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Removal of Dyes from Textile Wastewater Using Modified Eggshell Powder: Optimization and Application

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Abstract

This study investigates the potential of chemically modified eggshell powder (ESP) as a low-cost bio-adsorbent for the removal of methylene orange (MO) dye. Eggshells were treated with hydrochloric acid to enhance their adsorption properties, and the modified ESP was characterized using Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM). The adsorption study was conducted by varying contact time, adsorbent dosage, and initial dye concentration to determine the optimum operating conditions. The optimum conditions were identified as 75 minutes contact time, 1.5 g/L adsorbent dosage, and 90% initial MO concentration, achieving a maximum colour removal efficiency of 61.77% for synthetic dye solution. When applied to real textile wastewater, the process attained 39.14% colour removal, reducing the colour intensity from 327 ADMI to 199 ADMI, which complies with the Malaysian Standard B discharge limit. The findings demonstrate that modified ESP is a viable, eco-friendly, and cost-effective adsorbent for treating dye-laden textile effluent, contributing to sustainable wastewater management and waste valorisation.

1. Introduction

Textile industry has become the largest processing industry in Asia and in the world to produce wastewater (13). Consequently, water pollution has emerged as an increasingly serious issue, driven by the continuous discharge of industrial wastewater into the environment. Among the numerous pollutants discharged, synthetic dyes used in textile production are especially problematic. Textile industry utilizes a large volume of potable water and a wide range of synthetic chemicals at different stages during the textile production process (9). Azo dyes, which contain one or more azo groups structurally, are the largest class (above 60%) among the various groups of textile dyes and the most widely used dyes in the textile industry (2).

Methyl orange (MO) being the most widely produced and industrially used synthetic dyes (3). MO is an anionic azo dye due to the presence of chromophore groups (–N=N–) in its chemical structure (15). Due to their specific features, azo dyes are not or hardly biodegradable and resistant to light, heat, and oxidizing agents, which makes it difficult to remove them in wastewater treatment plants (14). These dyes in textile industry harms aquatic life, soil and human health due to their potential as an environmental pollutant (12). Therefore, dye-containing wastewater should be effectively treated using eco-friendly technologies to avoid negative effects on the environment, human health, and natural water resources (2).

Conventional dye removal methods, including chemical precipitation, membrane filtration, and activated carbon adsorption, are widely used but often involve high costs and complex processes, making them unsuitable for low dye concentrations or resource-limited facilities (10). Among available treatment techniques, adsorption has gained prominence due to its effectiveness in removing anionic dyes and its economic advantages (4)(15).

This has led to increased interest in developing low-cost, sustainable adsorbents from natural and waste-derived materials, which offer efficient pollutant removal and cost-effective solutions for textile wastewater treatment (6).

Eggshells, a by-product of the poultry industry, are promising low-cost adsorbents for wastewater treatment due to their abundance and environmental benefits (1). Eggshell powder (ESP) has shown effectiveness in removing dyes such as methyl orange, attributed to its porous structure and calcium carbonate–rich composition (~96%) (11)(16). Chemical modification can further enhance the surface properties and adsorption capacity of ESP. However, limited studies have assessed the performance of modified ESP for methyl orange removal from real textile wastewater, indicating the need for further optimization and evaluation.

2. Material and methods

2.1 Material

Eggshell waste was used as the primary raw material in this study and was collected from local households. The calcium carbonate ($CaCO_3$) in the eggshells makes them favourable for use in adsorption applications [17]. Eggshell powder (ESP) was chemically modified with hydrochloric acid (HCl) to improve the surface characteristics of the powder. Methylene orange (MO) dye was chosen as a representative contaminant for the simulation of textile wastewater, as it is one of the most commonly used dyes in the textile industry and is very visible in wastewater. All the chemicals and reagents used in this study were of analytical grade and were used as received without any purification. Distilled water was used in all the experiments.

2.2 Preparation of Modified Eggshell Powder (ESP)

Eggshell waste was collected from local households, washed with tap water, boiled to remove organic residues, and air-dried at room temperature for 24 h. The cleaned eggshells were then chemically modified using 0.1 M hydrochloric acid (HCl), prepared by diluting concentrated HCl (37% w/w) with distilled water to a final volume of 7 L. Approximately 1 kg of eggshells was immersed in the acid solution for 2 hours at room temperature with occasional stirring, following standard safety procedures in a fume hood. After treatment, the eggshells were oven-dried at 105 °C for 3 hours, crushed, ground, and sieved to obtain a uniform particle size of ~300 μm, then stored in airtight containers for subsequent adsorption experiments.

2.3 Characterization of ESP

2.3.1 Fourier Transform Infrared (FTIR)

FTIR analysis was used to identify functional groups on modified ESP before adsorption. ESP was collected, oven-dried at 105 °C to remove moisture, and ground into a fine powder. A small amount was placed on the FTIR sample holder, and the spectrum was recorded from 4000–400 cm^{-1}. The resulting spectrum revealed the key functional groups responsible for adsorption.

2.3.2 Field Emission Scanning Electron Microscopy (FESEM)

FESEM was used to examine the surface morphology of modified eggshell powder (ESP) before and after MO adsorption. Analysis before adsorption evaluated the initial surface texture, particle distribution, and porosity, while post-adsorption analysis assessed morphological changes resulting from dye uptake. Both fresh and spent ESP samples were oven-dried at 105 °C, mounted on aluminium stubs with conductive carbon tape, and gold-coated to enhance conductivity. FESEM images were captured at magnifications of 100×, 5000×, and 10000× to analyse surface features, pore structure, and dye deposition, with comparisons made to identify changes such as pore blockage and surface coverage (17).

2.4 Response Surface Methodology (RSM)

RSM based on the CCD was employed to analyse and optimise the batch adsorption process for the removal of MO using ESP. CCD was selected due to its ability to evaluate the individual and interactive effects of process variables and to develop a quadratic model with a reduced number of experimental runs.

The independent variables investigated were adsorbent dosage (X_1), contact time (X_2), and initial MO concentration (X_3). The responses evaluated were percentage colour removal (Y_1) and final colour intensity (Y_2). The factors, symbols, range and levels used in the CCD are summarised in Table 1.

The CCD generated a total of 20 experimental runs, including centre point replicates to assess experimental variability and model adequacy. The experimental data were analysed using Design-Expert to develop a second-order polynomial model describing the relationship between the independent variables and the responses.

Table 1 *Experimental ranges and levels of independent variables*

Independent Variables	Symbol	Range and Level		
		-1	0	+1
Adsorbent Dosage	X1	0.5	1.5	2.5
Contact Time	X2	30	75	120
Initial Concentration	X3	80	90	100

2.5 Batch Adsorption Experiment

Batch adsorption experiments were conducted to evaluate the colour removal efficiency of MO using ESP as an adsorbent under simulated and real wastewater conditions. To assess adsorption behaviour, key parameters including contact time, adsorbent dosage, and initial dye concentration were systematically varied, while all experiments were performed at room temperature (25–30 °C) and at the natural pH of the MO solution without adjustment. Based on preliminary trials, a Central Composite Design (CCD) under Response Surface Methodology (RSM) was employed to optimize ESP dosage.

For each run, 100 mL of MO solution at different dilution levels was prepared in 250 mL conical flasks where the 100% MO solution, no dilution was performed, whereas for 90% and 80% solutions, dilution was carried out by mixing 90 mL or 80 mL of MO solution with 10 mL or 20 mL of distilled water, respectively, to make up 100 mL of sample, and the initial colour intensity was measured using a UV–Vis spectrophotometer with the ADMI program. A known mass of ESP was then added, and the mixtures were agitated at 200 rpm with samples collected at predetermined intervals for analysis (5). Colour removal efficiency was determined by comparing initial and final colour intensities using Equation 1, and all measurements were performed in triplicate. The optimized conditions obtained from the CCD study were subsequently applied to real textile wastewater to validate the model and evaluate practical performance, with colour intensity measured before and after treatment to confirm the applicability and reliability of the optimized adsorption process.

$$Colour\ Removal\ (\%) = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

3. Results and Discussion

3.1 Characterization of ESP

3.1.1 FTIR Analysis

Fig. 1 FTIR spectrum of HCl-modified eggshell powder before adsorption confirmed that the material remained predominantly composed of calcium carbonate ($CaCO_3$) after acid treatment. A strong absorption peak at 1403.56 cm^{-1} corresponds to the asymmetric stretching of CO_3^{2-}, while peaks at 872.50 cm^{-1} and 712.09 cm^{-1} are attributed to the out-of-plane and in-plane bending vibrations of carbonate ions, characteristic of calcite. Additional minor peaks indicate slight structural changes due to acid modification, and the peak at 454.43 cm^{-1} is associated with Ca–O bending, confirming the inorganic nature of the material. The relatively low number of FTIR peaks reflects the limited functional groups in eggshell powder; although HCl modification did not introduce new functional groups, it likely enhanced surface properties such as porosity and reactivity (5). The presence of carbonate groups provides negatively charged sites that facilitate electrostatic interaction with methylene orange molecules, supporting the suitability of HCl-modified ESP as an effective adsorbent.

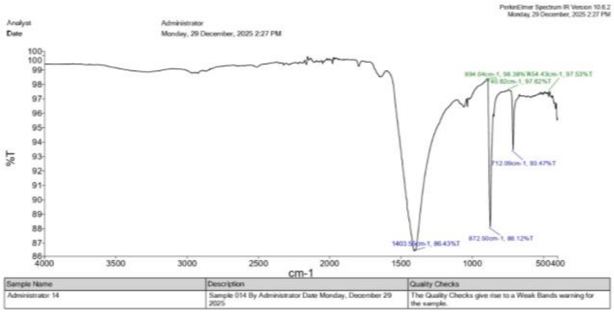


Fig. 1 *ESP Spectrum*

3.1.2 FESEM Analysis

FESEM analysis was conducted to compare the surface morphology of modified eggshell powder (ESP) before and after adsorption of methylene orange (MO), as shown in Fig. 2 and Fig. 3. In Fig. 2(a) at 100× magnification, the surface of ESP before adsorption appears irregular, rough, and heterogeneous with visible cavities. These macroscopic pores indicate a naturally porous structure that is beneficial for adsorption due to increased surface area.

When the magnification increases to Fig. 2(b) at 5000×, the surface texture becomes clearer, showing micro-cracks and small voids distributed across the surface. These micro-voids likely resulted from HCl modification, which enhanced surface roughness and potentially increased the number of active sites available for dye adsorption (7). At even higher magnification in Fig. 2(c) (10000×), the fine porous network is more pronounced. The presence of interconnected pores and irregular structures suggests that the modified ESP provides sufficient adsorption sites for MO molecules. This confirms that acid treatment successfully altered the surface morphology without destroying the structural integrity of $CaCO_3$.

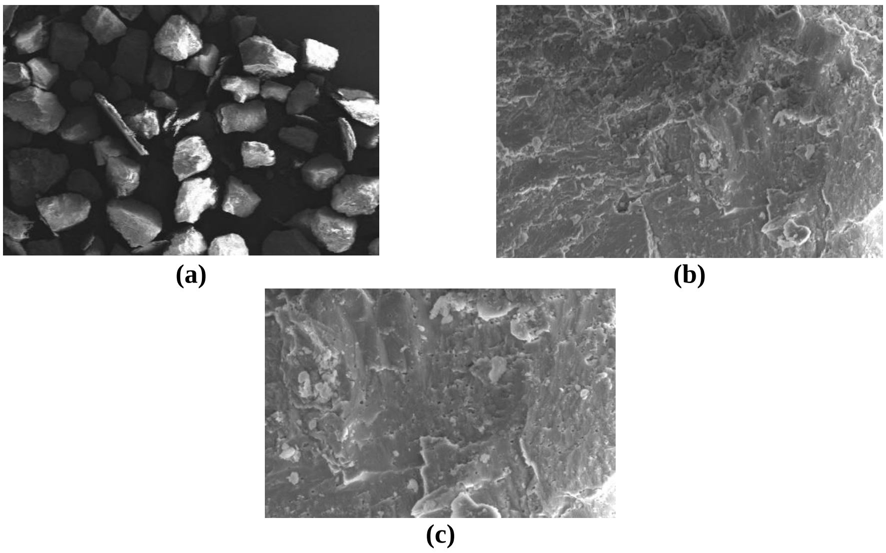
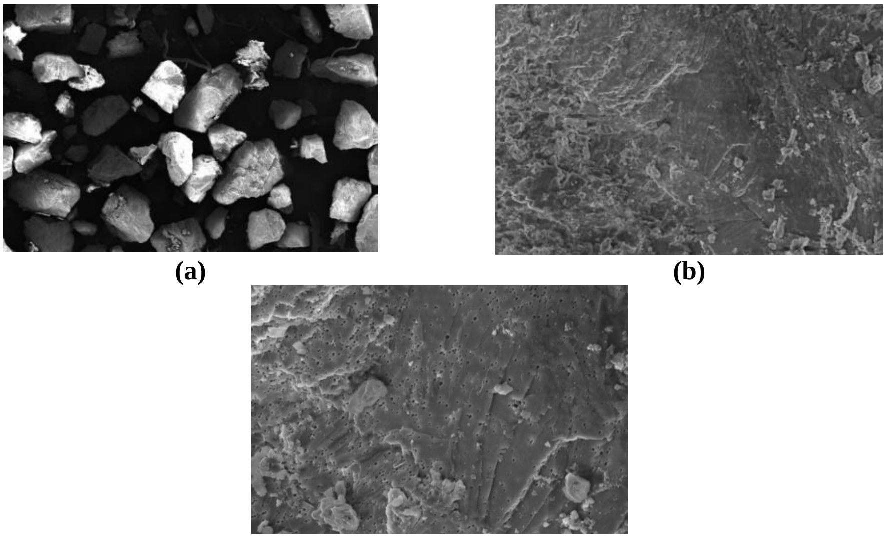


Fig. 2 *FESEM of ESP before adsorption at (a) 100x, (b) 5000x, (c) 10000x magnification*

In contrast, the morphology after adsorption (Fig. 3) shows significant changes. In Fig. 3(a) at 100× magnification, the surface appears denser and less porous compared to Fig. 2(a), indicating possible surface coverage by dye molecules. At 5000× magnification in Fig. 3(b), previously visible micro-cracks and pores appear partially filled or blocked. This suggests that MO molecules were adsorbed onto the surface and occupied available active sites. Finally, in Fig. 3(c) at 10000× magnification, the surface becomes noticeably smoother and more compact compared to Fig. 2(c). This reduction in surface roughness and porosity confirms the successful deposition of methylene orange onto the ESP surface and the occupation of active adsorption sites (7)(8). Overall, comparison between Fig. 2 and Fig. 3 clearly demonstrates morphological changes before and after adsorption, particularly pore blockage and surface smoothing, which provide strong visual evidence of MO adsorption onto modified ESP.



(a) (b)

(c)

Fig. 3 *FESEM of ESP after adsorption at (a) 100x, (b) 5000x, (c) 10000x magnification*

3.2 Experimental and CCD-Predicted Results

The initial MO solution had a colour intensity of 327 ADMI, representing typical dye-containing wastewater. Adsorption experiments were performed under optimised conditions obtained from Response Surface Methodology (RSM), which evaluated the effects of contact time, adsorbent dosage, and initial MO concentration on colour removal efficiency.

After completing the adsorption experiments, the experimental colour removal results were compared with the values predicted by the RSM–CCD model to evaluate its accuracy and reliability. Table 2 presents the experimental and predicted values for colour intensity and colour removal across all 20 runs.

Table 2 *Experimental conditions and results of responses from CCD*

Run	Experimental Variables			Response 1 Colour Intensity (ADMI)		Response 2 Colour Removal (%)	
	Contact Time (min)	Dosage (g/L)	Concentration (%)	Actual	Predicted	Actual	Predicted
1	30	0.5	100	179	177.14	45.26	45.83
2	75	1.5	90	127	137.46	61.16	57.96
3	30	0.5	80	166	167.04	49.24	48.92
4	75	0.5	90	149	141.05	54.43	56.86
5	75	1.5	100	173	159.85	47.09	51.12
6	120	0.5	80	158	161.84	51.68	50.51
7	120	1.5	90	177	152.45	45.87	53.38
8	75	1.5	90	125	137.46	61.77	57.96
9	30	1.5	90	161	155.65	50.76	52.40
10	120	2.5	100	181	187.44	44.65	42.68
11	30	2.5	100	185	188.64	43.43	42.32
12	75	2.5	90	169	147.05	48.32	55.03
13	75	1.5	90	126	137.46	61.47	57.96
14	75	1.5	90	129	137.46	60.55	57.96
15	30	2.5	80	168	170.54	48.62	47.85
16	75	1.5	80	159	142.25	51.38	56.50
17	75	1.5	90	130	137.46	60.24	57.96
18	75	1.5	90	128	137.46	60.86	57.96
19	120	0.5	100	174	178.94	46.79	45.28
20	120	2.5	80	153	162.34	53.21	50.36

Overall, the experimental results closely matched the values predicted by the RSM-CCD model, indicating good model accuracy. For instance, in Run 1, the experimental colour intensity (179 ADMI) and removal efficiency (45.26%) were in close agreement with the predicted values of 177.14 ADMI and 45.83%. Under optimised conditions (Run 8: 75 min contact time, 1.5 g/L ESP dosage, and 90% MO), the experimental colour removal of 61.77% also closely aligned with the predicted value of 57.96%.

Runs conducted at moderate adsorbent dosage (1.5 g/L) and mid-range contact time (75 min) consistently achieved higher colour removal, whereas lower dosages (0.5 g/L) or higher initial concentrations (100%) resulted in reduced efficiency. These findings confirm the reliability of the RSM model and demonstrate that the optimised conditions can be effectively applied for efficient MO removal, supporting the potential use of this adsorption process for practical wastewater treatment.

3.3 Statistical RSM Model Fit

Table 3 shows statistical parameters of the model with mean response of the dataset was 155.85 with a standard deviation of 15.60, resulting in a coefficient of variation (C.V.) of 10.01%. The relatively low C.V. indicates that the experimental data are reasonably precise, and the model provides consistent results. The coefficient of determination (R^2) was 0.712, suggesting that approximately 71.2% of the variability in the response could be explained by the model. However, the adjusted R^2 of 0.453 indicates that the explanatory power decreases after accounting for the number of predictors, implying that some terms may not significantly contribute to the response. Furthermore, the predicted R^2 was negative (-0.368), indicating poor predictive capability for new observations. The adequate precision was 4.64, exceeding the desirable threshold of 4, which demonstrates that the model has an acceptable signal-to-noise ratio and can be used to explore the design space.

The discrepancies observed between experimental and predicted values may be attributed to experimental uncertainties and the inherent complexity of the adsorption process. Overall, while the model captures a moderate portion of the variability, its limited predictive power and lower adjusted R^2 suggest that further refinement, such as including higher-order terms or transformations, may be necessary to improve accuracy and prediction reliability.

Table 3 *RSM model fit summary for response variables*

Statistical figure	Final Colour Intensity (ADMI)	Colour Removal (%)
Std. Dev.	15.60	4.77
Mean	155.85	52.34
C.V. %	10.01	9.12
R^2	0.7119	0.7118
Adjusted R^2	0.4526	0.4525
Predicted R^2	-0.3683	-0.3685
Adequate Precision	4.6386	4.6375

Given that the primary objective of this study was process optimisation rather than precise prediction, the quadratic model was considered suitable for identifying optimal operating conditions and understanding the influence of key variables. Overall, the statistical analysis indicates that the quadratic model provides a useful and interpretable framework for analysing the adsorption process. While its limitations are acknowledged, the model remains adequate for optimisation purposes and offers valuable insight into the effects of contact time, adsorbent dosage, and initial MO concentration on colour removal efficiency.

3.4 Process Optimization and Application to Real Wastewater

The adsorption process was optimised using Response Surface Methodology (RSM) with a Central Composite Design (CCD) to maximise colour removal efficiency by varying contact time, adsorbent dosage, and initial MO concentration. Based on the quadratic regression model and validation runs, the optimal conditions were identified as a contact time of 75 minutes, adsorbent dosage of 1.5 g/L, and initial MO concentration of 90%, with six repeated tests. Under these conditions (Run 8), the highest colour removal efficiency of 61.77% was achieved.

To assess practical applicability, the optimised conditions were applied to real textile wastewater with an initial colour intensity of 327 ADMI. After treatment, the colour intensity decreased to 199 ADMI, corresponding to a colour removal efficiency of approximately 39%. Both the synthetic MO solution and real wastewater achieved final colour intensities below 200 ADMI, meeting the Standard B discharge limit. Although colour removal in real wastewater was lower due to the presence of competing contaminants, the results confirm the effectiveness and potential of modified eggshell powder as a cost-effective and environmentally sustainable adsorbent for textile wastewater treatment.

4. Conclusion and Recommendation

This study successfully demonstrated the potential of chemically modified eggshell powder (ESP) as a sustainable, low-cost bio-adsorbent for the removal of methylene orange (MO) from textile wastewater. HCl-modified ESP exhibited favourable physicochemical properties, including a porous surface morphology and carbonate-based functional groups, as confirmed by FTIR and FESEM analyses, which enhanced its adsorption performance.

Optimisation using Response Surface Methodology (RSM) with Central Composite Design (CCD) identified the optimal adsorption conditions as a contact time of 75 minutes, adsorbent dosage of 1.5 g/L, and initial MO concentration of 90%. Under these conditions, the highest colour removal efficiency of 61.77% was achieved for synthetic MO wastewater, reducing the colour intensity to 125 ADMI. When applied to real textile wastewater, the optimised process achieved a colour removal of 39.14%, lowering the colour intensity from 327 ADMI to 199 ADMI and meeting the Malaysian Standard B discharge limit (<200 ADMI). The results indicate that adsorption efficiency is strongly influenced by contact time, adsorbent dosage, and initial dye concentration, as well as their interactions. Although the quadratic RSM model had some predictive limitations, it proved effective for process optimisation and performance evaluation. Overall, modified ESP was found to be an environmentally friendly, cost-effective, and viable adsorbent for textile wastewater decolourisation, particularly at lower dye concentrations.

For future work, adsorption performance could be further improved by investigating alternative modification techniques such as alkaline or surfactant-assisted treatments, nanomaterial incorporation, or the development of composite adsorbents with other low-cost materials. Studies on adsorbent durability, regeneration, and reusability are also recommended to enhance practical applicability. In terms of modelling, advanced tools such as artificial neural networks could be employed to better capture the non-linear behaviour of adsorption systems, while incorporating additional parameters such as pH, temperature, and agitation rate would provide a more comprehensive understanding of the process. Furthermore, evaluating the performance of modified ESP in continuous or column systems, treating mixed-dye wastewater, and extending its application to other dye classes and pollutants would broaden its industrial relevance and scalability.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Nur Farisha Norhirman Abdul Rahman, Nor Hazren Abdul Hamid;* ***data collection:*** *Nur Farisha Norhirman Abdul Rahman, Nor Hazren Abdul Hamid;* ***analysis and interpretation of results: :*** *Nur Farisha Norhirman Abdul Rahman, Nor Hazren Abdul Hamid;* ***draft manuscript preparation: :*** *Nur Farisha Norhirman Abdul Rahman, Nor Hazren Abdul Hamid. All authors reviewed the results and approved the final version of the manuscript.*

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