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ORIGINAL RESEARCH ARTICLE

Response Surface Optimization, Isotherm, Kinetic and Thermodynamic Studies of Methylene Blue Adsorption onto Conductive Poly(3-butylthiophene)

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ABSTRACT

Conductive poly(3-butylthiophene) (P3BT) was synthesized via oxidative polymerization and investigated as an adsorbent for methylene blue (MB) removal from aqueous solutions. Structural characterization using FTIR, SEM, XRD, and BET analyses confirmed the formation of a porous semi-crystalline polymer with a BET surface area of 392.5 $m^2 g^{-1}$ and abundant adsorption-active sites. Adsorption parameters, including pH, adsorbent dosage, initial dye concentration, contact time, and temperature, were optimized using Response Surface Methodology (RSM) based on Box–Behnken Design. The developed quadratic model was statistically significant ($p < 0.0001$) with excellent predictive capability ($R^2 = 0.9987$), and the adjusted R^2 and predicted R^2 were in agreement, with no significant lack of fit. Under optimized conditions of pH 7.5, 0.55 g L^{-1} adsorbent dosage, 105 mg L^{-1} dye concentration, 70 min contact time, and 40 °C, a maximum adsorption capacity of 270.46 mg g^{-1} and 99.6% methylene blue removal efficiency were achieved. Adsorption equilibrium followed the Langmuir isotherm model ($R^2 = 0.9939$), indicating monolayer adsorption on homogeneous active sites, while adsorption kinetics followed the pseudo-second-order model ($R^2 = 0.9978$), suggesting adsorption was governed predominantly by surface interaction processes. Thermodynamic studies revealed spontaneous, endothermic adsorption, characterized by a negative Gibbs free energy ($\Delta G° < 0$) and a positive enthalpy change ($\Delta H° = 60.01$ kJ mol^{-1}). Regeneration studies showed that P3BT retained approximately 59% adsorption efficiency after five adsorption–desorption cycles, demonstrating moderate reusability and structural stability. The findings establish that conductive P3BT is a promising high-capacity adsorbent for the remediation of dye-contaminated wastewater.

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INTRODUCTION

The discharge of dye-contaminated effluents from textile, paper, leather, and pharmaceutical industries represents a serious environmental problem due to the persistence, toxicity, and poor biodegradability of synthetic dyes, which can disrupt aquatic ecosystems by limiting light penetration and inhibiting photosynthesis (Habibu et al., 2023). Methylene blue (MB), a widely used cationic dye, is of particular concern owing to its high solubility, aromatic structure, and harmful effects on aquatic organisms and human health at elevated concentrations (Ladan et al., 2024). Although several treatment techniques, such as chemical oxidation, membrane filtration, and biological processes, have been explored, their application is often constrained by high costs, incomplete dye removal, and secondary pollution (Kocijan et al., 2023). Adsorption has therefore emerged as an effective and practical alternative due to its simplicity, high efficiency, and versatility (Satyam & Patra, 2024).

Conducting polymers have recently attracted significant attention as adsorbents due to their π-conjugated structures, tunable surface functionalities, redox properties, and strong affinity toward aromatic dye molecules through π–π interactions and electrostatic attraction (Birniwa et al., 2022). Among conducting polymers, polythiophene derivatives exhibit excellent chemical stability, electron delocalization, and adsorption potential for organic pollutants. However, compared with extensively studied conducting polymers such as polyaniline and polypyrrole, adsorption studies on poly(3-butylthiophene) remain very limited, despite its promising surface and electronic characteristics. Recent studies have

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emphasized the need for more detailed investigations into polythiophene-based adsorbents for dye remediation application. In this context, conducting polymers, especially polythiophene and its derivatives, have gained increasing attention as adsorbents because of their tunable surface properties and π-conjugated backbones, which enable strong π–π and electrostatic interactions with aromatic cationic dyes such as MB (Goswami et al., 2023).

Polythiophene-based materials have demonstrated high methylene blue removal efficiencies, attributed to favorable adsorption kinetics and strong interactions between the dye and the polymer (Husain et al., 2020). However, adsorption performance is highly dependent on operational parameters, necessitating systematic optimization approaches such as Response Surface Methodology (RSM) to evaluate variable interactions and identify optimal conditions with high predictive accuracy (Habibu et al., 2023).

Although several adsorbents, including activated carbon, metal–organic frameworks, chitosan composites, and conducting polymer nanocomposites, have been reported for methylene blue removal, many suffer from low regeneration efficiency, limited adsorption capacity, poor surface tunability, or expensive synthesis methods. Furthermore, limited studies have combined adsorption modeling, thermodynamic evaluation, and multivariable statistical optimization for methylene blue adsorption using conductive poly(3-butylthiophene). Therefore, this study investigates the adsorption behavior of methylene blue onto conductive P3BT using equilibrium, kinetic, thermodynamic, and Response Surface Methodology approaches.

MATERIALS AND METHODS

Materials

3-Butylthiophene monomer and ferric chloride ($FeCl_3$) were used for the synthesis of Poly(3-butylthiophene) (P3BT). Methylene blue (MB) dye of analytical grade was employed as the model cationic adsorbate. All reagents were used without further purification, and deionized water was used in all experimental procedures.

Synthesis of Poly(3-butylthiophene)

Poly(3-butylthiophene) was synthesized via oxidative chemical polymerization as described by. In a typical procedure, 3-butylthiophene monomer (2 ml) was dissolved in chloroform (50 ml) under continuous stirring at room temperature. Ferric chloride (7.5 g dissolved in 50 ml chloroform) was gradually introduced as an oxidizing agent to initiate polymerization. The reaction mixture was stirred continuously for 4 hours to ensure complete polymerization. The resulting polymer precipitate was filtered and washed repeatedly with 200 ml of methanol, then with deionized water to remove residual oxidant, unreacted monomer, and low-molecular-weight oligomers. The purified polymer was dried in a vacuum oven at 60 ℃ for 6 hours.

RESULTS AND DISCUSSION

Characterization of the Adsorbent

FTIR spectra were recorded to identify the functional groups responsible for methylene blue adsorption. SEM analysis was performed to evaluate surface morphology and porosity. XRD analysis was conducted to determine the crystallinity of the synthesized polymer, while BET surface area analysis was used to evaluate the textural properties and adsorption-active surface area of P3BT.

The chemical structure of the synthesized P3BT was analyzed using Fourier Transform Infrared FTIR (Carry 630). The FTIR confirmed thiophene ring bending vibrations (686 cm^{-1}), and the peak at 1737 cm^{-1} is due to the C=C stretching mode.

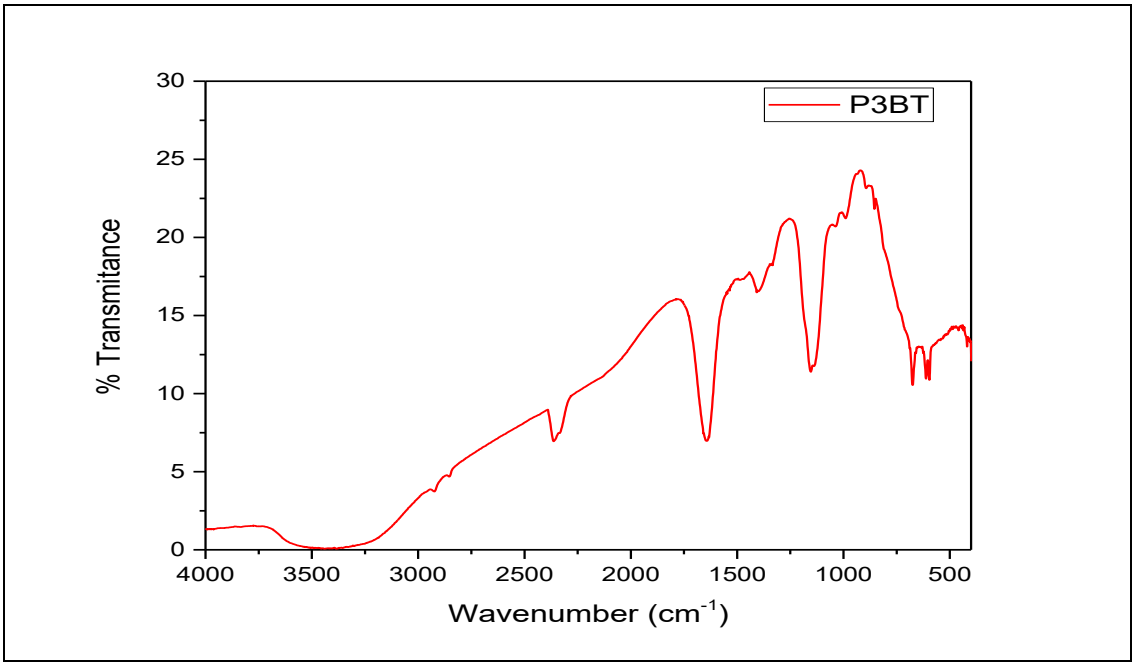

Figure 1: FTIR spectrum of Poly(3-butylthiophene)

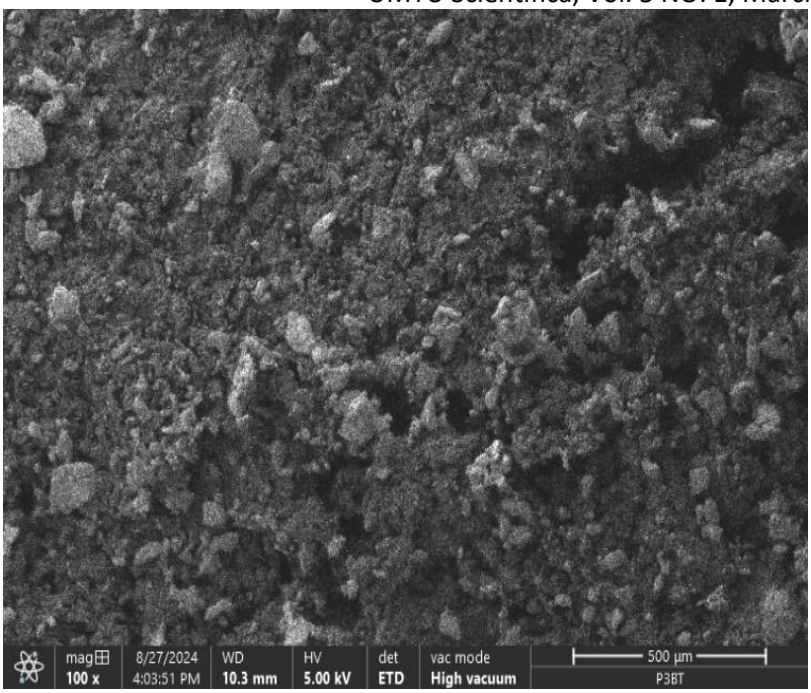

Figure 2*:* SEM micrograph of Poly(3-butylthiophene)

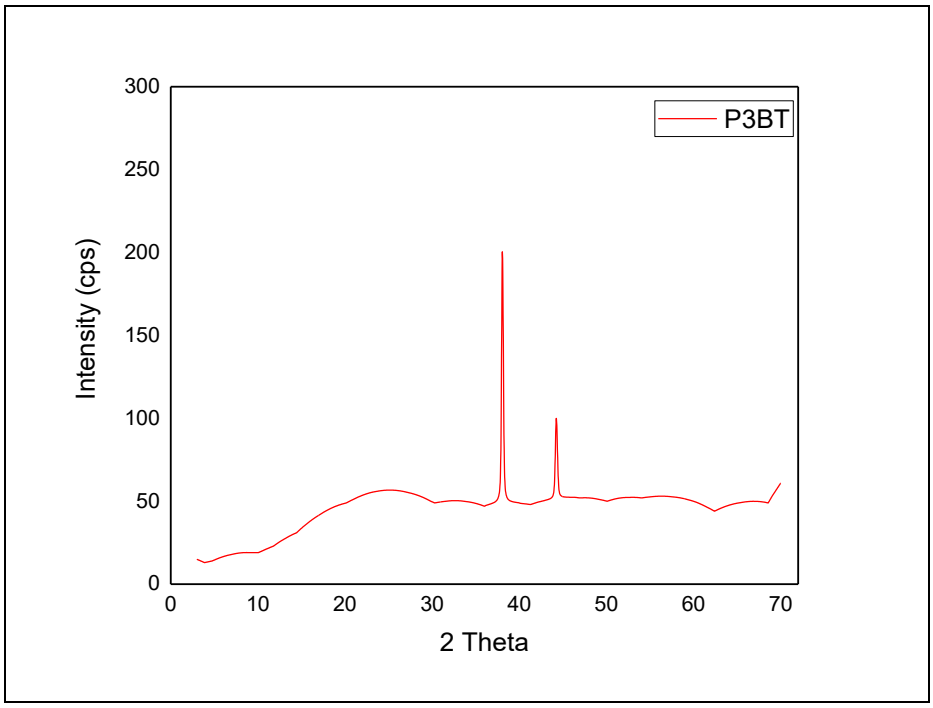

Figure 3*:* XRD peak of Poly(3-butylthiophene).

The FTIR spectrum confirmed successful formation of P3BT through characteristic thiophene ring vibrations and conjugated polymer backbone peaks. The presence of aromatic and sulfur-containing functional groups suggests the availability of adsorption-active sites capable of interacting with methylene blue molecules through π–π interactions and electrostatic attraction.

The FTIR spectrum is given in Figure 1.

Surface morphology and textural characteristics were examined using Scanning Electron Microscopy (SEM- (PHENOM PRO X)). SEM micrographs of P3BT (Figure 2) revealed a rough, porous surface morphology with irregular surface cavities that enhance adsorption by providing accessible active sites for methylene blue uptake.

Crystallinity was assessed by X-ray diffraction (Shimadzu XRD-6000). The XRD analysis (Figure 3) indicated that P3BT exhibited a predominantly amorphous/semi-crystalline structure, which is typical for conducting polymers synthesized via chemical oxidation. The amorphous nature is advantageous for adsorption due to enhanced accessibility of adsorption sites.

Brunauer–Emmett–Teller (BET) analysis revealed a specific surface area of [392.508 m^2/g], with a total pore volume of [0.220 cm^3/g] and an average pore diameter of [2.129 nm] (Table 1).

Table 1: BET Analysis of P3BT

Parameter	P3BT
Surface Area	392.508 m^2/g
Pore Volume	0.220 cm^3/g
Pore Size	2.129 nm

Table 2: Kinetics parameters for the adsorption of Methylene blue dye onto P3BT

Models	Kinetic Parameters	MB
Pseudo First Order	$q_{e,exp.}$(mg/g)	18.617
	$q_{e,cal.}$(mg/g)	2.467
	k_1(min^{-1})	0.0099
	R^2	0.7811
Pseudo Second Order	$q_{e,exp.}$(mg/g)	18.617
	$q_{e,cal.}$(mg/g)	18.083
	k_2(g/mg.min)	0.078
	R^2	0.9978
Intraparticle Diffusion	C (mg/g)	15.642
	k_{diff}(mg/g.$min^{1/2}$)	0.2454
	R^2	0.4991

Table 3: Isotherm parameters for the adsorption of Methylene blue dye onto P3BT

Adsorption isotherm	Parameters	MB
Langmuir	Q_M(mg/g)	270.46
	K_L(L mg^{-1})	0.0501
	R_L	0.5061
	R^2	0.9939
Temkin	A_T(L/mg)	1.2064
	B_T(J/mol)	10.918
	R^2	0.8771
Freundlich	n_f	0.4145
	1/n	2.4125
	k_f(mg/g)	2.4952
	R^2	0.6296

Table 4: Thermodynamic parameters for the adsorption of Methylene blue dye onto P3BT

Dyes	ΔG (kJ/mol) ΔG = ΔH – TΔS			ΔH (kJ/mol)	ΔS ($Jmol^{-1}K^{-1}$)
	308	315.5	323		
MB	- 8.8753	- 10.5527	- 12.2301	60.0105	223.655

The BET surface area of 392.5 $m^2 g^{-1}$ confirms the highly porous nature of P3BT and accounts for its high adsorption capacity for methylene blue. The large surface area significantly increases the number of accessible adsorption sites and promotes enhanced dye-surface interactions.

Batch Adsorption Experiments

Batch adsorption experiments were conducted to study the adsorptive behavior of poly(3-butylthiophene) for the adsorption of the cationic (MB) dye, as described by Muhammad et al. (2022). 0.55g of P3BT was added to 100 mL of 105mg/L MB solution, the solution was magnetically stirred at room temperature, 4 mL of the MB solution was taken out at a predetermined time and analyzed using UV spectrophotometer after proper centrifugation. To study the adsorption kinetics, the reaction was assumed to be complete when the dye solution became colorless, and measurements were stopped at this stage. All adsorption experiments were conducted in triplicate, and average values were reported.

The adsorptive capacity Qt (mg/g) and dye removal efficiency were calculated using equations 1 and 2 (Liu et al., 2020).

$$Qt = \frac{(Co-Ct)V}{m} \quad \text{..... (1)}$$

$$Removal\ efficiency\ (\%) = \frac{Co-Ce}{Ce} \text{x } 100 \quad \text{..... (2)}$$

where C_o (mg/L) is the initial concentration of the dye solution, C_t (mg/L) is the concentration of the dye solution at time t, C_e (mg/L) is the dye concentration at equilibrium, V (L) is the initial volume of the dye solution, and m (g) is the adsorbent mass (Shajahan et al., 2017).

- pH Effect: MB solutions (105 mg/L, 100 mL) were adjusted (pH 2 to 12) using 0.1 M HCl/NaOH. P3BT (0.55 g) was added and agitated (200 rpm, 42.5°C, 1 h).

- Dose/Concentration: Varied P3BT (0.1 - 1 g/L) and MB (10 to 200 mg/L) under optimal pH.
- Kinetics/Isotherms: Sampled at intervals (20 to 120 min) for residual MB analysis (UV-Vis at 664 nm).

Adsorption Kinetics

Adsorption kinetic studies were performed to investigate the rate and mechanism of MB uptake by P3BT (Table 2). Experiments were conducted at fixed initial dye concentration and adsorbent dosage, while varying the contact time (Imam & Hamza, 2025). The experimental data were analyzed using kinetic models, including the pseudo-first-order, pseudo-second-order, and intra-particle diffusion models, to determine the best-fitting model based on correlation coefficients and agreement between experimental and calculated adsorption capacities (Rabi'u et al., 2023).

Adsorption Isotherms

Adsorption isotherm studies (Table 3) were carried out by varying the initial MB concentration while maintaining constant temperature, adsorbent dosage, and contact time sufficient to reach equilibrium (Imam & Hamza, 2025). The equilibrium data were analyzed using Langmuir, Temkin, and the Freundlich isotherm models to describe the adsorption behavior and surface characteristics of P3BT. Model parameters were determined by linear regression.

Table 5: Analysis of Variance for the adsorption of Methylene blue dye onto P3BT

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	447.80	20	22.39	4121.77	< 0.0001	Significant
A-pH	8.82	1	8.82	1623.83	< 0.0001	
B-Time	10.84	1	10.84	1995.62	< 0.0001	
C-Dosage	5.42	1	5.42	997.26	< 0.0001	
D-Concentration	110.15	1	110.15	20276.44	< 0.0001	
E-Temperature	0.56	1	0.56	102.17	< 0.0001	
AB	1.54	1	1.54	283.05	< 0.0001	
AC	12.67	1	12.67	2333.07	< 0.0001	
AD	12.04	1	12.04	2216.59	< 0.0001	
AE	0.45	1	0.45	82.64	< 0.0001	
BC	17.85	1	17.85	3286.10	< 0.0001	
BD	5.02	1	5.02	923.68	< 0.0001	
BE	0.14	1	0.14	26.58	< 0.0001	
CD	28.68	1	28.68	5278.93	< 0.0001	
CE	0.16	1	0.16	28.72	< 0.0001	
DE	21.11	1	21.11	3886.85	< 0.0001	
A^2	41.70	1	41.70	7676.08	< 0.0001	
B^2	21.19	1	21.19	3901.45	< 0.0001	
C^2	82.97	1	82.97	15273.78	< 0.0001	
D^2	128.19	1	128.19	23597.71	< 0.0001	
E^2	110.55	1	110.55	20351.77	< 0.0001	
Residual	0.14	25	5.432E-003			
Lack of Fit	0.13	20	6.386E-003	3.95	0.0668	not significant
Pure Error	8.083E-003	5	1.617E-003			
Cor Total	447.94	45				
Std. Dev.	0.074		R-Squared	0.9997		
Mean	87.91		Adj R-Squared	0.9995		
C.V. %	0.084		Pred R-Squared	0.9988		
PRESS	0.52		Adeq Precision	257.014		

Final Equation in Terms of Actual Factors: % Dye Removal = 82.9683 + -0.7425 * A + 0.823125 * B + -0.581875 * C + 2.62375 * D + 0.18625 * E + 0.62 * AB + -1.78 * AC + 1.735 * AD + -0.335 * AE + 2.1125 * BC + -1.12 * BD + 0.19 * BE + -2.6775 * CD + 0.1975 * CE + -2.2975 * DE + 2.18583 * A^2 + 1.55833 * B^2 + 3.08333 * C^2 + 3.8325 * D^2 + 3.55917 * E^2

Thermodynamic Studies

Thermodynamic parameters (Table 4) governing the adsorption process were evaluated by conducting adsorption experiments at different temperatures. Standard Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were calculated using the temperature-dependent equilibrium constants. These parameters were used to assess the spontaneity, feasibility, and nature of the adsorption process (Ladan et al., 2025).

Experimental Design and Response Surface Methodology

Response Surface Methodology (RSM) was employed to statistically model and optimize the adsorption of methylene blue onto Poly(3-butylthiophene). A Box–Behnken Design (BBD) was selected for its efficiency in evaluating quadratic response surfaces, requiring fewer experimental runs than full factorial designs (Sulaiman et al., 2024). Five independent variables: adsorbent dosage (A), initial methylene blue concentration (B), pH of

solution (C), contact time (D), and temperature (E) - were investigated at three coded levels to assess their individual, interactive, and quadratic effects on methylene blue removal efficiency.

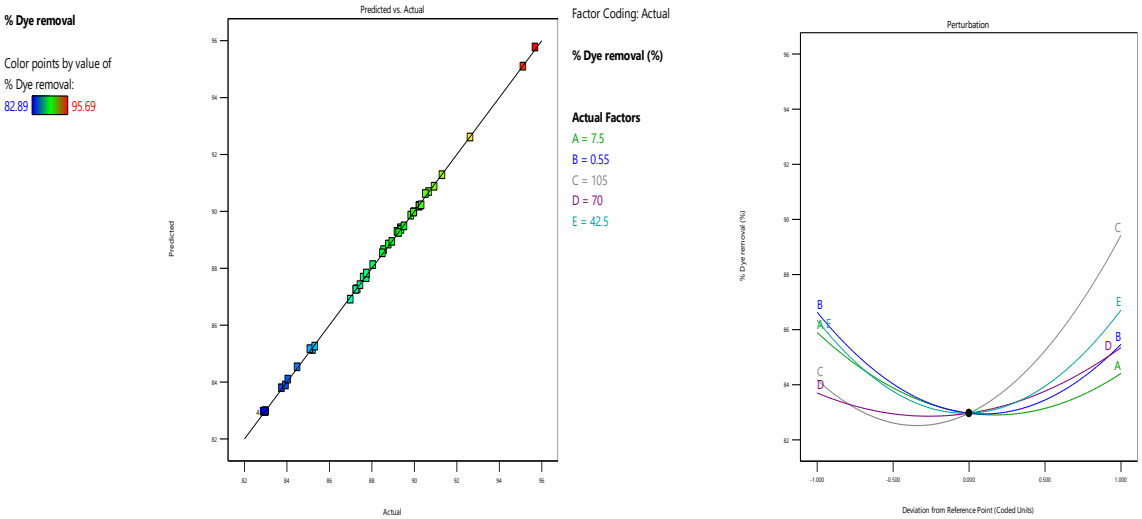

Figure 5i: (a) Plot of Predicted and Actual values for BBD methylene blue dye adsorption (b) Perturbation curve for BBD methylene blue dye adsorption.

The experimental data were fitted to a second-order polynomial model to describe the relationship between the response and the independent variables. The adequacy of the developed model was evaluated using analysis of variance (ANOVA), with statistical significance determined by the F-value and corresponding p-value (Table 5). The goodness of fit was assessed through the coefficient of determination (R^2), adjusted R^2, predicted R^2, and lack-of-fit tests. Three-dimensional response surface and contour plots were generated to visualize interaction effects among process variables and identify optimal operating conditions. Numerical optimization was subsequently performed to determine the combination of experimental parameters that maximized methylene blue removal efficiency while ensuring model reliability and predictive accuracy (Igwegbe et al., 2019).

The adsorption efficiency of P3BT toward methylene blue was evaluated under varying experimental conditions. The results demonstrated that adsorption efficiency increased with increasing contact time, as available adsorption sites were progressively occupied by methylene blue molecules until equilibrium was attained. Similarly, adsorption improved with increasing temperature, indicating enhanced molecular mobility and favorable interaction between MB molecules and the polymer surface.

Adsorption kinetics (Table 2) data were analyzed using pseudo-first-order, pseudo-second-order and intra-particle diffusion models to elucidate the adsorption mechanism. The pseudo-second-order model provided the best fit to the experimental data (R^2 = 0.9978), while the pseudo-first-order and intraparticle diffusion models yielded significantly lower R^2 values. The excellent agreement between the experimental and calculated adsorption capacities confirms that methylene blue adsorption onto P3BT is predominantly controlled by surface interaction processes involving valence-electron sharing or exchange. Additionally, the calculated equilibrium adsorption capacity closely matched the experimental values.

Equilibrium adsorption (Table 3) data were fitted to the Langmuir, Temkin, and Freundlich isotherm models. The Langmuir isotherm model exhibited the highest correlation coefficient (R^2 = 0.9939), indicating that methylene blue adsorption onto P3BT predominantly occurs as monolayer adsorption on relatively homogeneous active sites. The high maximum adsorption capacity (Qm = 270.46 mg g^{-1}) further demonstrates the strong affinity of P3BT toward methylene blue molecules. The comparatively lower fitting of the Freundlich and Temkin models suggests that multilayer adsorption and heterogeneous surface interactions contributed minimally to the overall adsorption process.

Thermodynamic parameters (Table 4) were evaluated to assess the nature of the adsorption process. The calculated Gibbs free energy change values are ($\Delta G°$ = - 8.8753, - 10.5527, and - 12.2301 kJ/mol), and the entropy change values are: ($\Delta S°$ = 223.655 J/mol·K). The negative Gibbs free energy values confirmed the spontaneous nature of methylene blue adsorption onto P3BT. The positive enthalpy value ($\Delta H°$ = 60.01 kJ mol^{-1}) indicates that the adsorption process is endothermic, while the positive entropy value suggests increased randomness at the solid-solution interface during adsorption.

Proposed Adsorption Mechanism

The adsorption of methylene blue onto P3BT is primarily governed by π–π interactions between the aromatic structure of methylene blue and the π-conjugated backbone of the conducting polymer. Electrostatic attraction between positively charged methylene blue molecules and adsorption-active sites on the polymer surface also contributes significantly to adsorption. Furthermore, sulfur-containing functional groups present within the thiophene rings may facilitate additional dye-surface interactions. The high BET surface area and porous morphology of P3BT further enhance dye accessibility and adsorption efficiency.

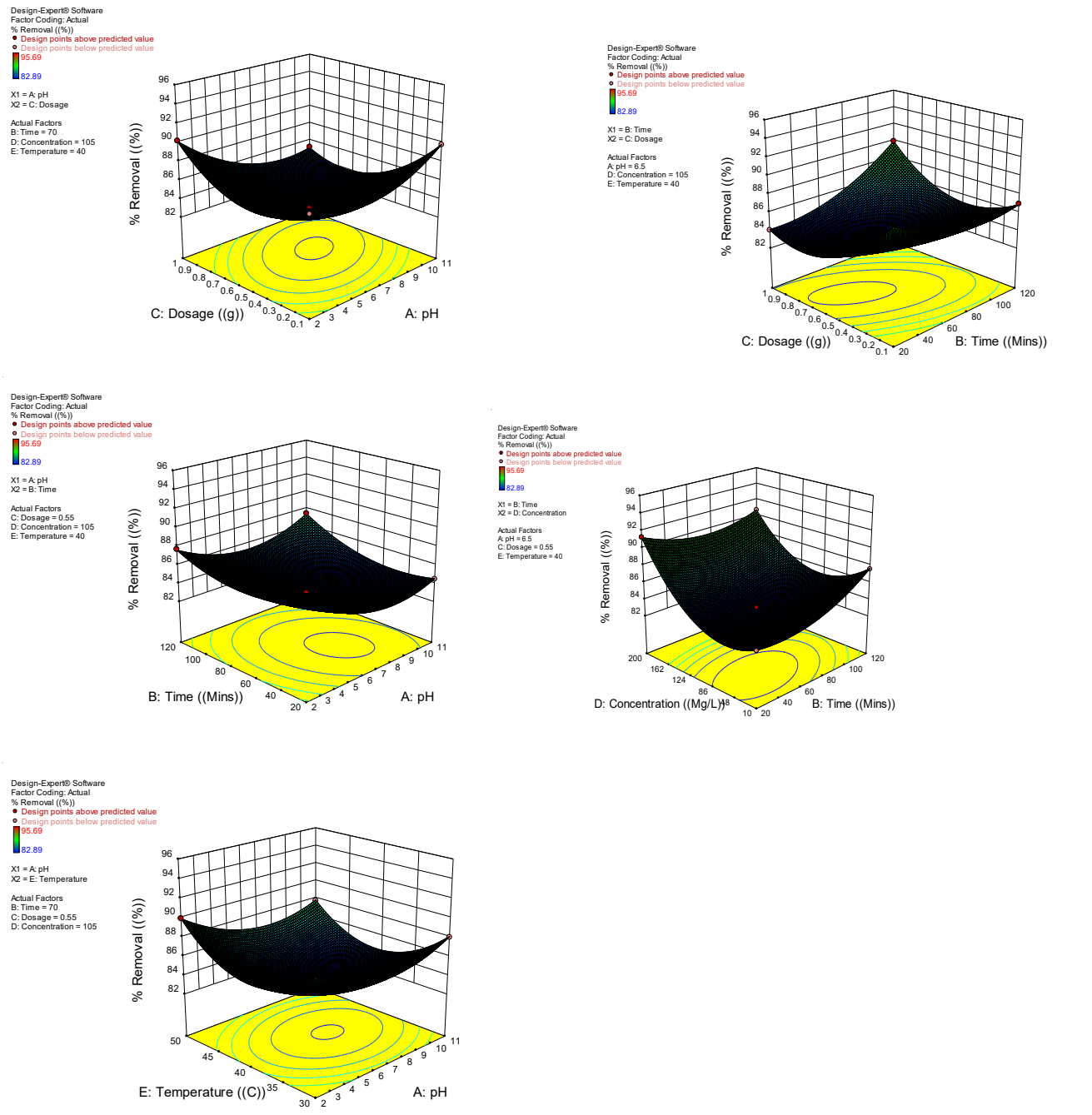

Figure 5ii*:* 3D interactions displaying the Effect of (a)Time and pH, (b) Adsorbent dosage and pH, (c) Adsorbent dosage and Time, (d) dye concentration and contact time and (e) Temperature and pH

Table 6: Comparative Adsorption Performance of P3BT and other conventionally used adsorbents

Adsorbent	qmax (mg/g)	Reusability (cycles)	Surface Area (m^2/g)	Source/Reference
Activated Carbon	150	~3	900	(El Jery et al., 2024)
Chitosan	120	~3	25	(Ahmad & Ansari, 2021)
MOFs (ZIF-8)	245	4–5	1300	(Sulaiman et al., 2024)
Poly(3-butylthiophene)	270.46	5	392.508	This study

Response Surface Methodology and Model Validation

The experimental data obtained from the Box-Behnken Design were fitted to a quadratic polynomial model. Analysis of variance (ANOVA) confirmed that the developed model was statistically significant. The developed quadratic model demonstrated excellent predictive capability (R^2 = 0.9987), indicating that it explained 99.87% of the variability in methylene blue removal efficiency. The close agreement between the adjusted R^2 and the predicted R^2 further confirms the model's reliability and adequacy. In addition, the insignificant lack-of-fit indicates that the developed model sufficiently represents the experimental adsorption system. The high F-value and p-value less than 0.0001

confirmed the statistical significance of the adsorption model and the strong influence of the selected operational variables on methylene blue adsorption.

Figure 5i(a) displays the similarity of actual and predicted values for the adsorption of methylene blue onto the adsorbent. The plot serves as a visual indicator of the model's goodness-of-fit and its ability to navigate the design space. Both data sets exhibit a significantly high degree of proximity, underscoring the suitability of the suggested model. The R^2 values of 0.9997 indicate a strong correlation between the actual and expected responses. The plot in Figure 5i(b) shows a perturbation plot that compares the effects of multiple factors at a specific point. The plot illustrates how the response changes as each factor moves away from a central reference point while all other factors are held constant. Thus, the perturbation analysis confirms that the dye removal process is highly sensitive to perturbations in factors, with factor C as the dominant driver.

Three-dimensional response surface plots (Figures 5ii(a)–(e)) illustrated the interactive effects of adsorbent dosage, initial dye concentration, contact time, pH, and temperature on MB removal efficiency. Strong interaction effects were observed, particularly between (Variable A – B) and (Variable B – C), highlighting the importance of multivariate optimization. Numerical optimization predicted optimal adsorption conditions: adsorbent dosage = 0.55 g/L, concentration = 105 mg/L, time = 70 min, pH = 6.5, and temperature = 40 ℃, under which a maximum MB removal efficiency of 99.6% was achieved and experimentally validated.

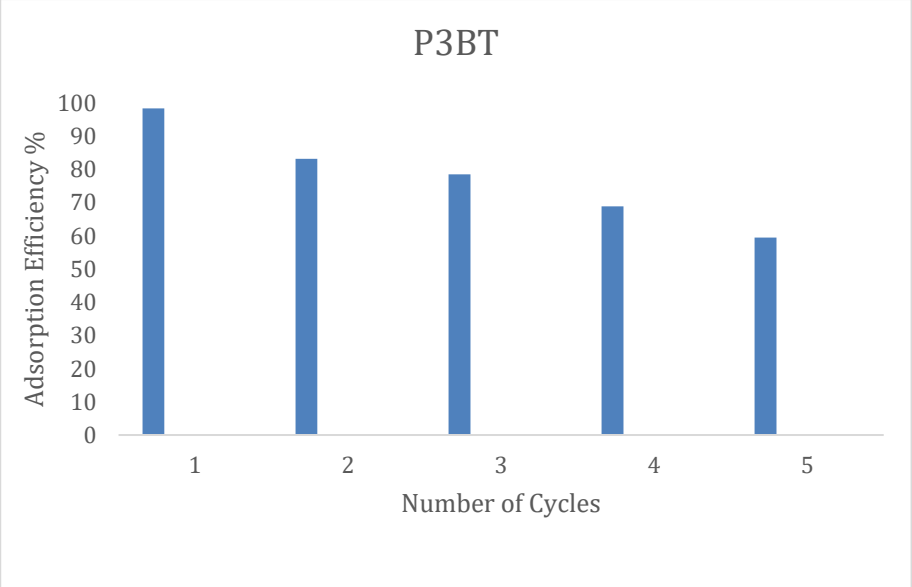

Figure 6; Regeneration of P3BT

Regeneration and Reusability

The regeneration and reuse performance of P3BT was systematically evaluated to determine its operational stability and long-term applicability. Desorption of methylene blue was achieved using 0.1 M HCl as the eluent, which effectively disrupted the dye–adsorbent interactions and restored the adsorption sites. After five consecutive adsorption–desorption cycles, P3BT retained 59% of its initial adsorption efficiency (Fig. 6), indicating moderate loss of active binding sites and strong structural integrity. The gradual decline in adsorption efficiency after repeated regeneration cycles may be attributed to partial blockage of adsorption-active sites, incomplete desorption of methylene blue molecules, and minor structural changes during acidic regeneration. These findings confirm that P3BT possesses moderate regeneration efficiency, indicating partial irreversible adsorption likely due to strong dye–polymer interactions, reinforcing its potential as a cost-effective and sustainable adsorbent for wastewater treatment applications.

The adsorption capacity of P3BT (270.46 mg g^{-1}), as shown in Table 6, compares favorably with many conventional adsorbents reported for methylene blue removal, including activated carbon (150 mg g^{-1}), chitosan-based adsorbents (120 mg g^{-1}), and several conducting polymer systems. The superior adsorption performance may be attributed to the conductive π-conjugated structure, high BET surface area, and enhanced dye-surface interaction capability of P3BT.

CONCLUSION

This study successfully synthesized Poly(3-butylthiophene) (P3BT) via oxidative chemical polymerization and systematically evaluated its potential as an adsorbent for methylene blue (MB) removal. FTIR, SEM, XRD, and BET analyses confirmed the formation of a polymer with appropriate functional groups, favorable surface morphology, and sufficient surface area to facilitate adsorption.

The findings demonstrate that conductive Poly(3-butylthiophene) has excellent potential as a high-capacity adsorbent for the removal of methylene blue from aqueous systems. The combined characterization, adsorption modeling, thermodynamic analysis, and RSM

optimization confirmed that P3BT exhibits strong adsorption affinity, favorable surface characteristics, and reliable predictive adsorption behavior. The high adsorption capacity (270.46 mg g^{-1}), strong model validation (R^2 = 0.9987), and moderate regeneration performance establish P3BT as a promising conducting polymer adsorbent for advanced wastewater remediation applications.

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