

Efficient Removal of Turbidity, COD, and Colour from Sugar Industry Wastewater Using a Novel Graphene Derived from Pressmud Waste: Optimization Study

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Abstract Wastewater from the sugar industry poses significant environmental challenges due to high turbidity, COD, and color levels, often exceeding permissible limits and impacting aquatic ecosystems and public health. Pressmud, a sugar industry by-product typically discarded, can be converted into graphene for effective wastewater treatment. Therefore, in this study, wastewater from sugar industry was treated using graphene, synthesized from pressmud through carbonization at 600°C and activation with potassium hydroxide at 120°C for 24 hours. The adsorption process was optimized using Response Surface Methodology (RSM) and Central Composite Design (CCD) to analyze the interactions between graphene dosage (0-1g) and pH (5-10). The results demonstrated substantial reductions in turbidity (up to 96.45%), COD (up to 92.51%), and color (up to 98.71%), with R^2 values confirming high predictive reliability. Optimal adsorption conditions were achieved at a graphene dosage of 0.5 g and a pH of 7.5. The findings underscore the potential of converting pressmud into high-value graphene for cost-effective and environmentally sustainable industrial wastewater treatment applications.

Keywords: Turbidity, color, turbidity, graphene, adsorption, industry wastewater.

Introduction

The sugar industry generates significant amounts of wastewater that pose serious environmental challenges due to high levels of color, turbidity, and chemical oxygen demand (COD). These pollutants exceed allowable discharge limits set by the Department of Environment (DOE) in Malaysia, leading to potential ecological and public health risks. Conventional treatment technologies often fail to meet stringent environmental standards. Among these, adsorption technology stands out as an efficient solution due to its simplicity, cost-effectiveness, and high removal efficiencies. Graphene, known for its exceptional adsorption capabilities and large surface area, has emerged as a superior adsorbent for removing color and COD from industrial effluents [1, 2].

The concept of "turning waste into wealth" aligns with the principles of a circular economy, emphasizing resource recovery and sustainability. Pressmud, a by-product of sugar production, is a potential candidate for value addition. Rondina *et al.* has demonstrated the successful conversion of pressmud into activated carbon, showcasing its utility in wastewater treatment [3]. However, limitation of activated carbon as an adsorbent for wastewater treatment is its high cost and reduced efficiency in adsorbing non-polar, high-molecular-weight, or highly soluble contaminants, coupled with challenges in regeneration and disposal. To be noted, graphene offers exceptional adsorption potential for wastewater treatment due to its high surface area, tunable surface chemistry, and excellent affinity for a wide range of organic and inorganic pollutants [1]. Accordingly, the potential of pressmud as a precursor for

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graphene synthesis remains unexplored, presenting an opportunity to integrate waste utilization with advanced material production. Importantly, pressmud presents a unique advantage as a precursor for graphene production due to its high calcium carbonate content, which plays a critical role during carbonization. As calcium carbonate decomposes, it releases carbon dioxide that promotes the development of a porous structure, thereby enhancing the adsorption capacity and surface characteristics of the resulting graphene.

In this study, treated wastewater from a sugar industry in Penang, Malaysia, was collected for further treatment using graphene synthesized from pressmud via adsorption. The collected wastewater, which had undergone preliminary treatment through dilution, failed to meet the discharge standards set by the Department of Environment (DOE), Malaysia, highlighting the inadequacy of the current treatment technology. To address this issue, a comprehensive optimization study was conducted using Response Surface Methodology (RSM) with Design Expert software (version 13.0.5.0). The adsorption process was systematically optimized to evaluate and enhance the removal efficiency of key pollutants, including color and COD, by investigating the interactions between critical variables such as graphene dosage and pH.

Materials and Methods

The pressmud was obtained from a local sugar factory in Penang, Malaysia. Potassium hydroxide (KOH, Chemiz) was used as the agent for activation. Sodium hydroxide (NaOH, Chemiz) and hydrochloric acid (HCl, Chemiz) were used to balance the pH of the synthetic contaminants: sugar industry wastewater.

Synthesis and Characterization of Graphene from Pressmud

Pressmud waste was obtained from a local sugar factory in the state of Penang, Malaysia. In this study, graphene was synthesized from pressmud using a combined carbonization and KOH activation method. Firstly, the pressmud was subjected to carbonization at 600°C to produce carbon. The carbon was subsequently activated using potassium hydroxide (KOH) at a KOH-to-water ratio of 3:1 [2]. The activation process was conducted at 120°C for 24 hours to enhance the porosity and surface properties of the carbon material. Following activation, the samples were thoroughly rinsed with deionized water until the pH of the filtrate reached neutral (pH 7) to remove residual activating agents. The purified material was then filtered and oven-dried at 120°C for 24 hours to obtain graphene. Finally, graphene derived from pressmud was characterized using Transmission Electron Microscopy (TEM, JEM-2100 JEOL, Japan) and Raman spectroscopy using LabRAM HR-800 (Daeil System, Korea).

Performance of Graphene from Pressmud Towards Wastewater from Sugar Factory Treatment

The adsorption performance of graphene synthesized from pressmud was evaluated using wastewater collected from the dilution tank of sugar factory. Samples were stored at 4°C to prevent microbial activity, and adsorption experiments were conducted using CCD-RSM methodology across 11 runs, with a fixed adsorption time of 30 minutes. Turbidity, COD, and color removal efficiencies were measured using standard analytical instruments. Turbidity was measured using a HACH 2100Q turbidimeter. Chemical Oxygen Demand (COD) was evaluated with a HACH DR6000 UV-Vis spectrophotometer following HACH Method 435 for high-range COD analysis. Color intensity was also measured using the DR6000 spectrophotometer, applying the ADMI 96 method. The wastewater from the dilution tank exhibited high pollution levels, with turbidity at 55.7 NTU, COD at 5328 mg/L, and color at 615 ADMI, significantly exceeding the permissible limits set by the Department of Environment (DOE) Malaysia. These results emphasize the need for improved treatment methods, showcasing the potential of graphene-based adsorption for industrial wastewater remediation [3].

Experimental Design Using CCD-RSM

Table 1 presents the experimental design generated using the Central Composite Design (CCD) approach to optimize the removal of turbidity, COD, and color (ADMI) from wastewater. The CCD framework was chosen for its ability to efficiently evaluate both linear and nonlinear interactions among the independent variables, namely graphene content (g) and pH, across three coded levels (-1, 0, +1). The design includes factorial, axial, and center points to ensure a robust exploration of the response surface. The experimental runs were conducted to assess the influence of these factors on the three key responses. The design matrix and corresponding response values are shown in Table 1, which ensures adequate representation of the interaction and quadratic effects of the variables. The experiments were randomized to minimize systematic errors, and each response was modeled using a second-order polynomial equation.

The general form of the response surface model used in this study is expressed as follows:

$$Z = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + \epsilon$$
 (1)

where Z represents the response (turbidity, COD, or color), β_0 is the intercept, β_i denotes the linear coefficients for the factors X_i , β_{ii} are the quadratic coefficients, β_{ij} are the interaction coefficients, and ϵ accounts for random error

Table 1. Design of experiment generated using CCD-RSM

Std	Run	Block	Factor 1	Factor 2	Response 1	Response 2	Response 3
			A:Graphene Content (g)	B:pH	COD (mg/L)	Colour ADMI	Turbidity
1	11	Block 1	0	5	4483	436	25.87
2	4	Block 1	1	5	5417	47	14.5
3	2	Block 1	0	10	4468	500	73.76
4	6	Block 1	1	10	3997	220	3.04
5	9	Block 1	0	7.5	4483	524	35.5
6	8	Block 1	1	7.5	4424	119	15.6
7	3	Block 1	0.5	5	4552	78	3.83
8	5	Block 1	0.5	10	3629	269	2.76
9	10	Block 1	0.5	7.5	4342	179	20.63
10	7	Block 1	0.5	7.5	4343	179	20.63
11	1	Block 1	0.5	7.5	4342	179	20.63

Results and Discussion

Characterization of Graphene Derived from Pressmud

Figure 1 presents the TEM images of graphene synthesized from pressmud, showcasing structural features critical for its adsorption performance. Image (A) reveals wrinkled and folded graphene sheets, which enhance the surface area and provide abundant active sites for adsorption. The lattice structure in Image (B), with its clear atomic arrangements, confirms the presence of few-layer graphene, offering high surface reactivity due to exposed sp^2 hybridized carbon atoms. Image (C) highlights overlapping graphene layers with interlayer spacing, facilitating the adsorption of contaminants through π - π interactions, electrostatic attraction, and van der Waals forces. These structural characteristics align with the adsorption mechanisms observed in similar studies, such as Safian *et al.* (2020), where multilayered graphene derived from biomass exhibited exceptional adsorption efficiency for pollutants due to its high surface area, functional groups, and interlayer interactions [3]. The unique morphology and defects in the graphene structure from pressmud further enhance its adsorption capabilities, making it an effective material for environmental remediation, particularly in removing organic and inorganic pollutants from wastewater.

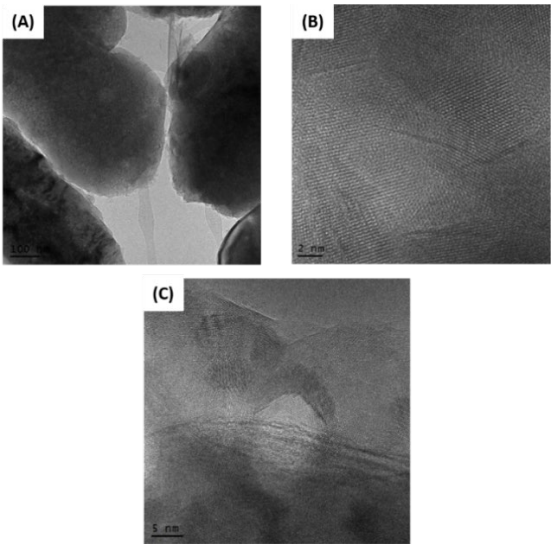


Figure 1. TEM image of graphene derived from pressmud; ; (A) 100 nm; (B) 2 nm; and (C) 5 nm

Figure 2 (A) highlighting the characteristic peaks that confirm its successful formation. The G peak at $\sim 1580\text{ cm}^{-1}$ represents the in-plane vibrations of sp^2 carbon atoms, indicating the graphitic structure of the material. The D peak at $\sim 1350\text{ cm}^{-1}$ is associated with the presence of defects or disorder in the graphene lattice, which can arise from the chemical activation process and the inherent properties of the pressmud precursor. The 2D peak at $\sim 2700\text{ cm}^{-1}$, while prominent, shows an intensity ratio (I_{2D}/I_G) below 2, suggesting the presence of few-layer graphene rather than a monolayer. These spectral features are consistent with those reported by Kumar *et al.* (2020), where graphene derived from biomass-based precursors exhibited similar Raman characteristics, confirming the viability of agricultural or industrial waste as cost-effective feedstock for graphene production [4]. The presence of the D peak in the spectrum also implies potential functionalization of the graphene surface, which can enhance its adsorption capabilities, making it particularly suitable for environmental applications such as wastewater treatment.

On the other hand, Figure 2 (B) looks very different. The strong D peak suggests that the material mostly consists of disordered carbon, with no clear G or 2D peaks to indicate a graphitic structure. This means that before processing, pressmud lacks the ordered structure needed for graphene applications. The drastic difference between the two spectra confirms that the synthesis process successfully transforms raw pressmud into graphene with a more structured carbon arrangement.

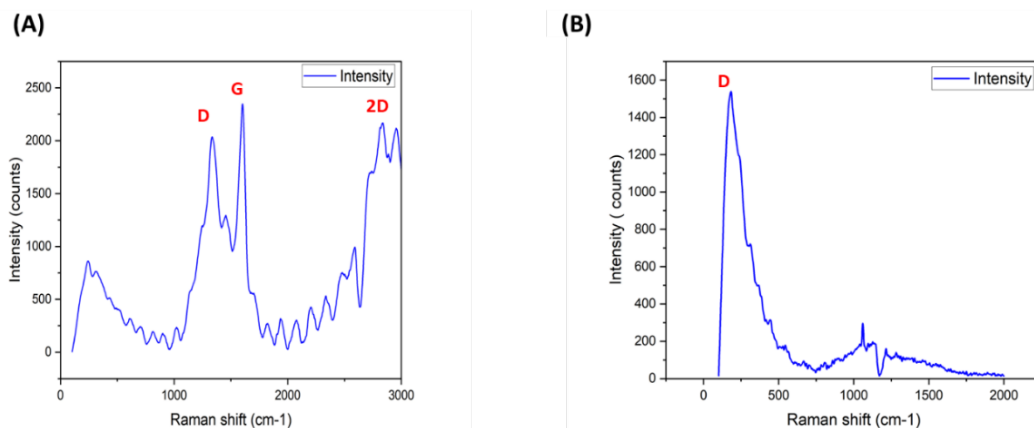


Figure 2. Raman spectra comparison; (A) graphene derived from pressmud; (b) raw pressmud

Process Optimization of Wastewater from Sugar Factory Treatment Using CCD-RSM

The optimization of wastewater treatment from the sugar industry using CCD-RSM demonstrated highly effective removal of turbidity, color, and chemical oxygen demand (COD). For turbidity, the model showed a remarkably strong correlation between experimental and predicted values, with $R^2 = 0.9645$, Adjusted $R^2 = 0.9290$, and Predicted $R^2 = 0.7344$, where high R^2 values confirm the model's reliability and precision. Similarly, for color, the model achieved an outstanding fit, with $R^2 = 0.9871$, Adjusted $R^2 = 0.9743$, and Predicted $R^2 = 0.8656$, emphasizing its exceptional accuracy in predicting removal efficiency under varying experimental conditions. Consequently, these results confirm the suitability of CCD-RSM in modeling and optimizing adsorption processes, particularly for turbidity and color removal, with remarkable adaptability and effectiveness.

Furthermore, for COD removal, the model demonstrated robust predictive capabilities, with $R^2 = 0.9251$, Adjusted $R^2 = 0.8930$, and Predicted $R^2 = 0.6927$. Although the Predicted R^2 value is slightly lower, the overall values remain near 1 [5], clearly highlighting the model's reliability and its ability to capture the interactions between variables such as pH and graphene content. Additionally, the high Adeq Precision values further validate the experimental design's strong signal-to-noise ratio, thereby supporting the adequacy of the model for process optimization. Overall, the statistical analysis strongly underlines the reliability of these models for process optimization, with R^2 , Adjusted R^2 , and Predicted R^2 values near 1 vividly showcasing the impressive adaptability and precision of the RSM approach in wastewater treatment processes. It is noteworthy that equations (2) to (7) illustrate the relationship between the input variables (pH and graphene content) and the response variables (turbidity, color, and COD) through quadratic regression models. These equations are significant as they enable the prediction of treatment efficiency under different operating conditions, offering a valuable tool for optimizing wastewater treatment processes in industrial applications [6].

$$\text{Turbidity}_{\text{coded value}} = 18.75 + 4.24A - 11.10B - 10.99AB - 9.52A^2 + 9.61B^2 \quad (2)$$

$$\text{Turbidity}_{\text{actual value}} = -91.92219 + 28.94330A + 5.30439B - 8.79600AB - 1.52324A^2 + 38.45896B^2 \quad (3)$$

$$\text{Colour}_{\text{coded value}} = 182.21 + 71.33A - 179.00B + 27.25AB - 13.53A^2 + 134.47B^2 \quad (4)$$

$$\text{Colour}_{\text{actual value}} = +241.69737 + 50.09649A - 1059.39474B + 21.8AB - 2.16421A^2 + 537.89474B^2 \quad (5)$$

$$\text{COD}_{\text{coded value}} = +4370.18 - 591.83A + 299.50B - 1099.50AB \quad (6)$$

$$\text{COD}_{\text{actual value}} = +2547.68182 + 203.06667A + 7196B - 8.79600AB \quad (7)$$

Analysis of Variance

Table 2 presents the ANOVA results for optimizing turbidity, color, and COD in sugar industry wastewater using graphene as a treatment agent. For turbidity optimization, the model exhibited an R-squared value of 0.9645, indicating that 96.45% of the variability in turbidity was explained by pH, graphene content, and their interactions. Graphene content had the most significant effect ($p = 0.0006$), followed by pH ($p = 0.0323$) and their interaction ($p = 0.0016$). These findings align with previous studies highlighting graphene's high adsorption efficiency for suspended particles [7]. Similarly, for color optimization, the model achieved a higher R-squared value of 0.9871, with significant contributions from pH ($p = 0.0012$) and graphene content ($p < 0.0001$). The quadratic effect of graphene content (B^2) also played a crucial role, suggesting a nonlinear response, consistent with observations in earlier research. These results underscore the importance of graphene's unique properties, such as its large surface area and functional adaptability, for removing turbidity and color in wastewater [8].

For COD optimization, the model demonstrated an adjusted R-squared value of 0.8930, with significant effects from pH ($p = 0.0017$), graphene content ($p = 0.0413$), and their interaction ($p = 0.0001$). The interaction term (AB) had the most substantial influence, indicating synergistic effects between pH and graphene content, which have been emphasized in previous studies on advanced materials for wastewater treatment. These results corroborate findings by Maleki *et al.* (2016), who reported that optimizing graphene dosage and operational parameters, such as pH, significantly enhances COD removal efficiency [9]. Overall, this study builds upon existing research, offering quantitative insights into the potential of graphene-based materials for industrial wastewater treatment, particularly for the sugar industry, where optimizing key parameters can lead to substantial improvements in treatment performance.

Table 2. ANOVA table for wastewater from sugar industry

Optimization of Turbidity						Optimization of Color					
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	1697.38	5	339.48	27.17	0.0012	Model	2.729E+005	5	54583.58	76.75	0.0001
A-pH	107.95	1	107.95	8.64	0.0323	A-pH	30530.67	1	30530.67	42.93	0.0012
B-Graphene Content	739.70	1	739.70	59.20	0.0006	B-Graphene Content	1.922E+005	1	1.922E+005	270.33	<0.0001
AB	483.56	1	483.56	38.70	0.0016	AB	2970.25	1	2970.25	4.18	0.0964
A ²	229.61	1	229.61	18.38	0.0078	A ²	463.50	1	463.50	0.65	0.4562
B ²	234.19	1	234.19	18.74	0.00075	B ²	45810.70	1	45810.70	64.42	0.0005
Residual	62.48	5	12.50			Residual	3555.71	5	711.14		
Lack of Fit	62.48	3	20.83			Lack of Fit	3555.71	3	1185.24		
Pure Error	0.000	2	0.000			Pure Error	0.000	2	0.000		
Cor Total	1759.85	10				Cor Total	2.765E+005	10			
R-Squared	0.9645					R-Squared	0.9871				
Adj R-Squared	0.9290					Adj R-Squared	0.9743				
Pred R-Squared	0.7344					Pred R-Squared	0.8656				
Adeq Precision	16.929					Adeq Precision	25.421				

Optimization of COD

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	7.475E+006	3	2.492E+006	28.81	0.0003
A-pH	2.102E+006	1	2.102E+006	24.30	0.0017
B-Graphene Content	5.382E+005	1	5.382E+005	6.22	0.0413
AB	4.836E+006	1	4.836E+006	55.92	0.0001
Residual	6.053E+005	7	86477.28		
Lack of Fit	6.053E+005	5	1.211E+005		
Pure Error	0.000	2	0.000		
Cor Total	8.081E+006	10			
Adj R-Squared	0.8930				
Pred R-Squared	0.6927				
Adeq Precision	19.075				

Optimization of Adsorption Performance Towards Wastewater from Sugar Factory Treatment Via Graphene from Pressmud

Figure 3(A-C) illustrate the 2D response surface plots showing the relationship between pH, graphene content, and the removal of turbidity, color, and COD. For turbidity optimization (Figure 3A), a clear pattern emerges: increasing graphene dosage, coupled with an optimal pH of 7.5, leads to a notable maximum removal efficiency of 96.45%. The interaction between pH and graphene content (AB term, $p = 0.0016$) plays a key role, as reflected by the high R^2 value of 0.9645. Similarly, color removal (Figure 3B) follows a strong linear trend, reaching a peak efficiency of 98.71% at a graphene dosage of 0.5 g and pH 7.5. The quadratic effect of graphene dosage ($p < 0.0001$) further enhances performance, validated by the model's exceptional R^2 value of 0.9871. COD removal, however, exhibits a more complex behavior (Figure 3C), with nonlinear interactions and a sharper curvature in the response surface. The highest COD removal efficiency of 92.51% is achieved under the same optimal conditions, driven by a significant interaction term (AB, $p = 0.0001$). While reliable ($R^2 = 0.9251$), the COD model is more sensitive to parameter variations, emphasizing the need for precise calibration to optimize this outcome.

In contrast, Figures 3(D-F) provide a 3D visualization of the interactions, offering additional insights into the dynamic relationships between the variables. The turbidity plot (Figure 3D) reveals a distinct peak at the optimal conditions, demonstrating the critical sensitivity of turbidity removal to changes in both pH and graphene dosage. Any deviation from these conditions results in a steep decline in performance. On the other hand, the color removal plot (Figure 3E) displays a broader and more gradual surface, reflecting greater flexibility in operational conditions while maintaining high removal efficiencies. For COD (Figure 3F), the 3D plot highlights a sharp ridge, emphasizing the heightened sensitivity of COD removal to even small adjustments in pH or graphene content. This narrow operational window underscores the importance of precise control for effective COD reduction. Overall, the findings highlight that while turbidity and color removal exhibit more predictable trends and operational flexibility, COD removal requires a more carefully tuned approach due to its intricate parameter interactions. The results validate the effectiveness of graphene-based treatments, achieving removal efficiencies of 96.45%, 98.71%, and 92.51% for turbidity, color, and COD, respectively, under optimal conditions.

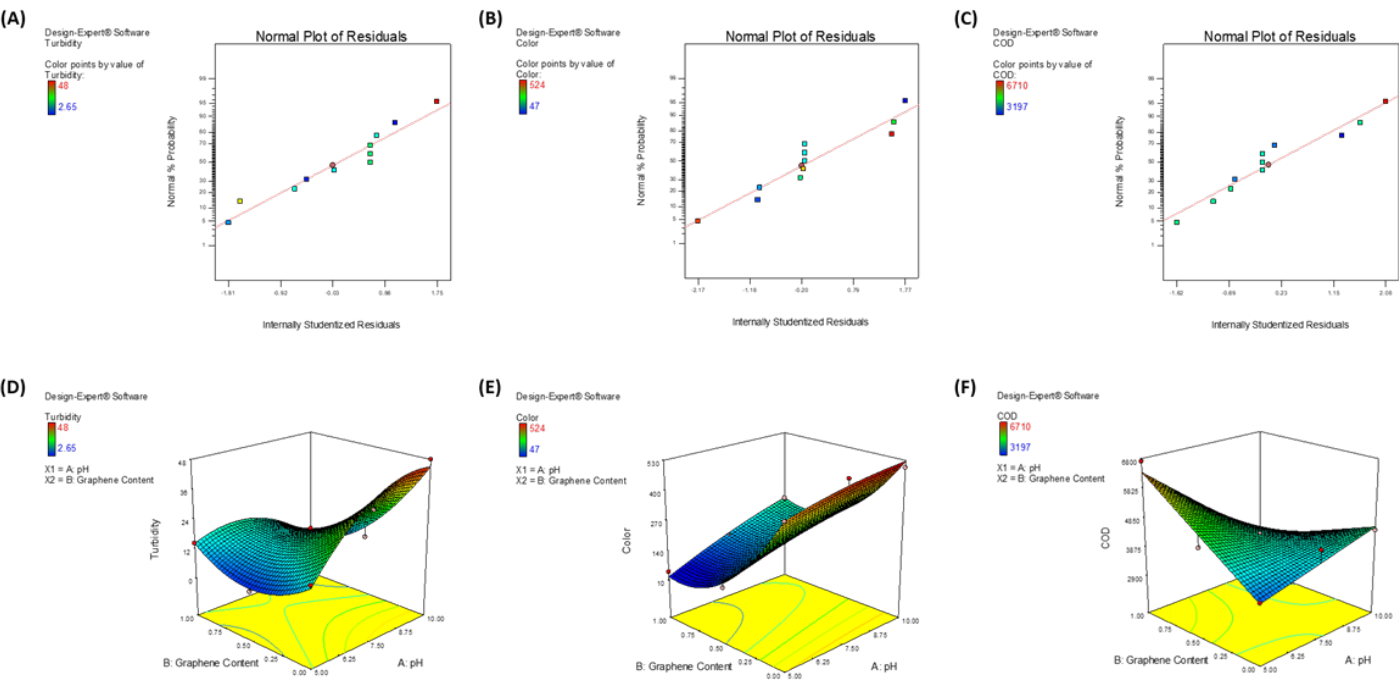


Figure 3. Predicted vs. actual yield plot (A-C) and surface plot (D-F) of adsorption performance towards wastewater from sugar factory treatment via graphene from pressmud

Table 3 highlights how pressmud-derived graphene compares to other common adsorbents used in wastewater treatment. It shows strong performance, removing over 96% of turbidity, nearly 99% of color, and more than 92% of COD using only 0.5 g/L of material. Notably, the treatment cost is relatively low estimated between RM 0.45 and RM 0.60 per cubic meter of wastewater. In comparison, both commercial activated carbon and rice husk-based carbon require higher dosages (1–2 g/L) and come with higher costs, ranging from RM 1.20 to RM 2.00 per cubic meter. While commercial graphene achieves excellent removal efficiencies, its price exceeding RM 300 per gram makes it impractical for widespread use. Pressmud-derived graphene, on the other hand, is produced from an agricultural waste material that is readily available and essentially free, making it an economical and environmentally sustainable option. This approach not only provides a high-performing adsorbent but also supports the concept of turning industrial waste into valuable resources (Safian *et al.*, 2020; Arumugham *et al.*, 2023).

Table 3. Comparison of cost-performance ratio of pressmud-derived graphene vs. other adsorbents

Adsorbent Type	Approximate Cost	Typical Dosage Required (g/L)	Average Removal Efficiency (%)	Estimated Treatment Cost (RM/m³)	Reference
Commercial activated carbon	RM 30–50 per kg	1–2 g/L	Turbidity: 80–90%, Color: 85–95%, COD: 75–90%	~RM 1.50–2.00	Safian <i>et al.</i> (2020) [4], Arumugham <i>et al.</i> (2023) [1]
Rice husk-derived activated carbon	RM 20–40 per kg	1–2 g/L	Turbidity: 85–95%, Color: 80–90%, COD: 70–85%	~RM 1.20–1.80	Safian <i>et al.</i> (2020) [4]

Adsorbent Type	Approximate Cost	Typical Dosage Required (g/L)	Average Removal Efficiency (%)	Estimated Treatment Cost (RM/m ³)	Reference
Commercial graphene	>RM 300 per gram	0.1–0.5 g/L	Turbidity: 95–99%, Color: 95–99%, COD: 90–98%	>RM 10–15	Arumugham <i>et al.</i> (2023) [1]
Pressmud-derived graphene	~RM 50–80 per kg	0.5 g/L	Turbidity: 96.45%, Color: 98.71%, COD: 92.51%	~RM 0.45–0.60	This study

Conclusions

This study demonstrated the effective use of graphene derived from pressmud for treating sugar industry wastewater, achieving optimal removal efficiencies of 96.45% for turbidity, 98.71% for color, and 92.51% for COD at a graphene dosage of 0.5 g and pH 7.5. The optimization process, guided by response surface methodology (RSM), confirmed these parameters as the most effective for maximizing treatment performance, with high R^2 values of 0.9645 for turbidity, 0.9871 for color, and 0.9251 for COD, indicating robust model reliability. Notably, COD removal exhibited heightened sensitivity to the interaction between pH and graphene dosage, emphasizing the importance of precise calibration. These results highlight the superior adsorption capacity of graphene, attributed to its large surface area and functional adaptability, in addressing complex industrial wastewater challenges. Furthermore, by converting pressmud, a sugar industry by-product, into high-value graphene, this study offers a sustainable and cost-effective solution for environmental remediation and resource-efficient wastewater management.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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