


## Photocatalysis and flocculation processes for recycling aquaculture effluent into nutrient-rich irrigation water

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### ABSTRACT

This research aimed to create a novel technique for recovering fertilizers from aquaculture effluent to overcome potential non-renewable fertilizers shortages. There are two steps: a photocatalyst technique for nutrient mobilization, succeeded by solids precipitating with two natural and one synthetic flocculants. The photocatalytic degradation of organonitrogen compounds in batch experiments started under the irradiation of sunlight. Following that, photocatalytic breakdown of organonitrogen compounds produces inorganic nitrogen constituents like  $\text{NH}_4^+$ ,  $\text{NO}_2^-$ , and  $\text{NO}_3^-$ , which could be used as manure. It was found that, after 12 h of circulation, the concentration of inorganic nitrogen became  $\text{NH}_4^+ = 17.2 \text{ g/L}$ ,  $\text{NO}_2^- = 18.1 \text{ mg/L}$ , and  $\text{NO}_3^- = 15.9 \text{ mg/L}$ . The jar test was adopted to assess the capacity of two natural compounds (tamarind kernel polysaccharide (TKP) and tannin-based substance (TBS)) and synthetic water-soluble polymer cationic polyacrylamide (SWP) to reduce turbidity, total suspended solids (TSS), chemical oxygen demand (COD) and colour. The findings revealed that with a dose of 20 mg/L of TBS, 20 mg/L of TKP, and 50 mg/L of SWP, the maximum turbidity reductions were 95, 93, and 94%, respectively. The TBS was slightly better than TKP and much better than SWP in terms of coagulation activities with TSS, COD and colour maximum removal efficiencies.

**Key words:** aquaculture, coagulation, fertilizers, flocculation, photocatalytic

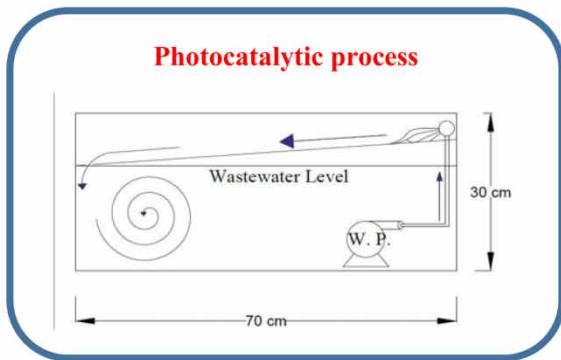
### HIGHLIGHTS

- Remediation aquaculture effluent to overcome potential non-renewable fertilizers shortages.
- In this work, the photocatalytic degradation of organonitrogen was studied.
- Two natural flocculants were used in the treatment process.

GRAPHICAL ABSTRACT



**Aquaculture  
wastewater**



**Solution rich by  
nutrient**

**Solid precipitation process**



## INTRODUCTION

Aquaculture is a rapidly expanding industry at this time, and the aquacultural sector produces 47% (about 51 million tons) of worldwide humanoid fish consumption, according to the [FAO \(2012\)](#). Aquaculture output could double over the next three decades to keep pace with population expansion and rising fish consumption. Aquaculture produced 74 million tons of food during 2015 ([Turcios & Papenbrock 2014](#)). The global fish business is growing, and the latest World Bank Study 83177-GLB ([World Bank 2013](#)) predicts that aquaculture will contribute 50% of global fish production by 2030. Both outdoor or indoor recirculating aquaculture methods are fully developed. Waste treatment inside the recirculation system and within the discharged effluents of these systems is crucial because of the concentrated practice of fish farming in most of these systems ([Van Rijn 2013](#)). The collected sludge contains nutrients that may cause a pollution problem if inadequately disposed of in water bodies ([Abdulredha et al. 2021](#); [Isra'a et al. 2021](#)). However, the aquaculture system sludge can also be utilized for fertilizing purposes after appropriate treatment. Thus, transforming aquaculture waste into beneficial fertilizers solves the issue of disposal, provides financial gains and solves the upcoming non-renewable fertilizer shortages ([Nile & Faris 2018](#); [Ezziddine et al. 2020](#)).

In Norway, vast and increasing quantities of aquaculture wastewater holding organonitrogen complexes like amino acids, urea, and proteins are generated from systems with direct flow-through of water and systems with recirculated water. The quantities of waste produced are proportionate to the amount of fish produced. In systems with water recirculation, the dissolution of particles into fine particles, colloids and dissolved compounds may impair water quality and pose a threat to fish health if not removed. As a result, concentrated fish culture must be developed along with effective wastewater treatment technologies ([Turcios & Papenbrock 2014](#); [Al Juboury & Abdul-Hameed 2019](#)).

Adsorption, chemical oxidation and reduction, and biological treatment are only a few of the physical, chemical, and biological methods employed in freshwater aquaculture systems today ([Crab et al. 2007](#); [Manoj & Vasudevan 2012](#); [Hashim et al. 2018](#); [Zwain et al. 2020](#)). Energy and the addition of other chemical agents are usually required for these operations, resulting in significant expenditures and even indirect pollution ([Aayef et al. 2021](#); [Abdulredha et al. 2021](#)). Photocatalysis appears to be the most promising method for cleaning wastewater because of numerous benefits, including the ability to work in moderate circumstances by directly employing sunlight, which may save money and energy. It may also break down organic molecules completely into CO<sub>2</sub> and H<sub>2</sub>O without the need for chemical agents, resulting in no environmental contamination. Furthermore, due to its exceptionally high oxidizing capacity, it breaks down nearly all organic molecules, indicating that it may be used to treat wastewater carrying a variety of organic contaminants. High quantities of organonitrogen compounds from aquatic animal food or faeces were found in aquaculture effluent, resulting in pollution, algae blooms, freshwater degradation, and even disease outbreaks ([Lin et al. 2002](#); [Vymazal 2010](#)). Although photocatalysis can oxidize such organonitrogen components, inorganic nitrogen compounds like NH<sub>4</sub><sup>+</sup>, NO<sub>2</sub>, and NO<sub>3</sub> could be used as fertilizers. According to [Saenz \(1987\)](#), nutrients in sewage might be fertilizers capable of increasing agricultural production. [Ujj-Meszaros \(1986\)](#) observed that irrigation with nitrogen-rich industrial sewage enhanced fodder beet production (11 Mg starch per ha) and crude protein digestion (5.9 Mg per ha). An alternative way to utilize the nutrients in aquacultural waste is to use the sludge from solids removal systems, more directly in soilless growth systems of vegetables (hydroponic and aquaponic systems). However, nutrients associated with solids are not readily available for plant growth. Mobilizing nutrients to soluble forms enables the use of nutrient-rich water in soilless growth systems. Techniques to remove solids from the treated aquacultural sludge include flocculation and sedimentation ([Ezziddine et al. 2020](#)).

Cationic polymers can be used as a replacement for the dual roles of flocculation and coagulation. The positive charges neutralize the negative charges on particle surfaces and bridge the destabilized particles into aggregates ([Yang et al. 2016](#)). In wastewater treatment, Cationic polymers have mainly been used as a flocculant to remove various forms of soluble and suspended compounds, like toxic metals, humic acid, dyes, and algae ([Lee et al. 2014](#)). Improving solids settling characteristics have been demonstrated in various effluents such as sewage, food processing wastewater, aquacultural wastewater, manure wastewater and dye wastewater ([No & Meyers 2000](#)). The solids removal efficiency of various synthetic cationic polymers for treating dilute sludge in aquaculture has been studied ([Ebeling et al. 2005](#)). [Bian et al. \(2015\)](#) studied the photocatalytic degradation of aquaculture wastewater using ultraviolet (UV) lamps as sunlight, resulting in the inorganic nitrogen compounds subsequently used as nourishments to reduce pollution. Their approach consisted of two steps: nutrient mobilization via aerobic digestion and solids precipitation using chitosan as a flocculant for recovering fertilizers from aquacultural effluent for application in soilless growing systems ([Bian et al. 2015](#); [Faraj et al. 2020](#)).

The purpose of the current research was to examine the effects of treating aquacultural wastewater with natural sunlight on the formation of inorganic nitrogen compounds as well as the removal of suspended solids using two natural flocculants, a tannin-based substance (a substance extracted from the bark of the tree *Acacia mearnsii* (TBS)) and tamarind kernel polysaccharide (TKP). In addition, when it comes to processing aquaculture waste for use as a fertilizer in soilless growth systems, the performance of the two natural flocculants was also compared to the preference of the synthetic water-soluble polymer cationic polyacrylamide (SWP). This novel approach for recovering nutrients from aquaculture wastewater to address possible non-renewable fertilizer shortages was investigated because no other researchers had looked into it. This approach was accomplished by comparing two natural cationic and synthetic water-soluble polymer cationic polyacrylamide flocculants for treating aquaculture effluent following photocatalysis.

## EXPERIMENTAL WORK

### Chemicals and materials

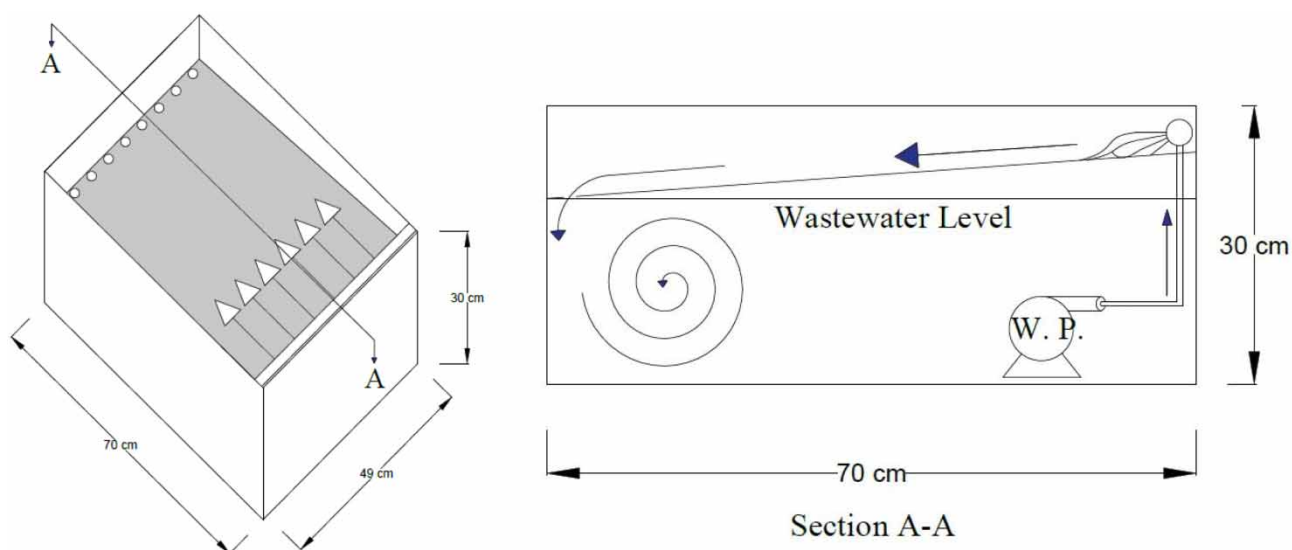
The natural flocculants were cationic TBS derived from the bark of the *Acacia mearnsii* tree, which is found in Tanac SA, Brazil, and TKP, which was provided by the Hindustan Gum & Chemicals Ltd, Bhiwani, Haryana, India. The enhanced TKP has been proposed as a novel matrix for regulating aspirin release. The synthetic flocculant was cationic polyacrylamide, a water-soluble polymer (equal to superfloc C495 BLUWAT blufloc CPAM, China). Tanfloc flocculent is effective at pH levels ranging from 4.5 to 8. In addition, it does not substantially alter the medium's pH. The flocculent, which was offered as a dried powder, was hydrated until it was completely dissolved. By adding 0.1 M HCl or 0.1 M NaOH to the resultant solution, the pH could be changed as needed (Jawad *et al.* 2021).

### Aquaculture water

The sludge was collected from the aquaculture ponds in a Freiha area in Karbala city (Iraq) a site of 7,500 to 8,000 m<sup>2</sup>. The utilized aquaculture effluent has a pH of 6.4 and comprises several types of organonitrogen compounds (urea, amino acids, and proteins). The total organic carbon (TOC) of the wastewater is 10.1 mg/L, the ammonium ion (NH<sub>4</sub><sup>+</sup>) concentration is 14.1 g/L, the nitrogen dioxide (NO<sub>2</sub><sup>-</sup>) concentration is 16.5 mg/L, and the nitrate (NO<sub>3</sub><sup>-</sup>) concentration is 8.8 mg/L. Thus, organic matter is present in the wastewater, with concentrations of volatile suspended solids (VSS) and total suspended solids (TSS) of 7.8 g/L and 7.9 g/L, respectively. The concentration of chemical oxygen demand (COD) was 5,900 mg/L.

### Photocatalysis & solid removal process

The photocatalysis was carried out using batch flow experiments and a transparent glass tank of 70 × 50 × 30 cm) and backwashed with water from a local sand filter (Figure 1). This tank was equipped with a submerged pump for water transport from the tank to



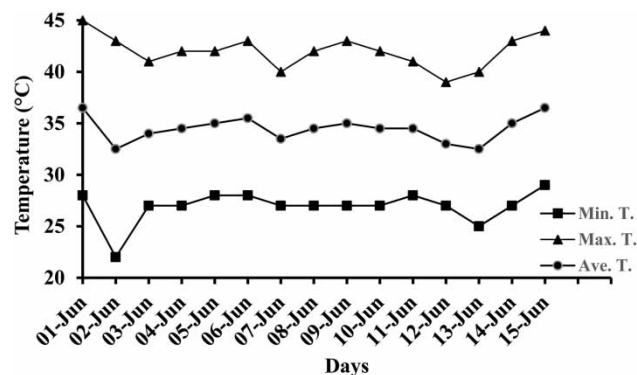
**Figure 1** | Schematic diagram of the reactor.

the top of the slant glass cover that covered the upper surface of the tank. This glass cover was inclined and this enabled the water to slide from the upper end to the lower end of the lid to make the wastewater be exposed to sunlight and back to the tank. All experiments were conducted utilizing natural solar energy from 1 June to 15 June, 2020, with an average daily temperature ranging from 33 to 37 °C with different values of flow rate (0.5, 1.5, 2.5, 3.5, 4.5 and 6 L/h). During the daytime (6 a.m. to 6 p.m.) and under direct sunlight, photocatalytic degradation was carried out with an intensity level of about 56 to 69 mW/cm<sup>2</sup> (Figure 2). After each run, the reactor was thoroughly cleaned with clean water, and 90 L from the same aquaculture effluent was introduced for the next cycle. Water samples were carefully withdrawn from the transparent state to examine various components such as nitrite, nitrate, and ammonium.

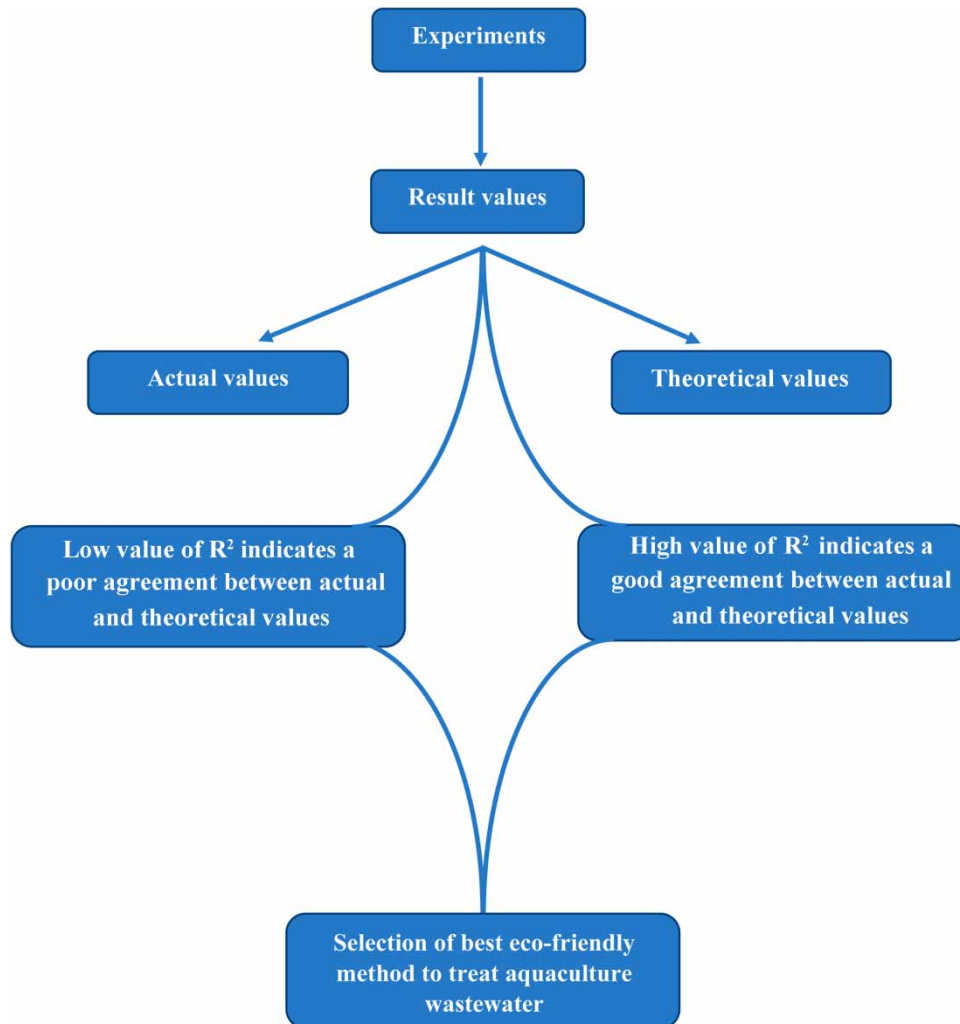
Jar tests described by Gutiérrez *et al.* (2016) with flocculation and sedimentation were performed to remove solid particles from the wastewater. Before the jar tests, stock solutions of 1,000 mg/L were prepared. Drops of a 30% HCl buffer was added under constant stirring until the cationic, and the flocculent was dissolved, yielding different doses of the cationic compounds ranging from 5 to 60 mg/L (Gutiérrez Martínez 2016). The experiments were conducted in batches utilizing jar-test equipment with six 500 mL jars and varied coagulant doses (Alatabi *et al.* 2017). Drops of diluted HCl or NaOH solution were added to modify the pH level of the solution between 3.5 to 8.5. After 120 seconds of rapid mixing (250 rpm), the treated water was slowly stirred (60 rpm) for 20 minutes to flocculate, then allowed to settle for another 20 minutes. After that, samples were carefully taken from the clear section for turbidity and TSS analyses in different connection times of 0, 2, 4, 6, 8, 10 and 12 h (Ortiz *et al.* 2021). Water was only supplied to the system to compensate for transpiration and evaporation losses, and water samples were obtained for analysis.

### Study & analysis process

A QuAAtro continuous segmented stream autoanalyzer was used to assess the extraction competence of organic and inorganic nitrogen compounds such as nitrite, nitrate, and ammonium. Before analysis, samples were centrifuged for 3 minutes at 4,500 rpm and passed through membranes with pore sizes of 0.45 m. A calibrated Hach 2100Q turbidimeter (Loveland, CO, USA) and a calibrated WTW (Bench model, German) were used to test the turbidity and pH of the sewage, respectively. TSS, COD, and colour were determined with a UV/VIS spectrophotometer (s::can Messtechnik GmbH's 'spectro::lyser'). TSS, COD, and colour removal efficiency were determined in independent trials employing TBS, TKP and SWP as coagulants. The highest removal efficiency was used to identify a suitable coagulant. All experiments were done in a sanitary laboratory in the civil engineering department, University of Kerbala. The results provided are averages and standard deviations for all flocculation trials, which were done in triplicate sets. Because the standard deviation lines do not extend far beyond the figure bounds, they are not visible on the plots. The R<sup>2</sup> indicates the degree of agreement between actual and theoretical values (Figures 2 and 3), which might be anywhere from 0 to 1 (Abdulredha *et al.* 2018). The suggested model exhibits a high level of agreement between actual and anticipated values, with R<sup>2</sup> around one (Abdul-Hameed & Al Juboury 2020; Abdulredha *et al.* 2020).



**Figure 2** | Temperature changes between 15 May and 15 June in the study area to assess the performance of the reactor (Iraqi Meteorological Organization & Seismology 2021).



**Figure 3** | Agreement between actual and theoretical values.

### Advantages and disadvantages

The benefit of this study is that it uses natural sunlight to recover the nutrients required for plant development while also repurposing aquaculture effluent into nutrient-rich irrigation water. This research looked at natural materials to see if they met the criteria for sustainability and economics. The lack of treatment of aquaculture effluent for use as drinking water was, however, a drawback of this study. This occurs because it contains components that are required for plant growth but pose a risk to human health.

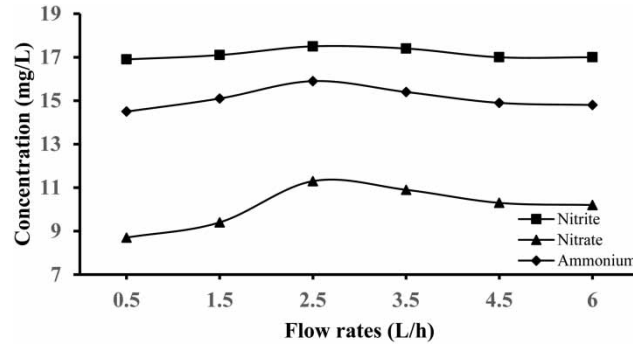
## RESULTS AND DISCUSSION

### Photocatalysis of aquaculture effluent

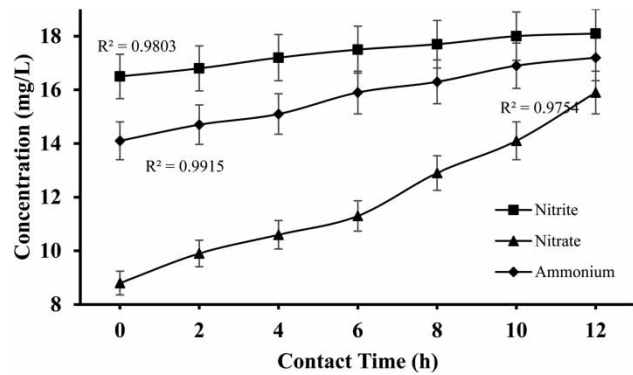
Organonitrogen complexes such as amino acids could be photocatalytically oxidized to produce carbon dioxide, water, and inorganic nitrogen compounds, including nitrite, nitrate, and ammonium. Therefore, the flow rates of wastewater circulation substantially impacted the decomposition of organonitrogen complexes and inorganic nitrogen compounds in farming effluent, as shown in Figure 4. In addition, Figure 4 revealed that 2.5 L/h was the best flow rate to achieve higher removal compared to other flow rates.

The amounts of nitrate and ammonium rose fast as the circulation period increased, corresponding to a reduction in organonitrogen concentration, as seen in Figure 5. The photocatalytic oxidation of organonitrogen complexes such as urea and proteins to inorganic nitrogen compounds is the primary cause.



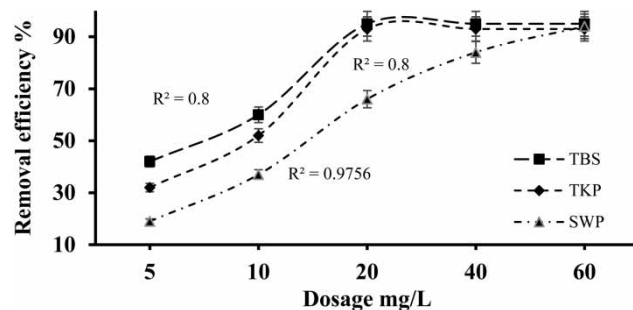


**Figure 4** | The impact of flow rates on nitrite, nitrate, and ammonium concentration levels in aquaculture effluent after 6 hours of photocatalytic circulation of aquaculture effluent.



**Figure 5** | Nitrite, nitrate, and ammonium level effluent after 12 hours of photocatalytic circulation of aquaculture effluent.

It is crucial to take note that the nitrite level dropped fast, as it is easily oxidized to nitrate during photocatalysis. After 12 h of circulation, the inorganic nitrogen concentrations increased to 17.2, 18.1 and 15.9 mg/L for ammonium, nitrite, and nitrate, respectively. The effluent’s interaction between sunlight and organonitrogen complexes and inorganic nitrogen compounds was facilitated by the low flow rate. A high flow rate was also deemed detrimental to photocatalytic efficiency due to the compounds’ limited contact and sunlight. This was further demonstrated by the fact that, as the circulating duration increased, the concentration of organonitrogen pollutants decreased rapidly, and virtually all organonitrogen contaminants were removed after 12 hours of circulation. The frequency of circulating rounds per reaction time (12 h) reduced dramatically, resulting in inefficient oxidation of organonitrogen complexes. The fast photocatalytic oxidation of organonitrogen complexes to create inorganic nitrogen products boosted the ammonium and nitrate concentrations at first. Because the organonitrogen in the system began to decline after 12 hours, ammonium and nitrate production from the photocatalytic breakdown of organonitrogen complexes slowed. These findings matched those of [Bian et al. \(2015\)](#).

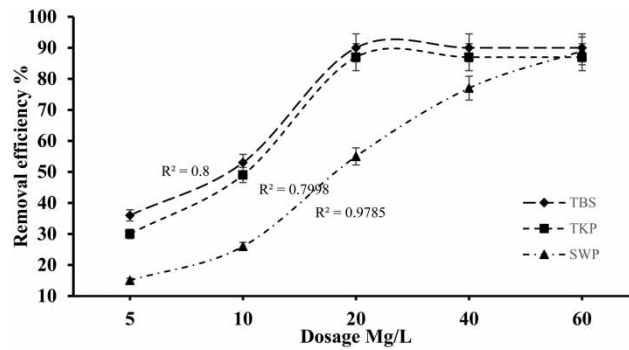


**Figure 6** | Turbidity removal efficiencies employing varied doses of TBS, TKP, and SWP as flocculants.

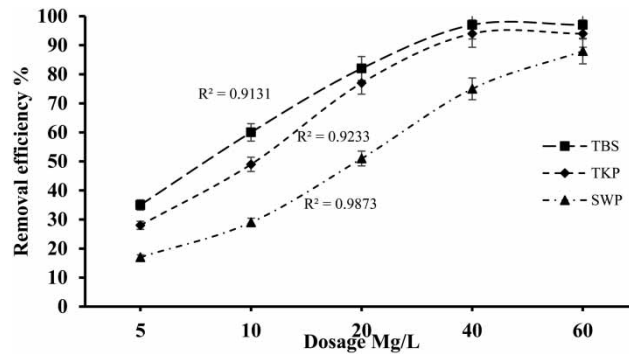
**Removal of solids from aquaculture wastewater**

Figure 6 depicts the effectiveness of coagulants in wastewater treatment. At doses of 20, 20, and 60 mg/L, the maximum coagulation activities of turbidity removal efficiencies of TBS, TKP, and SWP were determined to be 95, 93, and 94%, correspondingly (Figure 5). However, even larger doses of the SWP did not enhance treatment efficiency beyond the findings of 20 mg/L TBS and TKP, as shown in Figure 6. Thus, the treatment efficiency had not improved when the dose of the two natural flocculants was increased beyond a certain point.

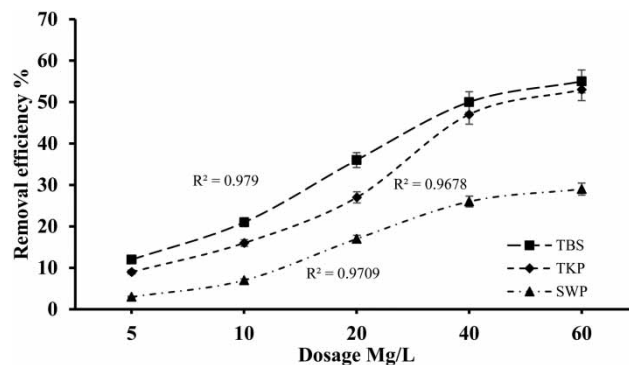
The highest TSS removal efficiencies using 20 g/L of TBS, 20 g/L of TKP and 60 g/L of SWP were found to be 90, 87 and 89%, respectively (Figure 7). While, the highest COD removal efficiencies using 40 g/L of TBS, 40 g/L of TKP and 60 g/L of SWP were found to be 97, 94 and 88%, respectively (Figure 8).



**Figure 7** | TSS removal efficiency employing different doses of TBS, TKP, and SWP as flocculants.



**Figure 8** | COD removal efficiency employing different doses of TBS, TKP, and SWP as flocculants.



**Figure 9** | Colour removal efficiency utilizing varied doses of TBS, TKP, and SWP as flocculants.



Colour removal though using the TBS, TKP and SWP, was also evaluated in this research. Figure 9 shows that the TBS performs similarly to the TKP in colour removal, even at large doses. However, the SWP achieved poor colour removal efficiencies compared with other coagulants (TBS and TK) even at high coagulants dosages. The best colour removal efficiencies achieved by the TBS, TKP and SWP were 55, 53 and 29%, respectively.

Before the jar test, the pH of the aquaculture effluent was 6.4. The pH changes were low after the jar test regardless of the coagulant type employed (TBS, TKP, or SWP), and they ranged between 6.0 to 7.8. Thus, there would be no need to alter the pH in any instance after the coagulation process. These findings align with those of Ezziddine *et al.* (2020) and Guibal (2004).

The results found that TBS was a slightly better coagulant than TKP and much better than SWP in terms of coagulant activity (TSS, turbidity, COD, and colour removal). These results agreed with those of Guibal (2004).

TBS and TKP have a high sensitivity for environmental heavy metals (e.g. lead, mercury, cadmium and chromium). Surprisingly, the chitosan treatment had no effect on the levels of metals required for plant development. Guibal (2004) explained the inadequate removal of certain metals by chitosan flocculation by claiming that the interaction of metal ions with dissolved chitosan had not produced settleable flocs. The metal–chitosan combination was shown to be stable in solution. According to Guibal (2004), chitosan is primarily employed to adsorb metal ions in a range of solid forms, like beads, flakes, and membranes, due to their low removal capability via other treatment technologies like coagulation and flocculation. TBS and TKP also have no toxicity, which is a concern since SWP residues might harm aquatic creatures (Bolto & Gregory 2007). This may not be an issue if the treated water from the preceding step is used as a fertilizer because research has shown that the presence of dissolved organic matter in the nutrient solution at low concentrations increases plant development and nutrient absorption, resulting in better yields (Goddek *et al.* 2016). These findings are consistent with those of Ezziddine *et al.* (2020).

The study's findings demonstrate that the sunlight photocatalytic degradation of organonitrogen contaminants to inorganic nitrogen compounds, followed by flocculation treatment with environmentally friendly compounds of raw aquaculture effluent, resulted in a nutrient-rich solution. This solution can be safely recovered and used as fertilizer for plant development in soilless growing systems (Al-Jaloud *et al.* 1993; Bian *et al.* 2015; Ezziddine *et al.* 2020).

## CONCLUSIONS

According to this study, photocatalysis with sunlight followed by solids precipitation with natural flocculants (TKP and TBS) is a potential approach for mobilizing and recycling nutrients in aquaculture effluent. In soilless growing systems, the nutrient solution produced can be utilized as fertilizers. During the photocatalysis process, the concentrations of inorganic nitrogen increased to 17.2, 18.1 and 15.9 mg/L for ammonium, nitrite, and nitrate, respectively, after 12 hours of circulation. Natural TBS, rather than natural TKP and SWP, were shown to be a viable option for removing TSS and colour following the photocatalysis procedure.

The highest turbidity reductions using TBS, TKP, and SWP after coagulation, flocculation and sedimentation were 95, 93, and 94% at doses of 20, 20, and 50 mg/L, respectively. TSS maximal removal efficiencies of TBS, TKP, and SWP were determined to be 90, 87, and 89%, respectively, at doses of 20, 20, and 50 g/L. At doses of 30, 30, and 50 mg/L, COD maximal removal with TBS, TKP, and SWP was determined to be 97, 94, and 88%, respectively. At the maximum dose, colour removal was 55, 53, and 29%, respectively.

In terms of coagulant activity, TBS was a somewhat better coagulant than TKP and significantly better than SWP. Following the two procedures on aquaculture effluent, the nutrient-rich clear phase water can be supplied for use as nourishment in soilless growing systems as the nitrogenous compounds significantly increased in the wastewater. In such scenarios, the effect of the retrieved nutrient water on plant development should be investigated further.

## DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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