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Optimization of Coagulation–Flocculation Treatment for Fish Farm Effluent Using Green Coagulants and Recovery of the Produced Sludge

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Abstract

Treatment of wastewater effluent is essential to reduce environmental impact and keep surface water clean, meeting sustainable criteria. While plant-based coagulants are known for their eco-friendly profiles, their dual application for high-efficiency nutrient removal and subsequent sludge valorization in fish farm systems remain under-explored. Therefore, this study was conducted to determine the optimum conditions for using natural coagulants to recover nutrients from fish farm effluent. Two types of natural coagulants, *Alhagi graecorum* leaves and apricot seeds, were evaluated for the treatment and recovery of nutrients from fish farm effluent due to their high removal efficiency, non-toxicity, and cost-effectiveness. In this study, optimization was performed using Response Surface Methodology (RSM) with a Central Composite Design (CCD) to investigate the effects of three factors: coagulant concentration (1000–7000 mg/L), wastewater pH (5–9), and settling time (15–35 min). The primary responses measured were the removal efficiencies of phosphate (PO_4) and nitrate (NO_3). According to the CCD results, maximum removal efficiencies reached 92.63% and 73.49% for PO_4 and NO_3, respectively. The optimal conditions were identified as pH 5, 1000 mg/L coagulant concentration, and a 35 min settling time for *A. graecorum*, and pH 9, 1000 mg/L concentration, and a 15 min settling time for apricot seed. These findings establish the optimal conditions for using these natural substances as effective agents for sustainable wastewater treatment and nutrient recovery.

Keywords: coagulation; environmental pollution; flocculation; natural coagulant; nutrients recovery

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1. Introduction

Wastewater treatment is accomplished by physical methods such as filtration, settling, adsorption, and membrane filtration; chemical methods such as electrochemical, coagulation, oxidation, ion exchange, and disinfection; and biological methods such as microbial biodegradation, phytoremediation, constructed wetlands, and bioreactor digestion [1]. Coagulation and flocculation (CF) are a simple, dependable, and energy-efficient water treatment method. Natural green coagulant is an environmentally friendly treatment that leads to the recovery of materials [2]. There are various advanced physical, chemical, and biological treatment methods to remove pollutants from water. Coagulation–flocculation, electrocoagulation, adsorption, advanced oxidation processes, and biomembrane technology are advanced methods adopted in water and wastewater treatment [3]. The coagulation–flocculation method is a suitable treatment technology for wastewater from aquatic waste due to its high removal efficiency, simplicity, and economic savings [4]. Coagulation involves chemical and physical processes that destabilize pollutants when natural coagulants are added, and flocculation then stimulates the aggregation of destabilized particles, forming large flakes that can settle [5]. The coagulants are classified by chemical composition into inorganic (aluminum sulfate), synthetic polymeric (polyaluminum chloride), and natural coagulants (plant leaves and seeds) [5].

Treatment by coagulation–flocculation involves a physico–chemical process that uses a coagulant to neutralize the negative charges in contaminated water, thereby reducing electrostatic repulsion within the electric double layer; this is known as the destabilization process [6]. The destabilization begins with an increase in ionic strength, which helps to promote double-layer compression, and/or with the adsorption of anions through the neutralization process of the particle's surface charge. During low mixing, destabilized particles aggregate to form flocs, then undergo free precipitation, and are finally separated from the contaminated water. Choong Lek et al. [7] reported the use of chickpea (*Cicer arietinum*) for the treatment of palm oil mill effluent and obtained a maximum turbidity removal of 86%. Some other researchers also reported the use of chitosan [8] and *Moringa oleifera* [9] for the treatment of palm oil mill effluent, achieving 95% and 99.5% turbidity reductions, respectively. Gaayda et al. [10] showed that the use of grape seed and *Austrocylindropuntia* mucilage can remove Congo red and turbidity from synthetic wastewater. According to response surface methodology, Box–Behnken design (RSM-BBD) results reached highest removal efficiency with 99.36 and 95.74% under optimum conditions of 0.45 mg/L of grape seed powder coagulant, 6 mL/ L of *Austrocylindropuntia* mucilage flocculant, at pH of 10, initial Congo red concentration of 5 mg/L, initial turbidity of 250 NTU, and after settling time of 120 min [11]. Several findings have examined the role of natural coagulants/flocculants for the treatment of wastewater, such as Aleppo pine seeds for the removal of Congo red dye [12], grape seed for the removal of chromium (VI) ions [10], and a mix of *Acanthus sennii* C., *Moringa stenopetala* B., and *Aloe vera* L. to remove chemical oxygen demand (COD) from wet coffee processing wastewater [13].

While many studies have investigated coagulation–flocculation for the removal of colloidal particles, dyes, and turbidity in various industrial wastewaters, there is a notable scarcity of research focusing on the simultaneous high-efficiency recovery of macronutrients (PO_4 and NO_3) specifically from aquaculture effluent [14,15]. Furthermore, most existing literature focuses on purification without exploring the agricultural valorization of the resulting sludge within a circular bioeconomy framework. This study addresses this research gap by evaluating the dual-purpose efficacy of *A. graecorum* leaves and apricot seeds, locally available, low-cost biomass in Iraq, as both treatment agents and nutrient-capture vehicles. To address this gap, research was conducted using coagulants extracted from the leaves *of A. graecorum* and from apricot seeds. The innovation of this study is to adopt a sustainable environmental approach to wastewater treatment and recovery in the fish farm industry, providing eco-friendly clarification. The use of natural coagulants was explored and optimized using a central composite design (CCD) to achieve optimal treatment and recovery with selected leaves of *A. graecorum* and apricot seeds. This study aimed to investigate the ability of locally user-friendly leaves of A. graecorum and apricot seeds as a low-cost coagulant for the treatment and recovery of nutrients from fish farm wastewater, in order to protect the ecosystem.

2. Materials and Methods

2.1. Preparation of Natural Coagulants and Wastewater

The leaves of the *A. graecorum* and apricot seeds were collected in the region of Basrah, Iraq. The selection of *A. graecorum* leaves and apricot seeds as green coagulants was based on their high abundance in Iraq and their distinct biochemical profiles, as reported in the previous literature. *A. graecorum leaves* were previously reported to contain tannins and polyphenols [16,17]. In contrast, apricot seeds were characterized by high protein, peptides, and pectin [18,19] which may function as a coagulant.

Wastewater used in the coagulation–flocculation tests was collected from a semi-intensive polyculture local fish farm in Basrah, Iraq, via grab sampling (n = 3) conducted during the fish grow-out period. This period was selected to capture the peak concentrations of organic and nutrient pollutants. This fish farm utilizes large earthen open basins for the simultaneous rearing of *Cyprinus carpio* (Common Carp) and *Mesopotamichthys sharpeyi* (Bunni). These basins operate on a semi-static water exchange basis. The aim of using natural coagulants to treat fish farm effluent was to recover nutrients (N and P) in settled sludges as fertilizer. Table 1 shows the initial characteristics of wastewater used in this optimization study.

Table 1. Fish farm effluent characteristics (values are presented as mean ± SD with n = 3).

Parameter	Turbidity	TSS	pH	NO_3	PO_4
Value	521 ± 26 NTU	1442 ± 70 mg/L	7.6 ± 0.2	0.19 ± 0.01 mg/L	43 ± 2.1 mg/L

The leaves of the *A. graecorum* and apricot seeds were previously dried in the oven (BINDER, Tuttlingen, Germany) at 80 °C for 48 h. The dried leaves and seeds were then crushed with a grinder (Mxbaoheng, Chicago, IL, USA), sieved with a 60 μm porous filter (Porex, Ningbo, China), and kept in a Duran bottle [20]. The preparation of the natural coagulant was performed by extraction with distilled water at a weight of green powder to volume of water (1000, 4000, and 7000 mg/L). After 1 h of stirring (Vitlab, Grossostheim, Germany), the solution was filtered through a cloth and then used for the coagulation experiment.

For initial fish farm effluent characterization, turbidity was measured with a turbidity meter (UPM GmbH, Augsburg, Germany), total suspended solids (TSS) were analyzed gravimetrically, and pH was measured with a portable pH meter (Mettler Toledo, Albstadt, Germany). As for the main parameter, NO_3 and PO_4 concentrations were analyzed using HACH DR6000 (HACH, Colorado, USA) following the HACH kit protocol.

2.2. Coagulation–Flocculation Process for Pollutant Removal

The test of the green coagulant was conducted using jar tests to investigate the removal efficiency of nutrients from fish farm effluent. For each experiment, 500 mL of the fish farm effluent was put in 1000 mL beakers. The coagulant concentration, wastewater pH, and settling time were varied according to the experimental design, as shown in Table 2, for the two tested green coagulants. At the end of the coagulation–flocculation experiments, the final NO_3 and PO_4 were measured using a HACH DR6000 instrument.

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Table 2. Range and levels of experimental input factors.

Factor	Name	Units	Minimum	Maximum	Low (−1) *	Central (0) *	High (+1) *
A	Concentration	mg/L	1000	7000	1000	4000	7000
B	pH		5	9	5	7	9
C	Settling Time	min.	15	35	15	30	35

* Refers to the standardized numerical values assigned to the independent variables in the central composite design (CCD). Low (−1) represents the minimum, central (0) represents the midpoint, and high (+1) represents the maximum experimental level for each factor.

2.3. Optimization Conditions

The CCD was used to investigate the effects of three independent variables: coagulant concentration (1000–7000 mg/L) of *A. graecorum* leaves and apricot seeds, wastewater treatment pH (5–9), and settling time (15–35 min). Table 2 shows the ranges and levels of the variables as outputs from Design-Expert software (version 13, Stat-Ease, Inc., Minneapolis, MN, USA). The 3-level 3-factor CCD is applied to analyze and validate coagulation–flocculation parameters affecting the removal of nutrients by *A. graecorum* leaves and apricot seeds.

The CCD optimization yields a total of 15 experiments for factors A, B, and C at the levels, minimum −1, central 0, and maximum +1, as shown in Table 3 for both tested green coagulants. In this study, factors A (coagulant concentration), B (pH), and C (settling time) with two responses (PO_4 and NO_3 removals) and a face-centered CCD were selected for experimentation to obtain a natural number of factors [13]. Analysis of Variance (ANOVA) was performed to validate the accuracy of RSM Models. The significance of the quadratic models was confirmed by high F-values and low p-values ($p < 0.05$) [21], indicating that the selected parameters have a statistically significant effect on nutrient recovery. Furthermore, the Lack of Fit for each model was found to be non-significant relative to the pure error, which implies that the models are highly reliable for predicting PO_4 and NO_3 removal within the space of the experimental design [22].

Table 3. Experimental runs for the three-factor by central composite design application.

Run	A: Concentration	B: pH	C: Settling Time
Unit	mg/L		minutes
1	7000	5	35
2	1000	5	35
3	7000	5	15
4	4000	5	25
5	1000	5	15
6	1000	7	25
7	4000	7	25
8	4000	7	35
9	4000	7	15
10	7000	7	25
11	1000	9	35
12	7000	9	35
13	7000	9	15
14	4000	9	25
15	1000	9	15

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2.4. Fertilizer Preparation

The residual sludge was used to cultivate *Raphanus sativus* L. seeds for a week, the period during which plant seed growth was observed. The seed growth observation aimed to determine the germination index (GI) of recovered sludge after treatment with green coagulant for fish farm effluent. The choice of *R. sativus* L. for the GI test was based on its established role as a model bioindicator in environmental research, as it is susceptible to external chemical environments and on its recommendation by international guidelines (the US EPA and OECD) for terrestrial plant growth and toxicity tests [23].

The fertilizer was prepared at a 1:10 (*g*/*v*) ratio, with 10 g of recovered sludge and 100 mL of distilled water. The mixture was left for 3 h, then filtered through filter paper No. 1 (Whatman, Kent, UK). Six different fertilizer concentrations were prepared: 0, 2, 4, 6, 8, and 10%. The 0% treatment serves as a control, using sterile distilled water only to compare the effect of fertilizer on the growth of *R. sativus* L. seeds.

Sterile 15 cm Petri dishes were used, and sterile filter paper was placed at the bottom of each dish. Then, 5 mL of each fertilizer solution was added, with 2 replicates per concentration, and 8 *Raphanus sativus* L. seeds were placed in each dish and incubated in the incubator at 25 °C for 5 days.

2.5. Germination Index (GI)

Germination experiments using fish farm sludge were conducted to examine nutrient recovery as fertilizer through seed germination and growth [24]. A germination rate of 90% or higher was adopted for *A. graecorum* and apricot seeds, as it indicates better seed root growth. The recovery of sludge as fertilizer will accelerate seed growth and elongate roots [25]. The proportion of the number of growing seeds and the percentage of increasing root lengths were calculated using Equations (1) and (2), while the germination index was measured by following Equation (3) [26].

$$\% \text{ Seed germination} = \frac{Number\ of\ growing\ seeds\ in\ dish}{Number\ of\ seeds\ grown\ in\ control} \times 100 \quad (1)$$

$$\% \text{ growing root lengths} = \frac{Average\ root\ length\ in\ the\ dish}{average\ root\ length\ in\ the\ control} \times 100 \quad (2)$$

$$\text{GI} = \frac{\%\text{Seed germination} \times \%\text{ Growing root lengths}}{100} \times 100 \quad (3)$$

3. Results and Discussions

3.1. Coagulation–Flocculation Efficiency of A. graecorum Leaves and Apricot Seeds

The jar test results for *A. graecorum* leaves and apricot seed as green coagulants are listed in Table 4. For *A. graecorum*, the maximum removal efficiencies were 98.05% and 94.74%, while apricot seed coagulants showed 99.9% and 92.63% for PO_4 and NO_3, respectively. The differences in the ability of natural coagulants are due to the charge density and molecular weight of the green coagulant [4]. Optimum results were achieved with a coagulant concentration of 1000 m/L for both green coagulants and settling times of 35 and 15 min for *A. graecorum* and apricot seed, respectively. Optimum pollutant removal was observed in regions of the plot with low coagulant concentration, demonstrating that these levels were adequate for treating the fish farm wastewater. The observed decrease in efficiency at higher concentrations (4000–7000 mg/L) may be attributed to the restabilization phenomenon [27], in which an excess of coagulant leads to charge reversal on particle surfaces, preventing effective flocculation. While the 1000 mg/L was chosen based on previous literature for consistent floc formation, the results suggest that future studies could investigate even lower dosages to pinpoint the absolute minimum threshold for nutrient recovery.

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Table 4. Actual observation of responses after the jar test.

Plant	Response	Units	No. of observations	Minimum	Maximum	Mean ± SD *
A. graecorum	PO_4 removal	%	15	13.72	98.05	72.84 ± 24.51
A. graecorum	NO_3 removal	%	15	57.89	94.74	78.89 ± 9.78
Apricot	PO_4 removal	%	15	35	99.9	79.73 ± 20.03
Apricot	NO_3 removal	%	15	23	92.63	69.02 ± 16.68

* SD: standard deviation.

3.2. Statistical Analysis and Regression Model

The generated equations are listed in Table 5. Quadratic polynomial and Two-Factor Interaction (2FI) models were used to determine the relationships between pollutant removal, PO_4, and NO_3, with coagulation concentration (A), pH (B), and settling time (C). Based on RSM, a CCD statistical analysis was used to select the most appropriate models from various adjustments to the experimental data, including regression significance, coefficient of determination (*R*-squared, R^2), and adjusted R-squared (R^2_{adj}). The regression models were significant at the 95% confidence level (Table 6). The response Models' R^2 were >80%, and the differences with R^2_{adj} were <0.2, which can be considered well-adjusted [13].

Table 5. Equations based on CCD for the three factors studied.

Response	*A. graecorum*	Apricot Seed
PO_4 removal	Quadratic ($PO_4 = 81.53 - 0.394A + 16.92B + 10.62C + 6.17AB - 0.1163AC - 17.3BC + 6.02A^2 - 26.08B^2 + 6.03C^2$)	Quadratic ($PO_4 = 70.64 - 10.47A + 1.67B - 5.4C + 2.59AB - 6.4AC + 1.39BC + 25.38A^2 + 14.62B^2 - 26.37C^2$)
NO_3 removal	2FI ($NO_3 = 78.89 + 3.61A + 3.45B + 0.501C + 3.39AB - 6.22AC + 8.46BC$)	2FI ($NO_3 = 68.02 - 7.15A + 9.81B + 1.53C + 7.98AB + 14.03AC - 2.45BC$)

Table 6. ANOVA data of pollutants removal efficiency (PO_4 and NO_3) using *A. graecorum* leaves and apricot seed.

Response	*A. graecorum*	Apricot Seed
PO_4 removal	Model: *p*-value = 0.0074 R^2 = 0.9543 Adjusted R^2 = 0.8721	Model: *p*-value = 0.0278 R^2 = 0.9196 Adjusted R^2 = 0.7749
NO_3 removal	Model: *p*-value = 0.007 R^2 = 0.9151 Adjusted R^2 = 0.8513	Model: *p*-value = 0.0009 R^2 = 0.9096 Adjusted R^2 = 0.8417

Table 6 shows that all data were well adjusted in the present work, and the model explains up to 95.43% of the variation in the response when using *A. graecorum* as a coagulant and up to 91.96% when using apricot seed.

3.3. The 3D Plots with the Effects of Variables on the Response

Three-dimensional (3D) Response surfaces graphically represent the relationships between the response variable and the experimental levels of three independent variables: coagulation concentration (A), pH (B), and settling time (C). The 3D graphs (Figures 1 and 2) for *A. graecorum* and apricot seed showed the correlation between the independent factor and response. These plots are created by fixing one variable at its optimal level while adapting the others within defined limits. Figures 1 and 2 for *A. graecorum* and apricot seed show that 1000 mg/L of coagulant concentration and pH 5 and 9 are optimal for nutrient removal, respectively.

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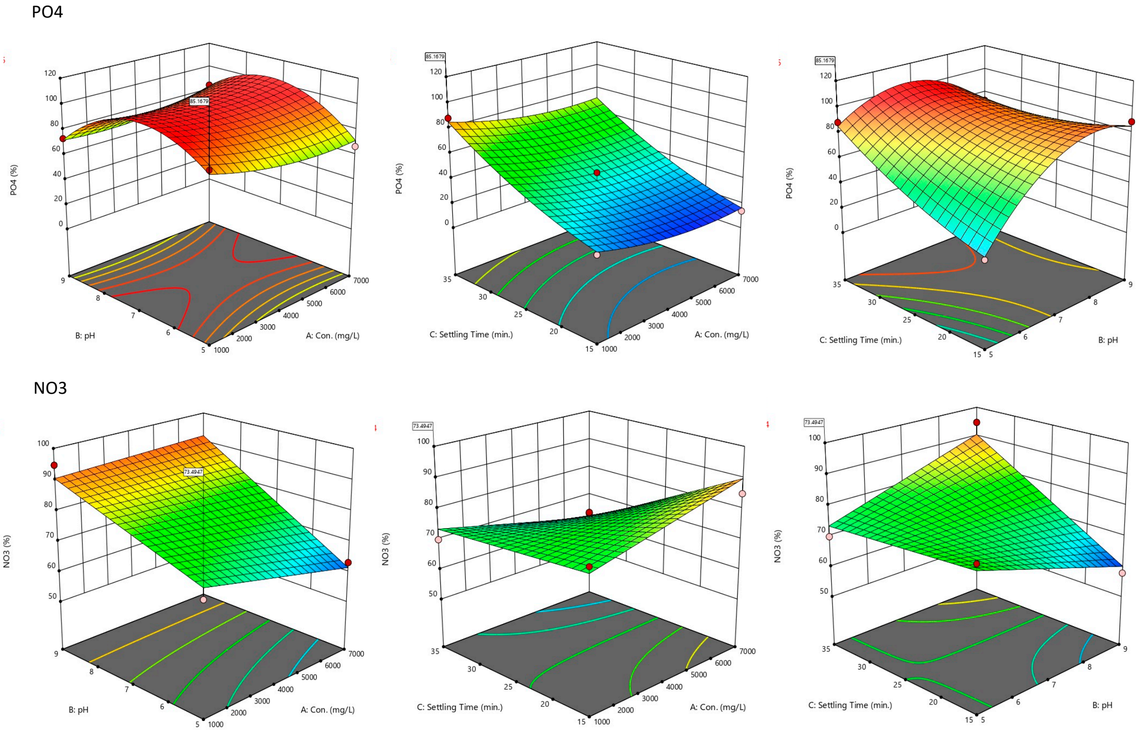

Figure 1. Response of nutrient removal efficiency using *A. graecorum* for the factors studied.

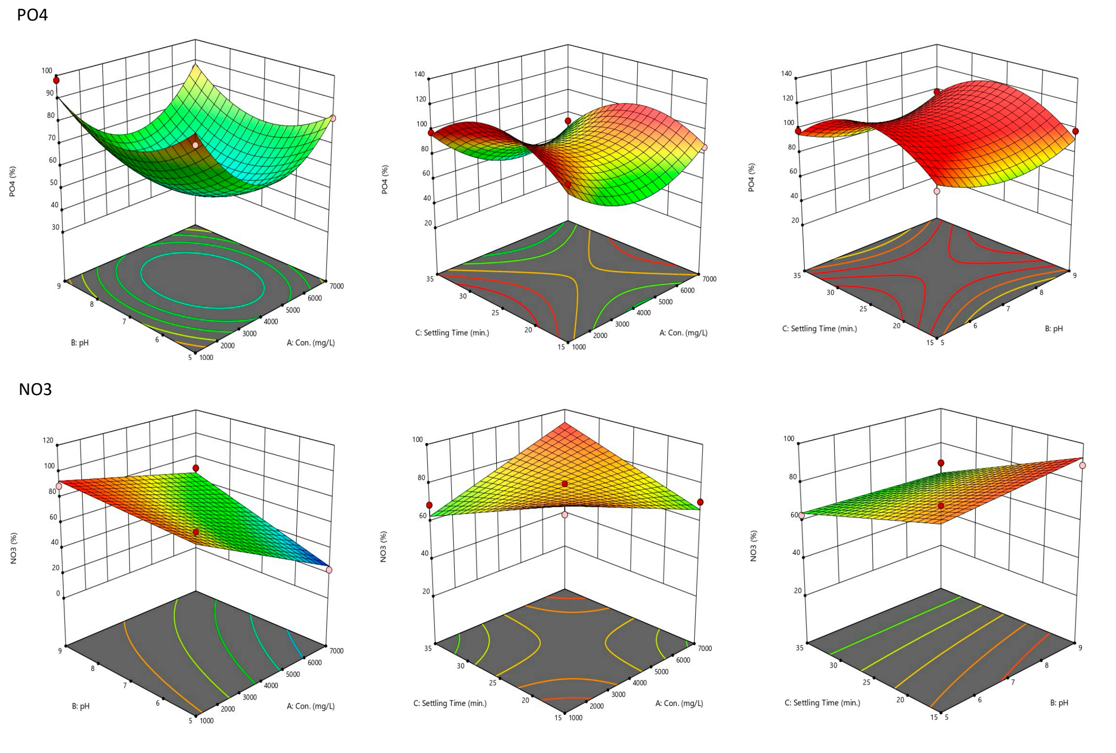

Figure 2. Response of nutrient removal efficiency using apricot seed for the factors studied.

3.4. Optimum Conditions Using CCD

Optimum conditions are summarized in Table 7. The CCD predicted the highest removal efficiency for *A. graecorum* are 86.27% (PO_4) and 73.49% (NO_3) and for apricot seed are 92.60% (PO_4) and 92.63% (NO_3).

Table 7. The predicted highest removal efficiency for *A. graecorum* and the apricot seed.

	Concentration (mg/L)	pH	Settling Time (min.)	PO_4 Removal	NO_3 Removal	Desirability
A. graecorum	1000	5	35	86.27	73.49	1
Apricot seed	1000	9	15	92.6	92.63	0.99

The CCD results showed an optimized coagulant quantity of 1000 mg/L, promoting high PO_4 and NO_3 removal at acidic pH (5) and basic pH (9) in media for *A. graecorum leaves* and apricot seed, respectively. The final process of coagulation–flocculation is the settling of produced flocs, which achieves high removal efficiency with a faster floc settling rate [12]. The faster flocs settling time was 35 min and 15 min for *A. graecorum* leaves and apricot seed, respectively (Table 7). In this study, the optimum condition was identified, with apricot seed providing the fastest settling time. The high removal efficiencies observed for PO_4 and NO_3 might be attributed to the interaction between the multi-functional groups of the plant extracts and the dissolved ions. In the case of *A. graecorum*, the presence of polyphenols likely facilitates charge neutralization [16,17], while the protein-rich matrix of apricot seeds promotes interparticle bridging [17,18]. These mechanisms allow for the transition from stable colloidal suspensions to settleable flocs.

While the nutrient removal efficiencies observed in this study are exceptionally high, they must be interpreted in the context of the specific effluent matrix and dosage requirements. Compared with conventional chemical coagulants such as alum or ferric chloride, which typically achieve similar removal rates at lower dosages (often 50–500 mg/L) [16,17], the green coagulants in this study required a minimum dosage of 1000 mg/L. However, the use of higher dosages is balanced by several practical advantages. Unlike chemical coagulants, which can significantly alter the pH of the treated water and introduce residual metallic ions, *A. graecorum* and apricot seeds are biodegradable and non-toxic. Furthermore, the higher volumes of sludge produced at these dosage levels are beneficial to this study's objective. From a practical perspective, applying 1000 mg/L of plant-based coagulant is feasible in the Basrah region due to the abundance and low cost of these raw materials. While industrial-scale dosing equipment would need to handle larger volumes of solids compared to alum-based systems, the elimination of chemical procurement costs and the added value of the bio-fertilizer byproduct provide a strong economic incentive for local fish farmers to adopt this green technology.

3.5. Potential of Sludge Recovery

According to Table 8, the results for *A. graecorum* showed that the highest GI was 420% at a concentration of 10%, while concentrations of 2, 4, 6, and 8% reached 291, 148, 247, and 358, respectively. Hailu et al. [28] found that fishpond effluent was as effective as chemical fertilizer for tomato cultivation, offering an affordable, environmentally friendly option. Eid et al. [29] found that using fish farm effluent for irrigation could reduce mineral fertilizer use by 40% while eliminating the need for typical irrigation water in potato production.

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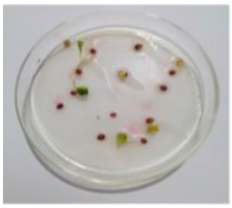
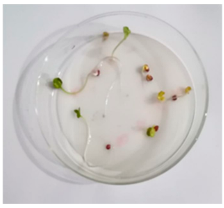
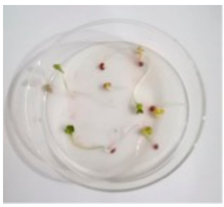
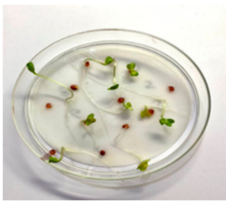
Table 8. Germination Index of fertilizers produced by *A. graecorum* and apricot seed.

Concentrations	Seed Growth%		Root Growth%		GI%	
	A. graecorum	Apricot seed	*A. graecorum*	Apricot seed	*A. graecorum*	Apricot seed
Control	100	100	100	100	100	100
2%	100	114	291	184	291	209
4%	85	114	175	196	148	223
6%	100	71	247	114	247	81
8%	100	114	358	162	358	184
10%	114	100	369	194	420	194

For apricot seed (Table 8), the results showed that GI reached 223% at a concentration of 4% (Table 9), while concentrations of 2, 6, 8, and 10% reached 209, 81, 184, and 194, respectively. The GI values observed, reaching up to 420%, indicate a profound biostimulatory effect on *R. sativus* L. seeds. This exceptionally high value is attributed to the synergistic presence of concentrated bioavailable nitrogen (from NO_3) and phosphorus (from PO_4) captured within the organic matrix of the plant-based coagulants. Nesan et al. [30] evaluated membrane filtration and phytoremediation with *Spirodela polyrhiza* to treat fish farm wastewater, demonstrating increased total nutrient removal and improved water quality.

While the GI results confirm the immediate non-toxicity and nutrient-rich nature of the produced sludge, its broader agricultural application must be interpreted with caution. The high nutrient density that benefits initial germination could, if applied in excess, lead to soil nutrient imbalances in sensitive species [31]. Furthermore, although green coagulants are biodegradable, the long-term mineralization rates of this sludge across different soil types (e.g., the saline soils typical of the Basrah region) are yet to be fully characterized. Future research is required to evaluate the slow-release potential of these nutrients and to ensure that the accumulation of organic matter does not negatively impact soil porosity or microbial community structures over multiple growing seasons [32].

Table 9. *A. graecorum* and apricot seed growth testing with sludge recovery as fertilizer.

Concentrations	*A. graecorum*	Apricot Seed
Control		
2%		

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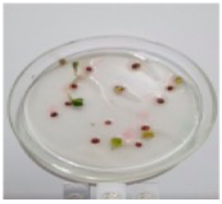
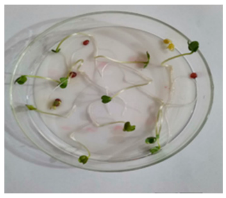
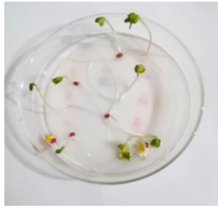
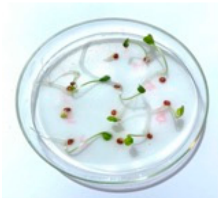
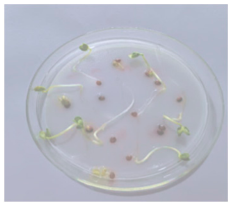
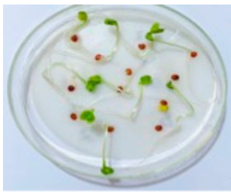
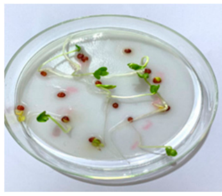
4%		
6%		
8%		
10%		

4. Conclusions

This study demonstrates the high potential of *Alhagi graecorum* leaves and apricot seeds as sustainable, low-cost green coagulants for treating fish farm effluent. The research successfully identified distinct operational envelopes for each material, revealing that *A. graecorum* excels in acidic environments, while apricot seed performs better in alkaline conditions. Rather than treating wastewater alone, the use of green coagulants facilitates the recovery of essential nutrients into a stable sludge. The high increase in the germination index of *Raphanus sativus* L. confirms that this sludge can be safely and effectively repurposed as a bio-fertilizer, offering a zero-waste solution for aquaculture management. Future research should focus on pilot-scale applications to assess the economic feasibility of large-scale nutrient recovery, while also evaluating the potential of using low concentrations (<1000 mg/L) and further functional compound characterization. Additionally, long-term studies are needed to assess the cumulative effects of recovered sludge on soil health and its performance across a broader range of food crops.

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