

Flocculation Efficiency of Organic and Inorganic Coagulants in Microalgae Bloom Harvesting

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ABSTRACT: The present study investigated the performance of inorganic (alum) and organic (chitosan) coagulants in the flocculation-harvesting of microalgae blooms. Water samples were collected from the eutrophic Gunung Lang Recreational Park, Ipoh, Malaysia. The analysis results indicated moderate contamination, with the early stages of algal bloom suggested by high pH and moderate turbidity. Optimisation of coagulant dosage and pH was carried out through a jar-test experiment and Response Surface Methodology (RSM). The best performance was observed with alum, achieving 98.7% harvesting efficiency at 105 mg/l and pH 8.5, while chitosan reached 86% efficiency at 180 mg/l and pH 5.0. Statistical analysis revealed that pH and dosage significantly impacted flocculation performance. These results demonstrated that alum and chitosan were cost-effective and efficient coagulants. Moreover, chitosan presented a biodegradable alternative, offering environmental sustainability in the long term. This study suggested that flocculation could be a competitive and scalable technique for improving water quality and recovering microalgae biomass, with potential applications in large-scale water treatment.

KEYWORDS: Microalgae harvesting; flocculation; algal blooms; coagulants; water treatment; biomass recovery

1. Introduction

Today, the depletion of natural resources, coupled with the challenges posed by climate change and environmental pollution, increased the stress on food, clean water, and sustainable energy globally [1]. As the world moved into a technologically advanced 21st century, resource scarcity emerged as one of the main economic hurdles to attaining basic physiological needs, as defined by Maslow's Hierarchy [2]. As a solution, microalgae emerged as a promising biological resource due to their rapid growth and high biomass productivity, which could be utilised in various fields such as water treatment, biofuels, and medicine. Microalgae-based technologies aligned with the circular economy by reducing waste and regenerating resources [3].

Harmful algal blooms (HABs), often driven by nutrient-rich waters due to eutrophication, became increasingly frequent, leading to significant water quality degradation and public health risks. In such cases, effective harvesting of microalgae was crucial to mitigate adverse effects. Among various harvesting methods, chemical flocculation offered a promising, cost-effective, and scalable option, involving the addition of coagulants to aggregate algae cells for easier separation from the water [4]. The impacts of HABs were wide-ranging, including degradation of water quality, production of toxins dangerous to humans and animals, and creation of hypoxic zones that impacted aquatic ecosystems. Under conducive conditions such as excess nutrients, warm temperatures, and calm waters, the frequency and intensity of these blooms were predicted to rise, raising grave ecological and public health concerns [5].

For controlling and utilising algal biomass, especially during blooms, effective harvesting technologies were required. Algae could be removed by various methods (centrifugation, filtration, flotation, or gravity settling), but most of these methods were energy-consuming or economically unfeasible for large-scale applications [6]. Chemical flocculation was considered a cost-effective and scalable method for microalgae biomass separation and recovery. This process required the addition of coagulants to form larger flocs of suspended algal cells, which could be more readily separated from water [7]. Although flocculation had several advantages, its efficiency was closely related to parameters such as coagulant type and dosage, pH, ionic strength of the medium, and algal species characteristics. To date, limited comparative studies had been conducted on the performance and optimisation of organic and inorganic coagulants for harvesting microalgae blooms. Alum (aluminum sulfate) was a popular inorganic coagulant, known for its relatively high alkalinity and excellent turbidity removal capability [8]. In contrast, chitosan, a biodegradable cationic polymer derived from chitin, was an environmentally friendly organic flocculant with good flocculation performance due to its high molecular weight and charge density [9].

This study investigated the comparative performance of alum, an inorganic coagulant, and chitosan, an organic and biodegradable coagulant, in harvesting microalgae from eutrophic water bodies. The optimal conditions for maximum flocculation yield were determined using RSM. The physicochemical properties of the bloom-affected lake water were also examined, and key controlling parameters were evaluated. In summary, the present work proposed a potentially useful, effective, and inexpensive method for harvesting algal biomass and improving water quality.

2. Materials and Methods

2.1. Lake water sample collection.

Water samples were obtained from Gunung Lang Recreational Park, Ipoh, Perak (4.6209° N, 101.0867° E), a site known for frequent microalgal blooms resulting from eutrophication [10]. Samples were collected from areas with stable biomass concentrations and low water flow to ensure minimal dilution by rainwater [11].

2.2. Analytical water parameter procedures.

Water quality parameters were assessed to characterize the samples prior to flocculation. Measurements included chemical oxygen demand (COD), turbidity, total suspended solids (TSS), ammonia, dissolved oxygen (DO), pH, and temperature, providing a baseline for

evaluating coagulant performance. COD was determined using conventional digestion with COD vials and analyzed with a DR900 spectrophotometer. Turbidity was measured with a turbidity meter, while TSS was determined by filtering 20 mL of the sample through pre-weighed glass fiber filters, followed by drying and calculating the change in mass. Ammonia levels were measured using the salicylate method with spectrophotometric detection following a 20-minute reaction period. DO, pH, and temperature were measured in situ using a YSI multiparameter probe [11].

2.3. Jar-test procedure.

Jar tests were conducted by adding varying coagulant dosages (30–180 mg/l) to beakers containing lake water. The process involved rapid mixing, followed by slow mixing and subsequent settling. Post-settlement turbidity was measured to evaluate the effectiveness of flocculation. The procedures followed conventional coagulation/flocculation steps, as detailed in Table 1. The pH and coagulant dosages were adjusted according to the RSM design [12]. After settling, the supernatant was carefully withdrawn using a syringe, and turbidity measurements were taken to assess flocculation performance.

Table 1. Characteristics of Jar-test procedure.

Characteristic	Description
Coagulant type	Aluminum sulfate, Chitosan
Dosage range	30–180 mg/l
pH range	5–12
Rapid mixing	3 min at 80 rpm
Slow mixing	20 min at 30 rpm
Settling period	20 min

2.4. Statistical analysis (response surface methodology).

Design-Expert software version 7.0 was used to analyze the effects of pH and coagulant dosage on flocculation efficiency. A Face-Centered Central Composite Design (FCCCD) with two factors, coagulant dosage and pH, was employed. The ranges of the experimental variables are presented in Table 2. Analysis of variance (ANOVA) was conducted to evaluate the statistical significance of each factor and their interactions. Model validation was performed using regression coefficients and residual analysis to ensure the adequacy and predictive capability of the developed model.

Table 2. RSM experimental design matrix.

Std	Dosage (mg/l)	pH
1-3	30	5
4-6	180	5
7-9	30	12
10-12	180	12
13	30	8.5
14	180	8.5
15-16	105	5,12
17-21	105	8.5

Coded variable transformation was done using the equation:

$$x_i = \frac{X_i - X_0}{\Delta X}$$

where x_i is the coded value, X_i is the actual value, X_0 is the center point, and ΔX is the step change.

2.5. Microalgae harvesting efficiency.

Harvesting efficiency was calculated by comparing initial and final biomass concentrations using the following equation [13]:

$$\text{Harvesting Efficiency (\%)} = \left(\frac{A - B}{A} \right) \times 100$$

Where A is Initial biomass (measured via turbidity), and B is Final biomass (post-flocculation turbidity)

3. Results and Discussion

3.1. Analysis of water quality parameters.

Water quality assessment classified the sample as moderately polluted (Class IIB), indicative of early-stage algal blooms. Key parameters included COD (10 mg/l), turbidity (27.7 NTU), TSS (13 mg/l), and DO (5.53 mg/l), all suggesting moderate contamination. The high pH value (9.45) reflected eutrophic conditions conducive to algal growth. These findings highlighted the importance of efficient flocculation in removing microalgae from bloom-affected waters [14]. Although the DO level was below saturation, it was still adequate to support aquatic life; however, its variability reflected ongoing algal photosynthesis and respiration processes [15]. The lake water temperature (28.5 °C) was suitable for algal growth, and the elevated pH further confirmed eutrophication due to the bloom [16]. These baseline conditions provided critical context for evaluating the performance of alum and chitosan under high-nutrient, alkaline conditions.

Table 3. Water parameters result and water quality classification.

Water Parameter	Value (\pm SD)	Unit	NWQS Water Quality Class*
Chemical Oxygen Demand	10.0 \pm 0.50	mg/l	Class II (\leq 10 mg/l)
Turbidity	27.7 \pm 4.04	NTU	Class IIA/IIB (\leq 50 NTU)
Total Suspended Solids	13.0 \pm 3.02	mg/l	Class I (\leq 25 mg/l)
Ammonia (NH ₃ -N)	0.05 \pm 0.43	mg/l	Class I (\leq 0.1 mg/l)
Dissolved Oxygen (DO)	5.53 \pm 6.30	mg/l	Class II (5–7 mg/l)
pH	9.45 \pm 2.30	–	Above Class IIA/B range (6–9)
Temperature	28.5 \pm 0.56	°C	Not classified under NWQS

*Note: Classification based on Department of Environment Malaysia, National Water Quality Standards (NWQS); classification ranges may vary slightly by application (e.g., drinking, recreational, aquatic life).

3.2. Algae bloom flocculation coagulation.

Flocculation experiments (Tables 4 and 5) revealed that alum achieved the highest harvesting efficiency of 98.7% at a dosage of 105 mg/l and pH 8.5. Chitosan, on the other hand, reached a maximum efficiency of 86% at 180 mg/l and pH 5.0. The comparison highlighted alum's superior efficiency at a lower dosage, making it more cost-effective. In contrast, chitosan, despite requiring a higher dosage, offered a biodegradable and environmentally friendly alternative. The results also emphasized the significant influence of pH and dosage on flocculation efficiency. For alum, optimal performance occurred under slightly alkaline

conditions (pH 8.5), whereas chitosan performed best under acidic conditions (pH 5.0). These findings aligned with previous studies suggesting that pH strongly affects the flocculation mechanism, with different coagulants exhibiting distinct responses to pH changes. The initial turbidity of the lake water used in the experiment was 27.7 NTU, which was considered a moderate level, corresponding to the early to mid-development phase of algal blooms [17]. This provided a reliable and practical basis for evaluating the performance of both coagulants under varied pH and dosage conditions. Overall, the findings underscored the crucial role of pH and dosage in flocculation-based harvesting and the pivotal contribution of their interaction to the success of the treatment process.

Table 4. Flocculation using alum.

Std	Factor 1: Coagulant Dosage	Factor: 2 pH	Harvesting Efficiency (%)
1	30.00	5.00	65.8
2	30.00	5.00	81.2
3	30.00	5.00	61.5
4	180.00	5.00	54.3
5	180.00	5.00	54.7
6	180.00	5.00	50.8
7	30.00	12.00	66.5
8	30.00	12.00	67.1
9	30.00	12.00	71.6
10	180.00	12.00	86.1
11	180.00	12.00	85.8
12	180.00	12.00	81.1
13	30.00	8.50	92.3
14	180.00	8.50	94.2
15	105.00	5.00	58.2
16	105.00	12.00	62.5
17	105.00	8.50	96.8
18	105.00	8.50	97.6
19	105.00	8.50	90.8
20	105.00	8.50	98.1
21	105.00	8.50	98.7

Table 5. Flocculation using chitosan.

Std	Factor 1: Coagulant Dosage	Factor: 2 pH	Harvesting Efficiency (%)
1	30.00	5.00	66.5
2	30.00	5.00	68
3	30.00	5.00	67.1
4	180.00	5.00	71.2
5	180.00	5.00	83.5
6	180.00	5.00	86
7	30.00	12.00	37.7
8	30.00	12.00	38.8
9	30.00	12.00	49.6
10	180.00	12.00	46.6
11	180.00	12.00	51
12	180.00	12.00	61.5
13	30.00	8.50	29.9
14	180.00	8.50	39.7
15	105.00	5.00	78.1
16	105.00	12.00	52.3
17	105.00	8.50	26.4
18	105.00	8.50	30.7
19	105.00	8.50	24.5
20	105.00	8.50	28.5

The solubility and charge density of the polymer were higher at low pH, which enhanced the flocculation performance of chitosan. Alum, on the other hand, performed better under neutral to slightly alkaline pH conditions due to the formation of aluminum hydroxide flocs,

which effectively entrapped and settled microalgae cells [18, 19]. These results were consistent with previous studies, underlining that the choice of coagulant and the optimization of the treatment process should be based on water chemistry and the desired performance outcomes [20].

3.3. Statistical analysis of alum and chitosan.

The statistical significance of the model equation for alum was determined using the F-test for ANOVA) (Table 6). With an F_{value} of 33.05 and a p_{value} of 0.0001, the model was found to be statistically significant. The ‘lack of fit’ F_{value} was 2.12 ($p_{\text{value}} > 0.05$), indicating that the model fit the experimental data well and was accurate. The signal-to-noise ratio, measured using the adequacy of precision, was 15.13, which was well above the minimum threshold of 4, confirming that the model had an adequate signal and could be used to navigate the design space. Among the regression terms, the linear term B (pH) significantly influenced flocculation efficiency ($p_{\text{value}} < 0.0001$), while the linear term A (coagulant dosage) was not significant ($p_{\text{value}} = 0.9619$) (Table 6). The interaction term between coagulant dosage (A) and pH (B) was also significant ($p_{\text{value}} < 0.05$). For the quadratic terms, B^2 was significant ($p_{\text{value}} < 0.0001$), whereas A^2 was not ($p_{\text{value}} = 0.3157$). The determination coefficient (R^2) was 0.9178, indicating a strong correlation between the independent variables and the response. The predicted R^2 (0.8251) was in good agreement with the adjusted R^2 (0.8904), confirming the reliability of the model (Table 7).

Table 6. Analysis of variance of the regression model for flocculation efficiency of alum.

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	5060.19	5	1012.04	33.50	< 0.0001	significant
A-Coagulant Dosage	0.071	1	0.071	2.365E-003	0.9619	
B-pH	633.83	1	633.83	20.98	0.0004	
AB	776.02	1	776.02	25.69	0.0001	
A^2	32.55	1	32.55	1.08	0.3157	
B^2	2768.89	1	2768.89	91.66	< 0.0001	
Residual	453.12	15	30.21			
Lack of Fit	156.93	3	52.31	2.12	0.1511	not significant
Pure Error	296.19	12	24.68			
Cor Total	5513.31	20				

Table 7. Variance analysis using response surface methods for the parameters of the second order polynomial equation for alum.

Parameter	Value	Parameter	Value
Std. Dev.	5.5	R^2	0.9178
Mean	76.94	Adj R^2	0.8904
C.V. %	7.14	Pred R^2	0.8251
PRESS	964.24	Adeq Precision	15.131

For chitosan, the model was statistically significant, with an F_{value} of 48.94 and a p_{value} of < 0.0001. The F_{value} for ‘lack of fit’ was 1.26 ($p_{\text{value}} > 0.05$), indicating that the model was consistent with the experimental data. The adequacy of precision, which measures the signal-to-noise ratio and must exceed 4 for a reliable model, was 19.023—demonstrating that the model generated by the Box–Behnken design was suitable for navigating the design space. Both linear regression terms, A (coagulant dosage) and B (pH), significantly influenced flocculation efficiency, with p_{value} of 0.0013 and < 0.0001, respectively (Table 8). The interaction between coagulant dosage (A) and pH (B) was also significant ($p_{\text{value}} < 0.05$),

contributing meaningfully to the model. Among the quadratic terms, B^2 was significant ($p_{\text{value}} < 0.0001$), whereas A^2 was not ($p_{\text{value}} = 0.8052$). The determination coefficient (R^2) was 0.9422, indicating a strong correlation between the independent variables and the response—slightly higher than that of the alum model. The predicted R^2 (0.8782) was in good agreement with the adjusted R^2 (0.9230), confirming the robustness and predictive accuracy of the model (Table 9).

Table 8. Analysis of variance of the regression model for flocculation efficiency of chitosan.

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	7544.10	5	1508.82	48.94	< 0.0001	significant
A-Coagulant Dosage	479.12	1	479.12	15.54	0.0013	
B-pH	2389.46	1	2389.46	77.51	< 0.0001	
AB	3.10	1	3.10	0.10	0.7555	
A^2	1.94	1	1.94	0.063	0.8052	
B^2	3056.71	1	3056.71	99.15	< 0.0001	
Residual	462.44	15	30.83			
Lack of Fit	110.58	3	36.86	1.26	0.3329	not significant
Pure Error	351.87	12	29.32			
Cor Total	8006.55	20				

Table 9. Variance analysis using response surface methods for the parameters of the second order polynomial equation for chitosan.

Parameter	Value	Parameter	Value
Std. Dev.	5.55	R^2	0.9422
Mean	50.73	Adj R^2	0.9230
C.V. %	10.94	Pred R^2	0.8782
PRESS	975.12	Adeq Precision	19.023

The use of RSM enabled the optimization of coagulant dosage and pH for maximum flocculation efficiency. ANOVA results confirmed the statistical significance of the models for both alum ($F_{\text{value}} = 33.05$, $p_{\text{value}} < 0.0001$) and chitosan ($F_{\text{value}} = 48.94$, $p_{\text{value}} < 0.0001$). The regression models showed a good fit with the experimental data, as reflected by high R^2 values (0.9178 for alum and 0.9422 for chitosan), indicating strong predictive reliability. Both models also revealed significant interactions between coagulant dosage and pH, which influenced flocculation efficiency. For both coagulants, increasing pH and dosage initially improved efficiency, followed by a decline when conditions deviated from the optimal values.

The three-dimensional (3D) response surface plots were used to visualize the effects and determine the optimal values of coagulant dosage and pH for maximum microalgae harvesting efficiency (Figure 1). These plots illustrated how each variable influenced flocculation efficiency along the Z-axis. Figure 1A displays the interaction between pH and coagulant dosage for alum, where efficiency increased with both parameters initially, then declined beyond the optimal point. In contrast, Figure 1B, showing chitosan, revealed a different trend: harvesting efficiency initially decreased with rising pH and dosage before increasing again.

The optimum flocculation efficiency for alum (Figure 1A) was achieved at pH 8.5 and a dosage of 105 mg/l, resulting in 98.7% efficiency. For chitosan (Figure 1B), the optimal condition was at pH 5.0 and 180 mg/l, achieving a maximum efficiency of 86%, which is lower than that of alum. These findings align with previous studies by Liu et al. [21] and Zhu et al. [22], which also highlighted the importance of pH and coagulant dosage in determining harvesting efficiency. The normal probability plots for both alum and chitosan (Figure 2)

showed that the data points followed a straight line, indicating that the residuals were normally distributed and validating the model assumptions.

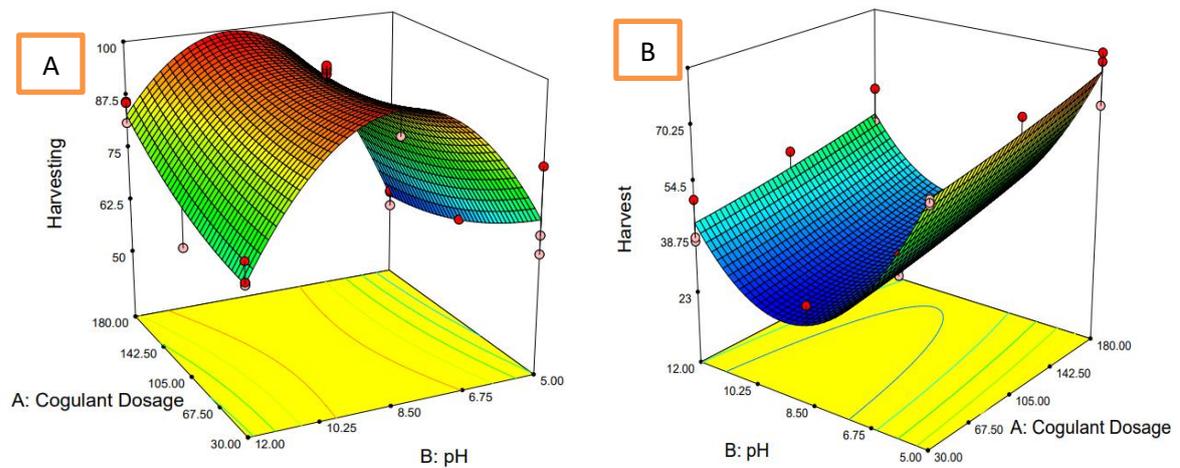


Figure 1. Design expert plot for 3D response surface for harvesting efficiency using alum (A) and chitosan (B).

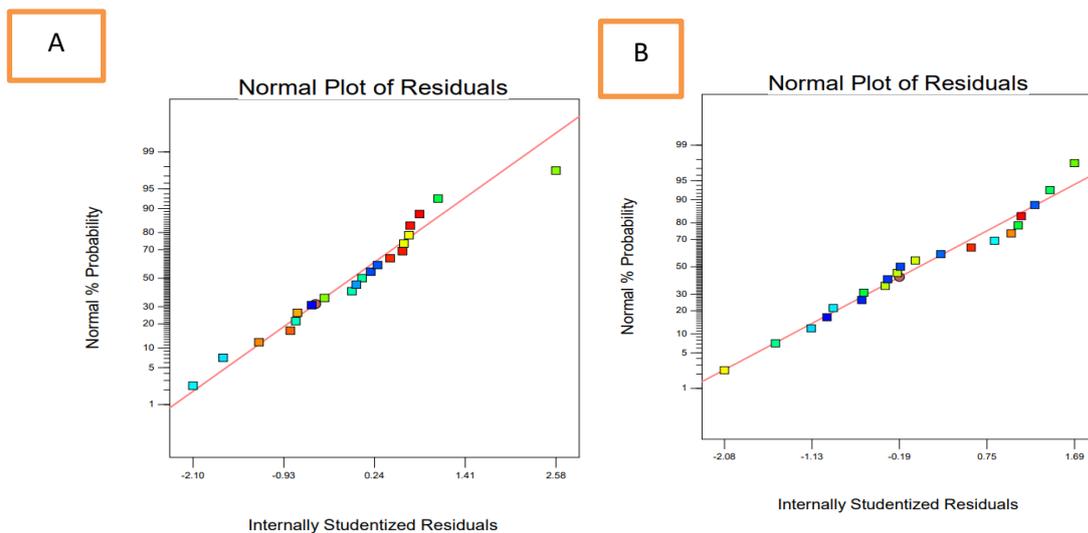


Figure 2. Design expert plot; normal probability plot of the internally standardized residual for harvesting efficiency using alum (A) and chitosan (B).

Previous study has shown that the coagulation process can be hindered when suboptimal particle formation occurs in the solution. When the pH is too low, the coagulation process may not proceed effectively, while excessively high pH levels can lead to the redispersion of coagulated particles. pH also influences the size of coagulated particles, which in turn affects the density of the resulting floc and its tendency and rate of settling [23]. This is particularly relevant in the context of microalgal aggregation after the completion of sedimentation. For both chitosan and alum, it was observed that higher pH levels tend to destabilize the flocs, causing dispersion, whereas lower pH levels promote aggregation, facilitating more effective microalgae harvesting.

4. Conclusion

This study demonstrates the potential of flocculation as an effective and scalable method for harvesting microalgae from eutrophic, bloom-affected waters. Alum was identified as the more efficient and cost-effective coagulant, while chitosan provided a biodegradable and environmentally sustainable alternative. The findings highlight the critical role of optimizing coagulant dosage and pH to achieve maximum flocculation efficiency. Both coagulants show promising potential for large-scale applications in water treatment and algal biomass recovery. Future research should focus on the development of hybrid coagulant systems and pilot-scale implementations to further improve the environmental and economic sustainability of microalgae harvesting technologies.

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Author Contribution

All authors contributed equally to all aspects of the work and approved the final version of the manuscript.

Competing Interest

All authors declared no competing interest.

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