1746	0.6820	
AD	233.63	1	233.63	5.53	0.0327	
BC	0.0729	1	0.0729	0.0017	0.9674	
BD	8.64	1	8.64	0.2047	0.6574	
CD	0.1444	1	0.1444	0.0034	0.9541	
A ²	1275.77	1	1275.77	30.21	<0.0001	
B ²	498.35	1	498.35	11.80	0.0037	
C ²	454.35	1	454.35	10.76	0.0051	
D ²	266.15	1	266.15	6.30	0.0240	
Residual	633.42	15	42.23			significant
Lack of Fit	633.42	10	63.34			
Pure Error	0.0000	5	0.0000			
Cor Total	3287.34	29				

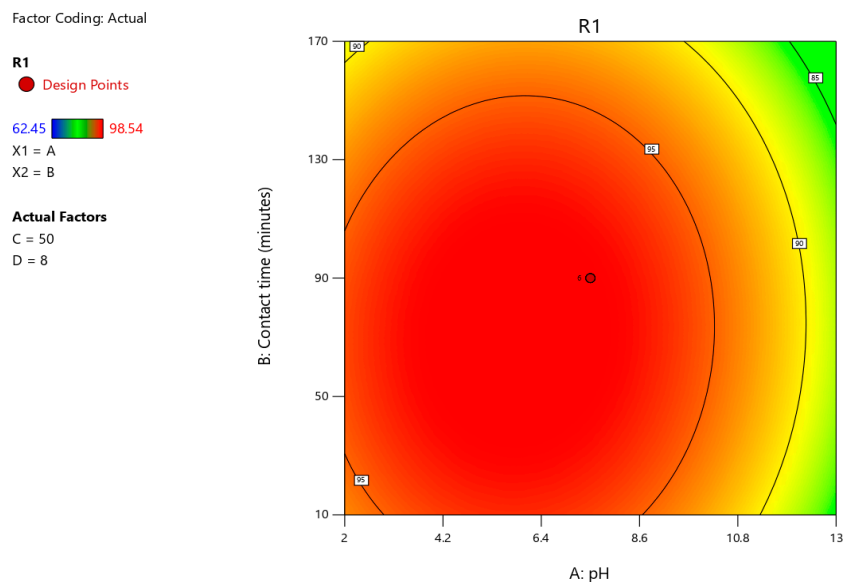


Figure 14. Response surface plots for the simultaneous interaction of Biochar dose with dye concentration.

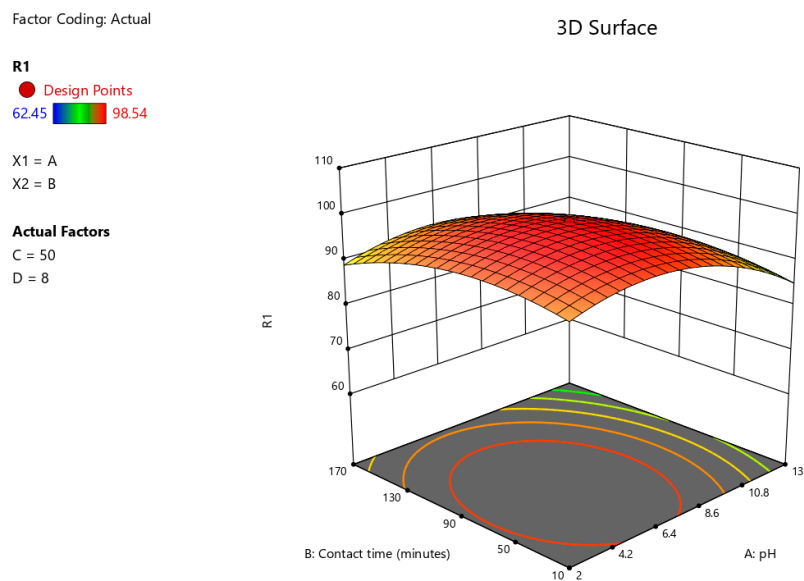


Figure 15. 3D response surface plots of interactive effects of optimized parameters of biochar dose, dye concentration, contact time, and temperature.

4. Conclusions

In conclusion, biochar produced from *Prosopis juliflora* shells through thermochemical process at 500 °C effectively removed Astrazon pink dye from aqueous solutions. These studies determined the optimized parameters for dye removal, with batch studies showing that the pseudo-second-order kinetics and the Freundlich isotherm models provided the best fits. Thermodynamic parameters indicate that the adsorption process is feasible, spontaneous, and endothermic, with an increase in randomness at the solid-solution interfaces. Response Surface Methodology (RSM) analysis further optimized the process parameters, confirming alignment between the analytical experiments and the

statistical approach at pH 7.5, a dye concentration of 50 mg/L, a biochar dose of 8 g/L, a temperature of 35 °C, and a contact time of 90 min.

Author Contributions: Conceptualisation, L.M. and L.M.; methodology, L.M.; software, L.M.; validation, L.M.; formal analysis, L.M.; investigation, L.M.; resources, L.M.; data curation, L.M.; writing—original draft preparation, L.M.; writing—review and editing, R.J.; visualisation, L.M.; supervision, R.J.; project administration, L.M. All authors have read and agreed to the published version of the manuscript.

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