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Impact of ultraviolet light spacing and fixed zinc oxide resin on total nitrogen and phosphate removal in a continuous photocatalytic reactor

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ABSTRACT

The tofu industry produces wastewater with high concentrations of total nitrogen (Total-N) and phosphate, which can pose serious environmental hazards if left untreated. This study investigates the performance of a continuous photocatalytic reactor using Resin-Immobilized Photocatalyst-ZnO (RIP-ZnO) in reducing these contaminants. The reactor was operated with three variations of RIP-ZnO mass (25, 37.5, and 50 g) and different distances between the UV lamp and water surface (0, 10, and 20 cm). Wastewater samples were collected intermittently at four time intervals (0, 4, 8, and 12 hours) to evaluate the residual concentrations of Total-N and phosphate, in accordance with SNI 6989.11:2019 standards. Among all tested combinations, the optimal result was achieved at the 4-hour sampling point using 37.5 g RIP-ZnO and a UV lamp distance of 0 cm, where the residual Total-N concentration was 19.60 mg/L and the residual phosphate concentration was 16.9 mg/L. The highest initial concentrations reached up to 70.35 mg/L for Total-N and 36.85 mg/L for phosphate. The removal mechanism involved photocatalytic oxidation by hydroxyl radicals (•OH) generated by ZnO and ion exchange facilitated by the resin matrix. Statistical analysis using one-way ANOVA showed that sampling time significantly influenced pollutant removal ($p < 0.05$), while UV lamp distance had a less pronounced effect. RIP-ZnO demonstrates high removal efficiency within the early operational phase of the reactor. However, prolonged usage beyond 4 hours leads to increased residual concentrations due to catalyst saturation. Therefore, operational timing and catalyst regeneration are critical considerations for maintaining optimal performance in continuous wastewater treatment systems.

Keywords: photocatalysis, tofu wastewater, RIP-ZnO, Total-N, phosphate.

INTRODUCTION

Fresh water is a valuable asset that every living thing needs. Global challenges caused by extreme weather likes climate change have led to a decrease in the availability of fresh water on earth even though 70% of the earth's surface is water, but of all the water on earth, only 2.5% of clean water can be used and only 1% can be accessed (Mishra, 2023). From 1984 to 2015, climate change and pollution of nature especially in water ecosystem by human caused the loss of over 90,000 km^2 of freshwater surface area (Scanlon et al., 2023). Specifically for water pollution, one of which is due to industrial wastewater. Industrial commercial activities that produce wastewater will mixed with rivers and stream if disposed without treatment (Yaashikaa et al., 2024). One form of water pollution that has a significant impact on reducing the quality and availability of fresh water is industrial wastewater, including waste from household industries such as the tofu industry. The tofu industry generates wastewater with high organic content due to the continued use of simple production technologies, resulting in low resource utilization efficiency, particularly

for raw materials and water, and substantial waste generation (Pamungkas et al., 2023). The liquid waste from tofu production has a chemical oxygen demand (COD) value ranging from 7.000 to 12,000 mg/L, which significantly exceeds the regulatory maximum COD limit of 300 mg/L for soybean processing, as stipulated in Indonesian Regulation PermenLHK No. 5 of 2014. This waste has the potential to pollute aquatic environments if discharged directly without proper treatment (Sayow et al., 2020). Moreover, the high total nitrogen (Total-N) and phosphate content in tofu wastewater can contribute to eutrophication (Siswoyo and Hermana, 2017). Biological methods can be used to treat tofu wastewater, such as anaerobic or aerobic processes using bacteria (Wahyu and Slamet, 2017). However, biological methods have limitations such as requiring large areas of land and long treatment times (Rosariawari et al., 2012), while physical and chemical methods require high operational costs and specifically for chemical methods will produce sludge which is classified as B3 waste and requires further treatment (Rahman et al., 2012). The photocatalysis method offers an alternative solution for treating tofu wastewater. Photocatalysis is an environmentally friendly and sustainable technology that utilizes energy to trigger chemical reactions efficiently (Sun et al., 2023). The basic principle of photocatalysis is a method that utilizes light energy which can be in the form of visible or ultraviolet light used to activate catalysts that function to accelerate the rate of chemical reactions. In the process, photocatalytic reactions usually involve three main processes: CO_2 reduction, water splitting, and organic compound release. Starting from the initial stage of the photocatalysis process, the photocatalyst molecules are first exposed to light and will lead to the formation of corresponding positive holes (h^+) in the valence band (VB) and photoexcited electrons in the conduction band (CB) (Chakravorty and Roy, 2024). Some nanomaterials, such as ZnO and TiO_2, are considered attractive for photocatalytic processes, but in particular ZnO shows higher photocatalytic efficiency and greater compound adsorption capacity than TiO_2 because ZnO has a larger surface area (Murti et al., 2025). ZnO has characterizations such as; large exciton binding energy (60 meV); wide band gap (3.7 eV); easily binds holes (hþ) and electrons (e-) generated from photogeneration; and has low toxicity. ZnO was shown to exhibit higher photocatalytic efficiency mainly for organic pollutants due to its nature as an n-type semiconductor (Sun et al., 2023). ZnO can degrade COD and BOD into CO_2 and H_2O through a photocatalytic oxidation mechanism activated by UV light (Adnan et al., 2022). However, the direct use of photocatalyst powders presents challenges, including difficulties in separating catalyst particles from the treated effluent (Pachwarya and Meena, 2011) and low adsorption capacity (Rosariawari et al., 2012). To address these limitations, ZnO photocatalysts can be immobilized in resins, forming resin-immobilized photocatalyst-ZnO (RIP-ZnO), which enhances photocatalytic activity and simplifies the separation process. RIP-ZnO has effectively reduced COD and BOD concentrations in tofu wastewater by using the main components of resin and ZnO (Lavyatra and Hidayah, 2022). Factors such as the mass of catalyst in the reactor and the distance between the UV lamp and water are known to affect the efficiency of the photocatalysis process. Although increasing the amount of catalyst can enhance the degradation of pollutants, excessive catalyst mass will cause agglomeration and decrease the efficiency of sewage treatment (Asrori et al., 2022). Optimal UV lamp spacing is also very important to ensure sufficient energy input for the photocatalytic reaction (Hermawan, 2019). Previous research conducted by Lavyatra and Hidayah (2022) is known to show the effectiveness of RIP-ZnO in a batch system to reduce COD in tofu wastewater. In this study, a continuous photocatalytic reactor was used to investigate the effects of RIP-ZnO mass variation and UV lamp spacing on the degradation of COD, total nitrogen (Total-N), and phosphate. Determination of the optimum reactor conditions is expected to support the initial step of large-scale application to be applied in treating tofu industry wastewater.

METHODS

This research was conducted with the main object using tofu industry wastewater with a continuous scale reactor. Tofu industry wastewater was collected directly from the source as a research sample. Before the experiment, the wastewater was filtered to remove coarse particles. The resin was modified with ZnO photocatalyst, forming resin-immobilized photocatalyst-ZnO (RIP-ZnO), which was synthesized by impregnation method and incorporated into the resin then dried

before use. The impregnation process begins with preparing the materials, including distilled water, resin, and ZnO catalyst, with a ratio of 1000 mL: 30 gr: 30 g. ZnO catalyst was then mixed with resin and distilled water in a glass beaker. For three days, the mixture was continuously stirred in the dark using a magnetic stirrer to ensure thorough homogenization. The mixture was filtered after 3 days to separate the solids from the solution, and the resulting material was washed with distilled water to obtain RIP-ZnO (Figure 1).

ZnO is known to have a wide band gap (3.37 eV). The wide band gap can create opportunities for UV-induced electron-hole pair formation (Ong et al., 2018). The activity of ZnO as a photocatalyst is also enhanced by electrostatic interaction with negatively charged pollutants and physical adsorption (Uribe-López et al., 2021). However, embedding ZnO in the insulating resin matrix could potentially inhibit the electron transport pathway, thereby increasing its specific resistance. Previous research has shown that when immobilizing ZnO in an insulating polymer matrix, such as in low-density polyethylene (LDPE) can increase the specific electrical resistance of the resulting composite due to the insulating nature of the resin that limits the mobility of charge carriers (Benabid et al., 2024). Meanwhile, the incorporation of ZnO with conducting polymers such as polyaniline (PANI) has been shown to increase the electrical conductivity and improve the charge separation efficiency of ZnO-based composites (Sahu et al., 2019). In addition to incorporation with conductor or insulator composites, treatment by encapsulating ZnO polymers can affect its electronic and optical properties by introducing an interfacial barrier. However, radical formation and photocatalytic surface area interactions, such as adsorption, can still proceed effectively (Gilja et al., 2018). Although this may reduce charge mobility, degradation of pollutants can still occur through these surface-based mechanisms.

Wastewater was introduced into the continuous photocatalytic reactor during the process, and the mass of RIP-ZnO was added based on predetermined treatment variations. In accordance with the experimental design, the UV lamp was positioned at a certain distance from the water surface. This study used several tools, including a continuous photocatalytic reactor, UV lamp, and supporting equipment. The photocatalytic reactor is specifically designed to treat wastewater, as well as the UV lamp with a wavelength specification of 365 nm which is used to activate the photocatalyst. Other supporting equipment such as a peristaltic pump to regulate wastewater flow, and a spectrophotometer to analyze Total-N concentration and phosphate concentration, follow the method referring to the SNI 6989.11:2019 standard. A pH measurement was also used to monitor the environmental conditions inside the reactor, with measurements based on the same standard. In the phosphate concentration and Total-N concentration tests, there are several additional reagents used such as ascorbic acid ($C_6H_8O_6$) for the phosphate concentration test, and potassium nitrate (KNO_3) for the Total-N concentration test.

The effluent was treated in the reactor with different residence times or sampling times. During the catalytic process, effluent samples were collected and sampled at 4-hour intervals (0 hour, 4 hours, 8 hours, and 12 hours) to assess the treatment effectiveness. The flow rate parameter of the tofu wastewater was set at 30 mL/min. The reactor was configured as a system of three tubes, each with a diameter of 3.1 cm and a height of 50 cm.

The mass of RIP-ZnO was varied (25, 37.5, and 50 g), as well as the distance between the UV lamp and the water surface (0, 10, and 20 cm). The experimental matrix is shown in Table 1.

Figure 1. RIP-ZnO preparation process scheme

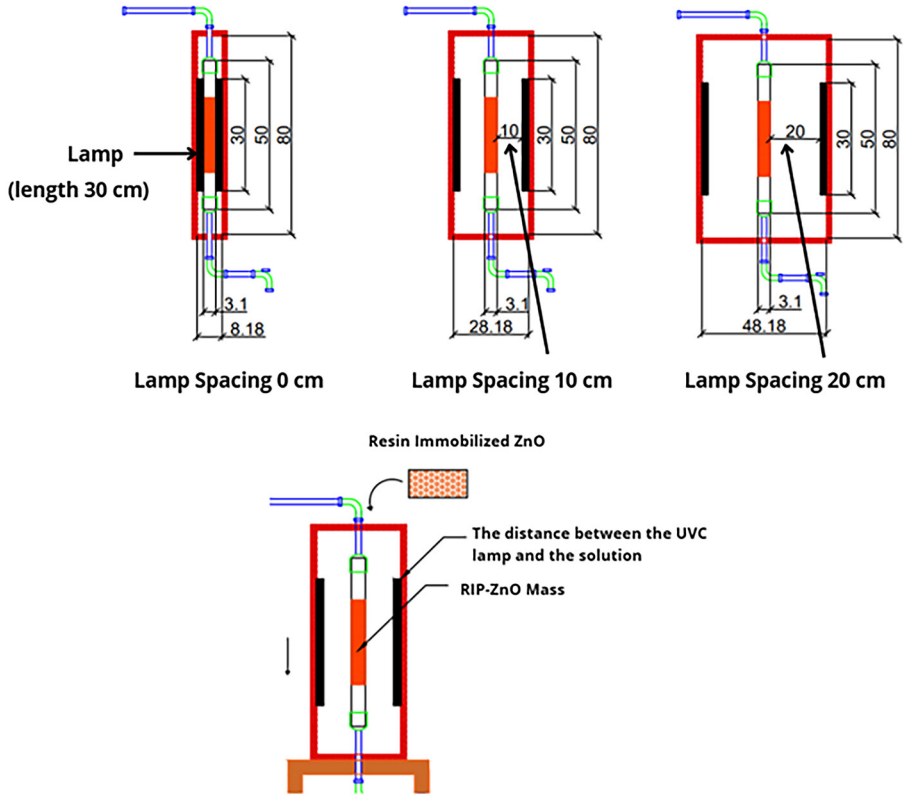

Figure 2. RIP-ZnO reactor design

After conducting the experiments based on the planned research matrix, the results will be analyzed statistically using one-way analysis of variance (ANOVA) to determine the average effect on the removal efficiency of Total-N and phosphate. Statistical analysis will be performed using Minitab 17, a free version of the software.

RESULTS AND DISCUSSION

Total-N removal

The initial Total-N concentration in the tofu wastewater was measured in the range of 59.5 to 70.35 mg/L. The analysis showed that both resin and RIP-ZnO effectively reduced Total-N concentration in the early stages of operation. However, when residual concentration was monitored across increasing contact times (0, 4, 8, and 12 hours), a clear trend of increasing residual Total-N concentration was observed in all variations of RIP-ZnO mass and UV lamp distances, as shown in Figure 2.

This trend indicates that as the reactor operates longer, the RIP-ZnO media becomes progressively saturated, reducing its capacity to retain or degrade nitrogen-based compounds. For instance, in the 37.5 g RIP-ZnO treatment with a 0 cm UV lamp distance, the residual concentration increased from 18.3 mg/L at the initial time to 30.45 mg/L at 12 hours. Similar trends were observed for other mass and distance combinations. This increase in residual Total-N concentration over time suggests a decrease in the degradation or adsorption capacity of RIP-ZnO due to surface saturation. The saturation is caused by the accumulation of Total-N on the active sites of the photocatalyst, resulting in reduced interactions between the UV-activated RIP-ZnO and the pollutants. As operation continues, residual concentrations rise due to limited photocatalytic reaction sites and possible formation of a product layer that hinders further degradation (Luhur et al., 2020). This phenomenon has been supported by previous studies indicating that radiation leads to catalyst fouling and reduced photocatalytic activity (Vifta and Suyanta, 2016; Abbas, 2021).

In addition to the effect of saturation, phytodegradation activity will decreases as the photocatalyst becomes saturated with as long as radiation time (Vifta and Suyanta, 2016). Furthermore, the UV lamp distance also played a role in determine the effectiveness of the photocatalytic

Table 1. Research variable matrix

RIP-ZnO mass (g)	Distance between liquid and lamp (cm)	Sampling time (hour)	Code
25	0	0	A
		4	
		8	
		12	
	10	0	B
		4	
		8	
		12	
	20	0	C
		4	
		8	
		12	
37.5	0	0	D
		4	
		8	
		12	
	10	0	E
		4	
		8	
		12	
	20	0	F
		4	
		8	
		12	
50	0	0	G
		4	
		8	
		12	
	10	0	H
		4	
		8	
		12	
	20	0	I
		4	
		8	
		12	

process. The closer length of the lamp to the water surface (0 cm) makes effective the photon transfer to the surface of catalyst and resulting lower initial residual concentrations. At greater distances, the UV intensity reaching the RIP-ZnO is lower and makes a less effective photoreaction and thus higher residual Total-N concentration. This observation supports the finding that after 40 minutes, no additional ionic substances are removed from the wastewater (Abbas, 2021).

The study also found that the optimal distance between the UV lamp and the water for effective Total-N degradation is 0 cm. Although the optimal condition was observed at 37.5 g RIP-ZnO and a 0 cm lamp distance, further operation beyond the 4 hour led to residual concentrations that exceeded the quality standard threshold. Therefore, while the reactor setup is initially effective, operational time must be carefully controlled to maintain treatment performance.

To better understand the effect of UV lamp placement setting, it is important to consider the principle of radiative flux. The power radiated over a given area results in a flux density or power density per unit area. As the distance from the radiation source increases, the flux density decreases. The flux density of radiation (irradiance) is directly proportional to the intensity of radiation emitted from a source. Radiation is the amount of light power received per unit surface area which is closely related to parameters such as the direction of light and light intensity (Zavafer et al., 2023). The greater the decrease in Total-N removal efficiency is proportional to the closer the distance between the UV lamp and the water. This is due to the shorter distance between the lamps, which triggers photochemical reactions which then increase the photocatalytic activity of RIP-ZnO, as electron pairs are generated faster (Sativa et al., 2021). The experimental results show that the optimal RIP-ZnO mass for Total-N removal in the photocatalytic reactor is 37.5 g as shown by the graph in Figure 3 (a) which has the smallest Total-N residual among other mass variations. This result shows that increasing the mass of RIP-ZnO does not necessarily lead to a proportional increase in Total-N removal efficiency. umotherseesmass of Increasing the mass of RIP-ZnO can increase the percentage of Total-N removal by providing more electron holes available to degrade Total-N. However, adding a larger mass of RIP-ZnO may lead to a decrease in removal efficiency due to the potential agglomeration of the photocatalyst, which reduces the active surface area and, consequently, the photocatalysis activity. This decrease in surface area may reduce the overall pollutant removal efficiency (Ridho, 2021). The percentage of total-N removal was interpreted to be inversely proportional to the total residual total-N. Figure 3 explains that the total residual-N increased as the sampling time increased. However, this indicates that the greater the percentage of total-N removal will result in a smaller total residual. The optimal operating conditions in the continuous photocatalytic reactor for Total-N reduction were found to be achieved using a RIP-ZnO mass of 37.5 g and a UV lamp distance of 0 cm. These conditions allowed adequate contact between RIP-ZnO and tofu wastewater without clumping, and the 0 cm lamp spacing promoted better electron hole formation. The best removal efficiency was observed in the sampling time range of 0 to 4 hours, where the final Total-N concentration met the quality standard of 19.60 mg/L.

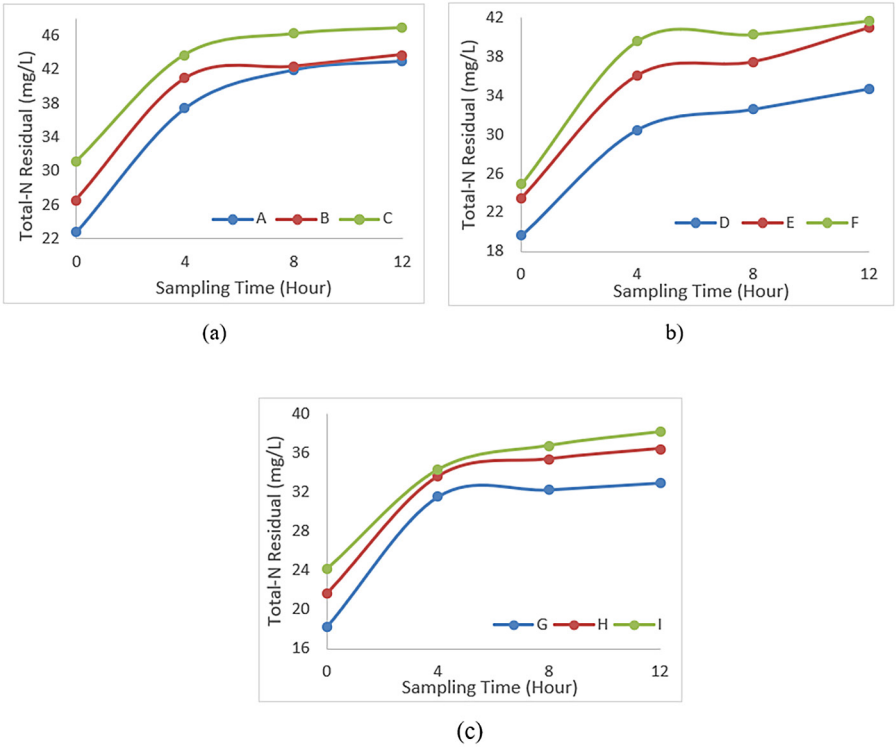

Figure 3. Total-N residual concentration (mg/L) of tofu wastewater using RIP-ZnO 25 g, a) RIP-ZnO 37.5 g, b) and RIP-ZnO 50 g, c) in a photocatalytic reactor

However, at the 4-hour sampling time, the Total-N level approached the quality standard with a value of total-N residual 30.45 mg/L. These findings are consistent with the study by Lavyatra and Hidayah (2022), which showed that ZnO-based resin-immobilized photocatalysts were highly effective in reducing BOD in tofu wastewater, particularly during the early stages of treatment. However, their batch reactor system also exhibited a decline in removal efficiency over time, attributed to the saturation of active sites on the photocatalyst. Similarly, in our continuous system, the highest removal performance for both Total-N and phosphate occurred at the 4 hour sampling point, followed by a gradual increase in residual concentrations at 8 and 12 hours. This comparison supports the conclusion that surface saturation is a limiting factor in prolonged photocatalytic operation, regardless of the reactor configuration.

Phosphate removal

Phosphate levels in raw tofu wastewater ranged from 27.22 to 36.85 mg/L. The use of RIP-ZnO and resin media was effective in lowering phosphate concentrations during the early operation stage. However, similar to the Total-N results, phosphate residual concentrations increased over time with continued reactor operation, as shown in Figure 4.

Phosphate levels in raw tofu wastewater ranged from 27.22 to 36.85 mg/L. The use of RIP-ZnO and resin media was effective in lowering phosphate concentrations during the early operation stage. However, similar to the Total-N results, phosphate residual concentrations increased over time with continued reactor operation, as shown in Figure 4. In the case of 37.5 g RIP-ZnO and a 0 cm UV lamp distance, the residual phosphate concentration rose from 6.4 mg/L at the initial time to 16.9 mg/L at 12 hours. This pattern was also found in the 25 g and 50 g RIP-ZnO mass variations, indicating that prolonged exposure does not continue to degrade phosphate effectively. The increase in phosphate residual is attributed to the surface saturation of the RIP-ZnO media, causing an equilibrium between adsorption and desorption. Once the active sites become saturated, phosphate is no longer effectively removed, resulting in increased residual levels (Aryanto and Nugraha, 2015). The UV lamp distance also influenced phosphate degradation; shorter distances led to more efficient UV activation of ZnO and lower residuals. The optimal result with residual 16.9 mg/L was obtained with 37.5 g RIP-ZnO and 0 cm lamp distance at the 4-hour sampling time.

However, as time progresses when RIP-ZnO become saturated, their reducing degradation capability and leading to an increase in the concentration of phosphate contaminants in the wastewater compared to earlier sampling times, resulting in a decrease total-N concentration (Luhur et al., 2020). The operating time of the reactor is directly related to the duration of photocatalyst irradiation. As the operating time increases, the length of irradiation also increases, leading to a greater accumulation of adsorbed products on the photocatalyst. The accumulation of adsorbed products on the photocatalyst surface can obstruct the interaction between UV light, the photocatalyst, and the pollutants that remain undegraded. This obstruction leads to progressively less effective photodegradation. Additionally, it is hypothesized that as the irradiation time increases, the photocatalyst becomes saturated, resulting in a decrease in its photodegradation activity (Vifta and Suyanta, 2016). The decline in degradation efficiency is likely due to the saturation of the active sites on the RIP-ZnO and native resin by phosphate and other pollutants from the tofu wastewater, which leads to the establishment of an adsorption-desorption equilibrium on the surfaces of both the RIP-ZnO and the native resin (Aryanto and Nugraha, 2015). Phosphate removal consistently exhibited a decreasing trend from the 0-hour to the 12-hour sampling time. It is estimated that the saturation time of the RIP-ZnO media for phosphate removal occurs at approximately 40 minutes. After this period, the photocatalytic degradation of ionic substances reaches its maximum capacity and remains stable, even with further extension of contact time. This suggests that no additional ionic substances are being removed from the wastewater beyond this point. The power radiated over a specific area will have a corresponding flux density, or power density, per unit area. Consequently, when the UV lamp is closer to the water, the percentage of phosphate reduction is higher. This is because a shorter distance between the lamp and the water enhances the initiation of the photochemical reaction, leading to a higher photocatalytic activity of RIP-ZnO, as electron pairs are generated more rapidly (Sativa et al., 2021). Increasing the RIP-ZnO mass can improve phosphate removal by providing more

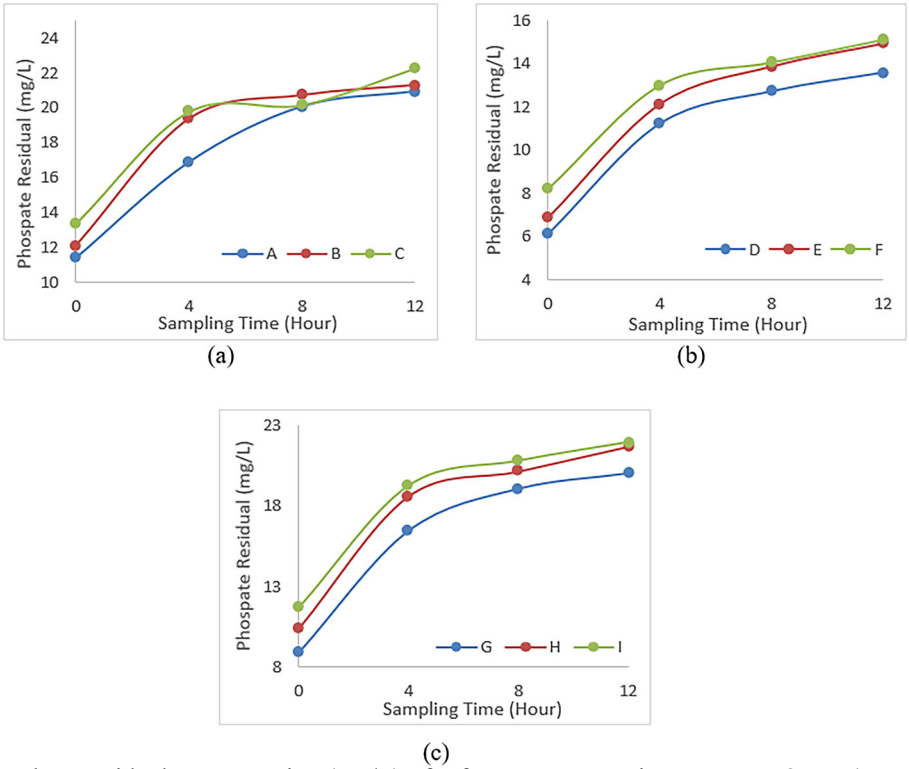

Figure 4. Phosphate residual concentration (mg/L) of tofu wastewater using RIP-ZnO 25 g, a) RIP-ZnO 37.5 g, b) and RIP-ZnO 50 g, c) in photocatalytic reactor

electron holes for degradation, excessive mass may lead agglomeration of the photocatalyst, reducing its effective surface area and decreasing the pollutant removal efficiency (Ridho, 2021).

The optimal operating condition for phosphate removal in the continuous photocatalytic reactor was achieved by using a RIP-ZnO mass of 37.5 g and a UV lamp distance of 0 cm. This setup ensures that the RIP-ZnO maintains effective contact with tofu wastewater, minimizing clumping. A UV lamp positioned 0 cm from the water surface facilitates better electron-hole formation. The best removal efficiency was observed in the range of 0 to 4-hour sampling times. However, even under these optimal conditions, the final phosphate concentration did not meet the quality standard of 6.12–11.23 mg/L.

Statistical analysis and operational implications

One-way ANOVA analysis confirmed that variation in sampling time significantly affected the residual concentrations of both Total-N and phosphate, with p-values below 0.05. In contrast, variations in UV lamp distance did not yield statistically significant differences, though trends still indicated that shorter distances favor lower residual concentrations.

This study highlights the importance of operational timing in continuous photocatalytic systems. While RIP-ZnO demonstrates effective pollutant removal in early operation, efficiency diminishes over time due to surface saturation, which reflects in the increasing residual concentrations. Continuous systems must therefore be optimized not only for catalyst quantity and UV exposure but also for effective cycling or regeneration of the photocatalyst to sustain long-term performance.

CONCLUSIONS

This study demonstrated that a continuous photocatalytic reactor employing Resin-Immobilized Photocatalyst-ZnO (RIP-ZnO) effectively reduced Total-N and phosphate concentrations in tofu industry wastewater. The optimal condition was observed using 37.5 g of RIP-ZnO and a UV lamp distance of 0 cm, evaluated at the 4-hour sampling time, where the lowest residual concentrations were achieved for both pollutants (Total-N: 19.60 mg/L, phosphate: 16.9 mg/L). However, the system's performance declined

with extended operation due to catalyst saturation, as indicated by rising residual concentrations at 8 and 12 hours.

Statistical analysis confirmed that variation in sampling time significantly affected pollutant levels ($p < 0.05$), whereas lamp distance had a less significant effect. Increasing RIP-ZnO mass beyond the optimal point did not consistently improve results, likely due to photocatalyst agglomeration. Future studies should investigate regeneration strategies or periodic replacement of RIP-ZnO to maintain high removal efficiencies in longer operations.

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