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*Research Article*

# Nitrate Removal Enhancement Using Pulse Electrolysis and Aluminum Electrode

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## ABSTRACT

This work focuses on nitrate elimination from municipal wastewater by an electrochemical method with pulsed DC electrolysis. The performance of electroreduction of nitrate was investigated without alkaline electrolyte addition. The nitrate removal reactor was fabricated from acrylic and aluminum electrodes. The nitrate concentration was analyzed by the light absorption of the substance using UV-Vis spectroscopy. The precipitated compounds were determined by X-ray diffraction technique. The experimental results with 180 min treatment indicated the optimum condition for nitrate removal, which was carried out on the pulse frequency of 10 kHz, pulse width of 80%, and electric current density of 16 mA/cm$^2$. This condition was able to eradicate nitrate up to 90.73% (4.3 mg/L) from an initial nitrate concentration of 46.0 mg/L with a nitrogen selectivity of 80.08%. Furthermore, the weight loss of aluminum anodes was as low as 0.8%. The main precipitation formed in the electrolyte cell was aluminum hydroxide.

**Keywords:** Nitrate in water sources, Nitrate removal method, Pulsed DC electrolysis

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## Introduction

Currently, general wastewater sources are often found to have a large accumulation of nitrogen compounds. The occurrence of nitrogen compounds in water sources comes from various sources, such as wastewater from communities that result from the use of tap water or groundwater for household activities such as cooking, dish washing or cleaning. Nitrogen compounds from this type of wastewater typically arise from the decomposition of organic substances contaminated in the water. Wastewater from industrial factories may also originate from the production process involving chemicals that may be released without proper treatment. Wastewater from agriculture can result from the use of nitrogen fertilizers and the use of insecticides such as neonicotinoid insecticides. These chemicals that remain on the soil surface can flow into water sources due to runoff or rainwater washing, leading to the accumulation of nitrogen compounds in water sources [1-3].

The consequences of nitrogen compound accumulation in water sources lead to a process that converts these compounds into nitrate ($NO_3^-$), known as nitrification. This process occurs in two stages. The first stage is ammonia oxidation, where bacteria from the ammonia-oxidizing bacteria group, such as Nitrosomonas, oxidize ammonia ($NH_3$) or ammonium ($NH_4^+$) produced from the decomposition of organic matter in the water into nitrite ($NO_2^-$). The second stage is nitrite oxidation, where the nitrite formed in the first stage is further oxidized into nitrate, which is a stable form that can dissolve in water [4]. The nitrate formed from nitrification can be absorbed by aquatic plants. However, if there is an excessive amount of nitrate in water, it may lead to a condition known as eutrophication, which is the rapid growth of aquatic plants. This excessive growth of aquatic plants significantly reduces the dissolved oxygen in the water, leading to a loss of oxygen, which can cause the death of fish or other aquatic organisms and result in environmental problems [5].

Various technologies have been developed and used to remove nitrate from wastewater including: biological denitrification, physical process and electrochemical denitrification [6-8]. In biological denitrification process, bacteria was used to transform nitrate to nitrogen gas and evaporate into the air [9]. For wetlands wastewater treatment systems, aquatic plants and microorganisms were used to absorb and treat nitrate in water. Biological denitrification is the cheapest method for treatment of nitrate in wastewater. However, this process takes a long time to treat because the reaction or transformation of nitrate to nitrogen gas occurs naturally. Furthermore, for industrial wastewater, it greatly affect the microbial activity and hence biological denitrification technique is not suitable for nitrate removal from industrial wastewater [10].

Among the physical processes, ion exchange and reverse osmosis are the most widely applied treatment methods for nitrate removal [6]. Electrochemical denitrification process has been recognized as a promising method for nitrate removal through oxidative and reductive reaction into water, nitrogen and oxygen [11]. These methods include electroreduction and electrocoagulation [6, 8, 12]. In every electrochemical process, the electrode material is a key variable to be studied [7, 13]. Good electrode materials should have high catalytic activity, high stability, good corrosion resistance and low cost [14-16]. In addition, pulsed-electrocoagulation process for the treatment of different wastewaters has been widely studied [10, 17, 18]. This is due to pulsed-electrocoagulation process is more efficient in wastewater treatment than the DC electrocoagulation technique [19].

The method known as water electrolysis has recently garnered significant attention due to its potential applications in wastewater treatment. This technique involves the use of electrical energy to split water molecules into oxygen and hydrogen gases, effectively removing nitrate contaminants without the need for chemical additives. One of the primary advantages of this method is its straightforward operation and ease of control, making it highly adaptable to various treatment requirements. Additionally, the flexibility in system design allows for integration into diverse wastewater treatment processes, enhancing its applicability and scalability. [20-23]. The efficiency of the electrolysis process is influenced by multiple factors, including the energy input, electrode configuration, and type of electrical supply used. Traditional electrolysis often employs a constant potential or current across the electrodes, a technique referred to as direct current (DC) electrolysis. For this process to proceed effectively, a minimum voltage exceeding 1.6 V is typically required [23]. However, in recent years, pulse electrolysis has emerged as a promising alternative to traditional DC electrolysis for wastewater treatment. This technique introduces periodic pulses of electrical current rather than a continuous flow, offering several key advantages. Pulse electrolysis has been shown to improve system efficiency by reducing energy consumption and mitigating issues such as electrode fouling and corrosion. These benefits position it as a superior and more sustainable approach for the effective removal of nitrate contaminants from wastewater compared to conventional methods [20].

This research aimed to study an eco-friendly approach for treating nitrate-contaminated wastewater by utilizing the pulse electrolysis method with aluminum electrodes, eliminating the need for chemical additives. The study examined the impact of varying the frequency and pulse width of electrical signals on key performance indicators, including nitrate removal efficiency, nitrogen selectivity, electrode corrosion rates, and electrical power consumption. A pulsed direct current (pulsed DC) was employed as the treatment signal due to its potential to reduce the ohmic drop and minimize capacitive losses during the formation of the electric double layer. The periodic switching of polarity in pulse electrolysis ensures that ions cannot fully align or store charge, leading to enhanced operational efficiency. This method not only facilitates the effective conversion of ammonia into nitrogen and hydrogen gases but also reduces energy consumption and mitigates electrode corrosion compared to conventional direct current methods. Consequently, the pulse electrolysis technique offers a promising and sustainable alternative for enhancing nitrate removal efficiency and minimizing the environmental footprint of wastewater treatment processes.

## Experimental

### *Materials and chemicals*

Synthetic wastewater used in the experiment was prepared using potassium nitrate ($KNO_3$) purchased from Merck, Germany, and was prepared at a concentration of 46.0 mg/L. In the electrolysis process, aluminum electrode plates (no.1100, purity 99%) were used. Chemicals used for analyzing nitrites, ammonia, and ammonium were obtained from Hanna Instruments (Thailand) Ltd.

### *Synthesis of wastewater*

Potassium nitrate with a weight of 0.1647 g was prepared and put in a 100-mL beaker. Deionized water was poured into the beaker for 50 ml to dissolve potassium and synthetic nitrate water was obtained.

Later, it was poured into a 1,000-mL volumetric flask. Deionized water was used as a solvent and poured into the volumetric flask. By adjusting the volume of deionized water according to the volume mark from the volume adjustment bottle until it gives synthetic nitrate water with a nitrate concentration of 100 mg/L for 1,000 mL water. Then, it was diluted with 1,222 mL of deionized water resulting in synthetic wastewater contained nitrate with a nitrate concentration of 46.0 mg/L and a volume of 2,222 mL.

### *Experimental setup*

Figure 1 shows the schematic diagram of the experimental setup. The components consisted of a DC power supply, function generator, electronic switch circuit, electrode, electrolysis cell. DC power supply (GW Instek, PSW 30-36) with a maximum voltage of 30 V was used to supply a voltage of 5V. Electrolysis cell was made of acrylic sheet with a dimension (W×L×H) of $8\times10\times7\ cm^3$ and a thickness of 4 mm. The electrodes inside the electrolysis cell consisted of 3 pairs of anode and cathode. The electrodes were made of aluminum (99% purity). Each one has a dimension (W×L) of $8 \times 10\ cm^2$ and a thickness of 0.5 mm. The distance between each electrode was 0.5 mm.

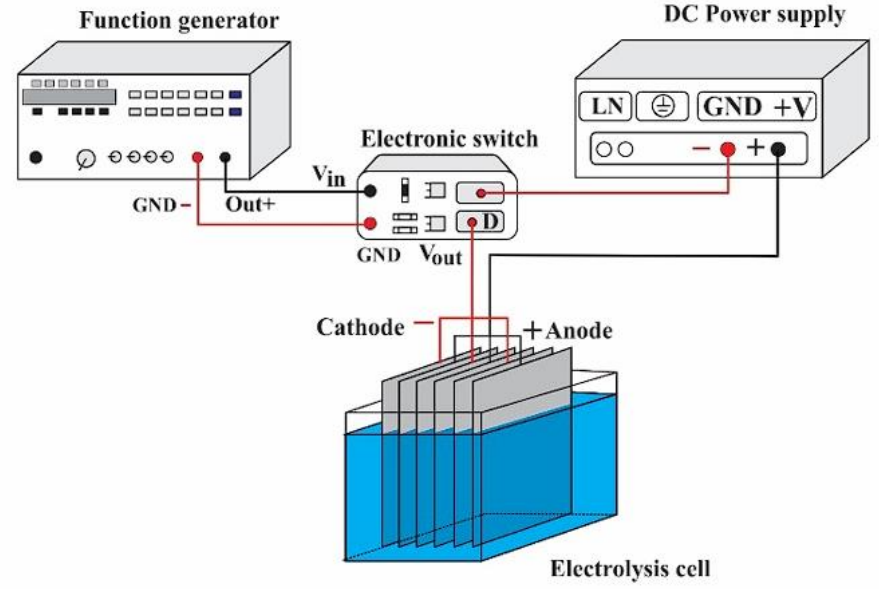

**Figure 1** Schematic diagram of the experimental setup.

The operation begins by connecting the positive terminal of the DC power supply to the anode of the electrolysis cell. Meanwhile, the cathode of the electrolysis cell is linked to the drain (D) terminal of a MOSFET D4184 module, with the system ground connected to the source (S) terminal. A function generator is utilized to manage the on-off switching mechanism by transmitting a pulse-width modulation (PWM) signal to the gate (G) terminal of the MOSFET. To ensure the circuit operates reliably, a 100-ohm resistor is inserted in series with the gate terminal to regulate the current flow, thereby preventing excessive current that could potentially damage the MOSFET and minimizing ripple effects in the PWM signal. In the circuit illustrated in Figure 2, two MOSFETs are employed to regulate the power supplied to the electrolysis cell. The function generator controls the PWM signal, which determines the switching state of the MOSFETs. When the PWM signal is in the high state, the MOSFETs are activated, effectively completing the circuit and allowing current to flow from the DC power supply to the electrolysis cell. Conversely, when the PWM signal is in the low state, the MOSFETs deactivate, interrupting the circuit and halting the flow of current. This on-off switching operation, governed by the PWM signal, induces a pulsed current flow into the electrolysis cell. The use of PWM control provides significant advantages in terms of operational efficiency and precision. By periodically switching the current, the pulsed nature of the flow mitigates energy losses associated with continuous direct current. Additionally, the pulsing action facilitates a more uniform distribution of ions within the electrolysis cell, reducing the risk of electrode overheating or uneven wear. This design ensures the efficient performance of the electrochemical process while preserving the integrity of the electrode materials and minimizing energy consumption.

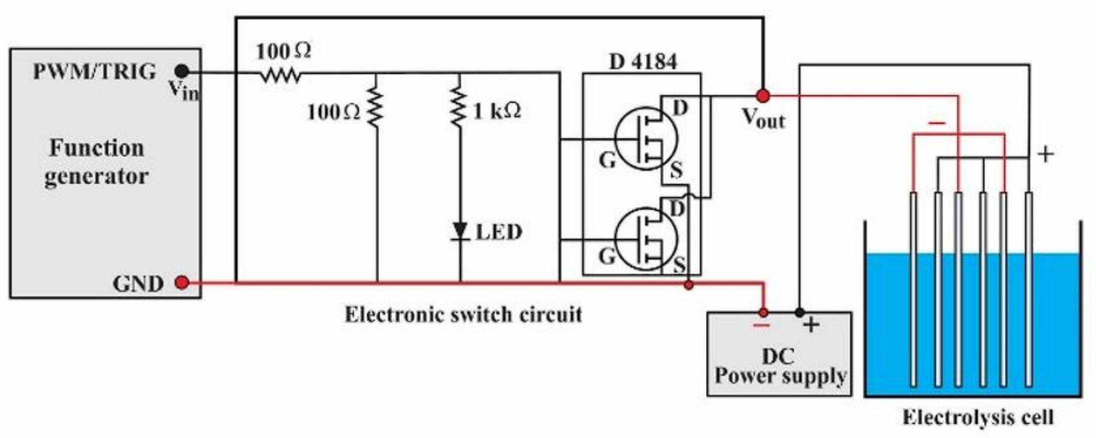

**Figure 2** Diagram of pulsed direct current.

The duty cycle of the pulse, generated by the PWM, determines the proportion of time that the MOSFET remains open during each operational cycle. For instance, a duty cycle of 80% signifies that the MOSFET is open, allowing current to flow for 80% of the total cycle time, while the remaining 20% of the cycle is in an off-state. This characteristic plays a critical role in controlling the power delivery to the electrolysis cell, as the amount of energy imparted during each cycle directly correlates with the duration the MOSFET remains active. The pulse frequency, which refers to the number of complete on-off cycles per second, can be precisely adjusted according to the desired characteristics of the PWM signal. This adjustment is achieved using the function generator, allowing the frequency to be varied over a broad range, typically from a few hertz (Hz) to several kilohertz (kHz), depending on the specifications and capabilities of the function generator and the overall system design. Lower frequencies result in longer pulse durations, while higher frequencies provide shorter, more rapid pulses. Selecting the appropriate frequency is critical, as it influences the efficiency of ion movement and the uniformity of current distribution within the electrolysis cell. To ensure stable operation of the PWM circuit and to maintain signal integrity, a pull-down resistor is connected between the PWM signal leg and the ground. This resistor ensures that the PWM signal defaults to a low state when the function generator is not actively driving the signal, thereby preventing unintended activation of the MOSFET. The inclusion of this resistor is particularly important for minimizing noise and avoiding potential erratic behavior in the circuit caused by floating signals.

Additionally, an LED indicator is incorporated into the circuit to visually represent the status of the PWM signal. The LED provides real-time feedback on the operation of the PWM, illuminating when the signal is in a high state and turning off during the low state. A 1-kilo ohm resistor is placed in series with the LED to act as a current limiter, ensuring that the current passing through the LED does not exceed its rated capacity. This not only prolongs the lifespan of the LED but also protects the circuit from potential damage due to overcurrent conditions. The integration of these components ensures that the PWM signal is accurately controlled, visually monitored, and effectively grounded when necessary. This configuration is critical for achieving precise control of the pulsed current delivered to the electrolysis cell, enhancing the overall efficiency, reliability, and reproducibility of the electrochemical process.

### *Analytical and calculation methods*

The nitrate concentration in the synthesized wastewater was measured using the UV-Vis spectrophotometer (Shimadzu, UV-2600). The absorbance at a wavelength of 218 nm correlates with the nitrate concentration. The nitrate concentration was determined using the equation obtained from the calibration curve. The nitrite and ammonia concentrations were measured by the colorimetric method using a multiparameter photometer (Hanna Instruments (Thailand), HI 83306). A scanning electron microscope (FEI, Quanta 250) was used to observe the surface morphology of electrode. The structure of precipitates from treated water was investigated by an X-ray diffractometer (Rigaku, Smartlab). The nitrate removal efficiency ($R_{[NO_3^-]}$) was calculated by Eq.(1) [24].

$$R_{[NO_3^-]}(\%) = \frac{[NO_3^-]_0 - [NO_3^-]_t}{[NO_3^-]_0} \times 100 \tag{1}$$

where $[NO_3^-]_0$ is the initial nitrate concentration and $[NO_3^-]_t$ is the nitrate concentration at regular time during the electrolysis process. The capability of nitrogen transformation ($S_{N_2}$) is estimated by Eq.(2) [24].

$$S_{N_2}(\%) = \frac{[NO_3^-]_0 - [NO_3^-]_t - [NO_2^-] - [NH_4^+]}{[NO_3^-]_0} \times 100 \tag{2}$$

where [$NO_2^-$] and [$NH_4^+$] are the concentrations of nitrite and ammonia at regular times during the electrolysis process, respectively. Electrical energy consumption (E) in kW.h was calculated from Eq.(3) [25].

$$E = \frac{UIt}{V\cdot(C_0-C)\cdot 60} \tag{3}$$

where U is the voltage across the couple of aluminum electrodes, I is the average current, t is the treatment time, V is the wastewater volume, $C_0$ is the initial nitrate concentration and C is the nitrate concentration after treatment (mg/L). The loss of the anode (m) at a given electric current and electrolysis time was calculated by Eq.(4) [24-25].

$$m = \frac{ItM}{ZF} \tag{4}$$

where F is the Faraday's constant, M is the molecular mass of anode (g/mol) and Z is the number of electron transfers in the reaction.

## Results and Discussion

### *Effect of frequency and duty cycle*

Figures 3 and 4 show the performance of nitrate removal using different applied currents as a function of time for the duty cycle of 50% and 80%, respectively. It can be observed that the nitrate content in wastewater decreased with increasing reaction time. The nitrate removal state can be separated into 2 periods. For the first period (0-90 min) and the duty cycle of 50%, as shown in Figure 3 and Table 1, the treatment process using the direct current (DC) had a higher removal efficiency (71.78 %) as compared to that using the pulsed direct current (pulsed DC) with frequencies of 1 and 10 kHz (approximately 53 and 45%). The nitrate removal efficiency after 90 min still behaved similarly, but the removal efficiency changed to 57.98, 37.27, and 36.75% for the DC and pulsed DC signal at 1 and 10 kHz, respectively. In consideration of the removal rate of nitrate, the DC showed a more significant nitrate removal rate in the early period than the removal rate derived from the pulsed DC signal. However, in the second period (90-180 min), it is interesting to note that the elimination rate values of nitrate from all signals became the equivalent value of about 0.10 ppm/min.

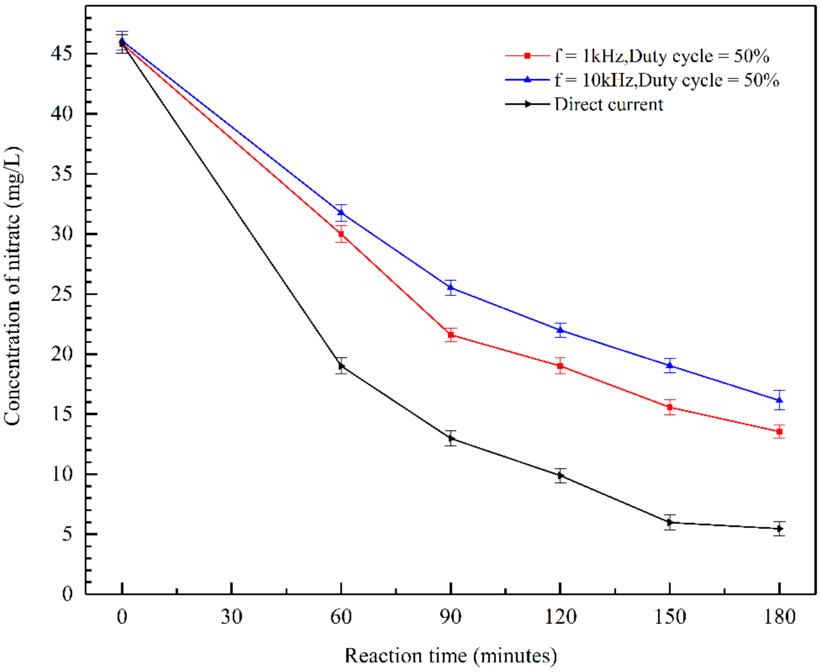

**Figure 3** Nitrate declination with time for the treatment current of 50% duty cycle at 1 and 10 kHz.

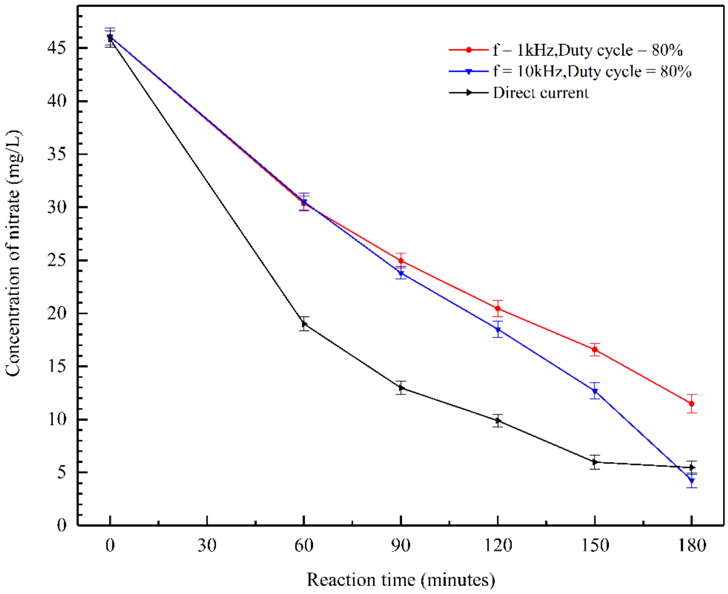

**Figure 4** Nitrate declination with time for the treatment current of 80% duty cycle 1 and 10 kHz.

**Table 1** Removal rate and removal efficiency of nitrate from wastewater using various signals.

| Conditional signal | Duty cycle (%) | Frequency (kHz) | Reduction rate (ppm/min) | | Nitrate removal efficiency (%) | |
|---|---|---|---|---|---|---|
| | | | **0-90 min** | **90-180 min** | **0-90 min** | **90-180 min** |
| Pulsed DC | 50 | 1 | 0.27 | 0.09 | 53.04 | 37.27 |
| | | 10 | 0.23 | 0.10 | 44.52 | 36.75 |
| | 80 | 1 | 0.23 | 0.15 | 45.74 | 53.98 |
| | | 10 | 0.25 | 0.22 | 48.24 | 82.09 |
| DC | 100 | N/A | 0.37 | 0.08 | 71.78 | 57.98 |

Regarding wastewater treatment using the pulsed DC with a duty cycle of 80%, as illustrated in Figure 4, there were no significant differences in the elimination rates of nitrate between 1 and 10 kHz. Their nitrate concentrations at 90 min treatment were still higher than the amount of nitrate in the wastewater treated by the DC. On the contrary, the elimination rate of nitrate of the pulsed DC after 90 min was greater than that of the DC, as seen in Table 1. The removal rate values were 0.15 and 0.22 ppm/min for nitrate removal at 1 and 10 kHz, respectively. These rates were larger about 1.9 and 2.8 times compared to the nitrate removal rate by using the DC. Considering nitrate removal efficiency, the using pulsed DC at duty cycle 80% and frequency 10 Hz displayed the highest value (82.09%) for treatment after 180 min. Meanwhile, the DC had a removal efficiency of only 57.98%. As a result, pulsed DC at duty cycle 80% and frequency 10 kHz was capable of nitrate elimination to about 5 ppm. These values were as low as those derived from using the direct current.

It can be concluded that the use of pulsed DC at 10 kHz and 80% duty cycle is the most suitable condition for reducing nitrate concentration in this experiment because it helps control the formation of electric double layer (EDL) at the electrode area, resulting in more efficient charge transfer and less passivation that may interfere with the redox process. Comparing to lower frequencies such as 1 kHz, which still caused the formation of electric double layer, 10 kHz pulses and 80% duty cycle gave results comparable to or better than DC. Increasing the frequency above this may increase the electrical load unnecessarily. Although DC can reduce nitrate, it has the disadvantage of continuous passivation, which reduces the efficiency of the electrode in the long term.

In addition, the final concentration of nitrate did not exceed the limitation of nitrate concentration in the surface water (5 mg/L) proclaimed in the National Environment Board Announcement (NEBA) issued number 8 (https://www.pcd.go.th/laws/4168/) as seen in Table 2. Furthermore, the remnant total nitrogen of the treatment process using the pulsed DC of 10 kHz and 80% duty cycle was the lowest value (9.2 mg/L). The total nitrogen was considered in the summation of the amount of nitrate, nitrite, and ammonia. This value was lower than the quantity limitation of total nitrogen proclaimed in the Standards for controlling the effluent drainage from community wastewater treatment systems issued by The Ministry of Natural Resources and Environment of Thailand (https://shorturl.at/rXNy6). Using the pulsed DC with these conditions can reduce the total nitrogen over 5 times as compared to the initial total nitrogen (46.0 mg/L). This indicated that total nitrogen was converted to the nitrogen gas approximately by 36.7 mg/L. In Figure 5, the treatment condition of duty cycle of 80% and frequency 10 kHz showed a little bit higher efficiency value in the nitrate to nitrogen gas conversion when compared with using DC treatment. However, the amount of ammonia in the treated water, as shown in Table 2, was in the range of 1.7 to 4.8 mg/L, which was relatively high compared to the ammonia concentration limit (0.5 mg/L) proclaimed in NEBA of Thailand. This issue is interesting for finding a treatment method to remove ammonia without further chemicals.

**Table 2** Final concentration of nitrogen species in treated water for 180 min.

| Condition | | | Amount of nitrogen species | | | | |
|---|---|---|---|---|---|---|---|
| **Current** | **Duty cycle (%)** | **Frequency (kHz)** | **Nitrate (mg/L)** | **Nitrite (mg/L)** | **Ammonia (mg/L)** | **Nitrogen gas (mg/L)** | **Total nitrogen (mg/L)** |
| - | - | - | 46.0 | 0.0 | 0.0 | 0.0 | 46.0 |
| Pulsed | 50 | 1 | 13.6 | 1.0 | 4.8 | 26.6 | 19.4 |
| | | 10 | 16.1 | 3.0 | 3.8 | 23.1 | 22.9 |
| | 80 | 1 | 11.5 | 1.0 | 1.7 | 31.8 | 14.2 |
| | | 10 | 4.3 | 1.0 | 3.9 | 36.8 | 9.2 |
| DC | 100 | NA | 5.5 | 0.0 | 4.2 | 36.3 | 9.7 |

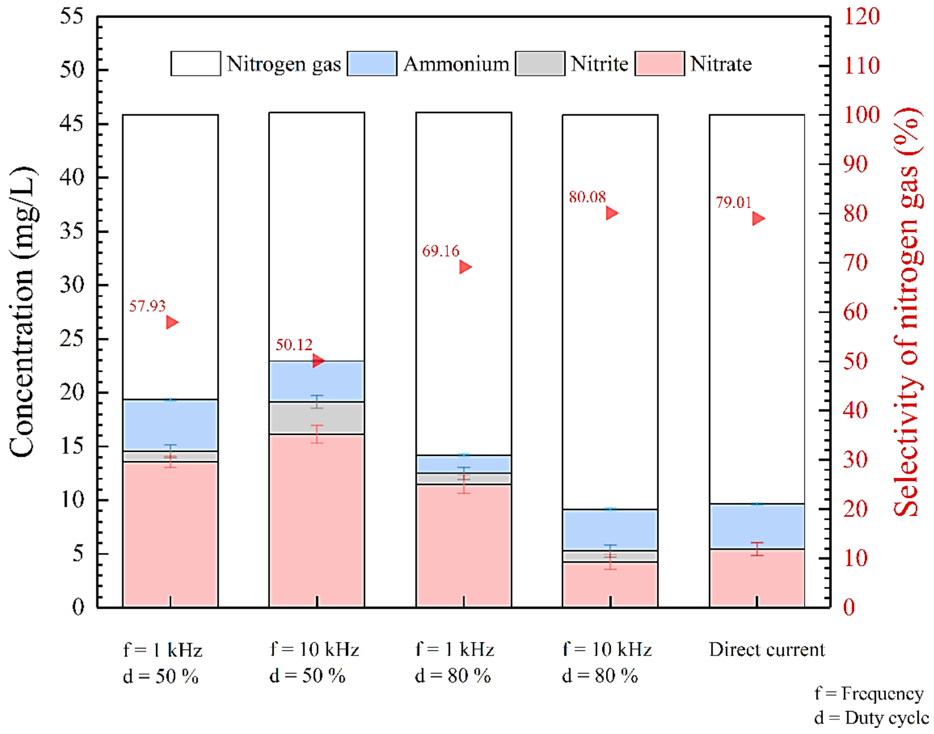

**Figure 5** Final concentration of nitrogen species and nitrogen selectivity after water treatment.

### *Analysis of energy consumption*

The experiment revealed that both DC electrolysis and pulse electrolysis with an 80% duty cycle at 10 kHz are highly effective in reducing nitrate concentrations and possess high nitrogen gas removal capabilities. However, the energy analysis indicated that DC electrolysis consumed more energy than the pulse electrolysis with an 80% duty cycle at 10 kHz, using 0.0113 $kW.h/mg_{nitrate}$ compared to 0.0020 $kW.h/mg_{nitrate}$ for the pulse process. This demonstrated that while both methods have similar nitrate reduction efficiencies, the pulse process was more energy-efficient.

## **Assessment of aluminum anode loss and analysis of sediments produced from the electrolysis process**

In the electrolysis process, the aluminum anode electrode undergoes loss due to electrochemical oxidation, resulting in the formation of aluminum ions ($Al^{3+}$) [24, 26]. These oxidized aluminum ions can react with hydroxide ions ($OH^-$) within the electrolysis system [27], leading to the formation of precipitates within the electrolysis system.

### *Analysis of electrode consumption*

Figure 6 shows that the pulse electrolysis process with a pulse width of 80% and a frequency of 10 kHz resulted in a weight loss of the aluminum electrode after treatment of 0.0801 $g/m^3$ or 0.8% of its initial weight before treatment. This weight loss was less compared to the DC electrolysis process, which had a weight loss of 0.1469 $g/m^3$ or 1.5% of the initial weight after treatment.

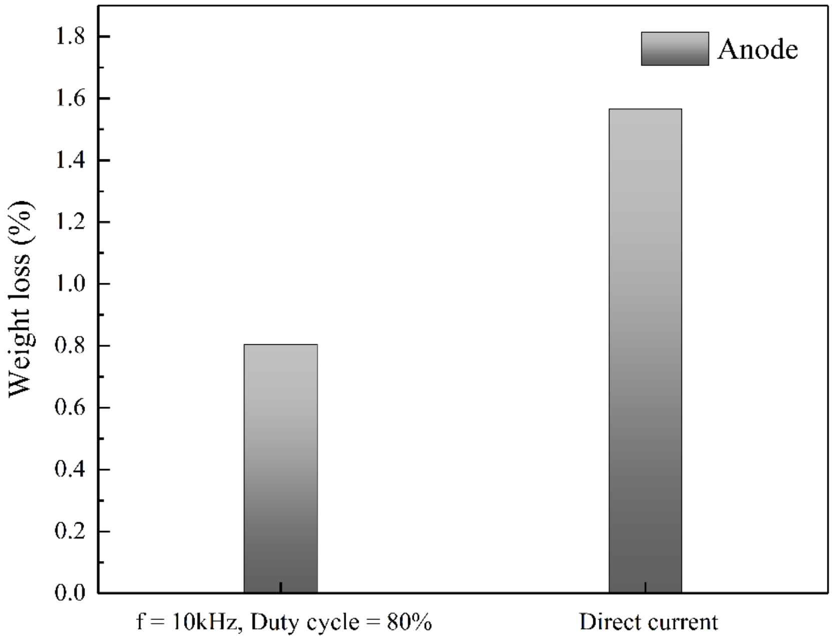

**Figure 6** The precipitate results from aluminum electrodes in pulsed DC and DC nitrate treatment systems.

*Analysis of electrode surface*

From Figure 7, it is observed that the aluminum anode surface exhibited corrosion after undergoing electrolysis. In the case of pulse electrolysis, the corrosion appeard as large pits with a small amount on the electrode surface, as shown in Figure 7(c). This was due to the current being supplied in pulses, where during the off-period, the electric double layer (EDL) had the opportunity to return to equilibrium, resulting in a more uniform current distribution during the on-period.

In contrast, corrosion resulting from DC electrolysis showed small pits with a large amount on the anode surface, as illustrated in Figure 7(b). The continuous current supply led to the accumulation of ions and chemicals at the anode, forming a concentrated EDL, which caused corrosion to occur more intensely at certain spots due to the uneven distribution of the current.

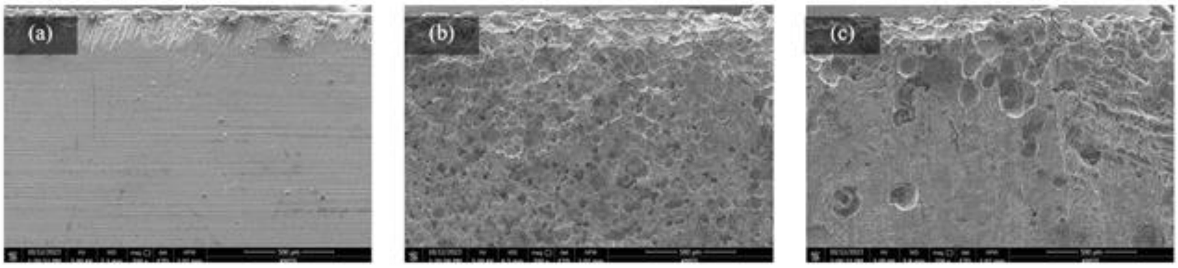

**Figure 7** SEM images of the electrode at 200x magnification: (a) before the electrolysis process, (b) after the DC electrolysis process and (c) after the pulse electrolysis process.

*Analysis of precipitate in the process*

From the XRD analysis, which was performed over a 2θ range from 10° to 80° with a scanning speed of 10.00°/min and a step size of 0.01°, it was found that the precipitate exhibited specific structural characteristics. The analysis revealed that the precipitate has an orthorhombic structure, showing peaks corresponding to the (011), (110), (021), (112), and (212) planes at 15.2°, 17.1°, 22.8°, 34.1°, and 45.1°, respectively. These peaks aligned with the JCPDS standard database, number 01-081-0070.

Additionally, the analysis also identified monoclinic structural features, with peaks corresponding to the (002), (110), (022), (-132), (204), (-313), (332), and (017) planes at 18.8°, 20.3°, 27.9°, 40.7°, 53.2°, 63.3°, 67.3°, and 70.8°, respectively. These peaks correspond to the JCPDS standard database, number 01-083-2256.

The structure of the precipitate thus consists of a mixture of potassium nitrate ($KNO_3$) and aluminum hydroxide ($Al(OH)_3$), as illustrated in Figure 8.

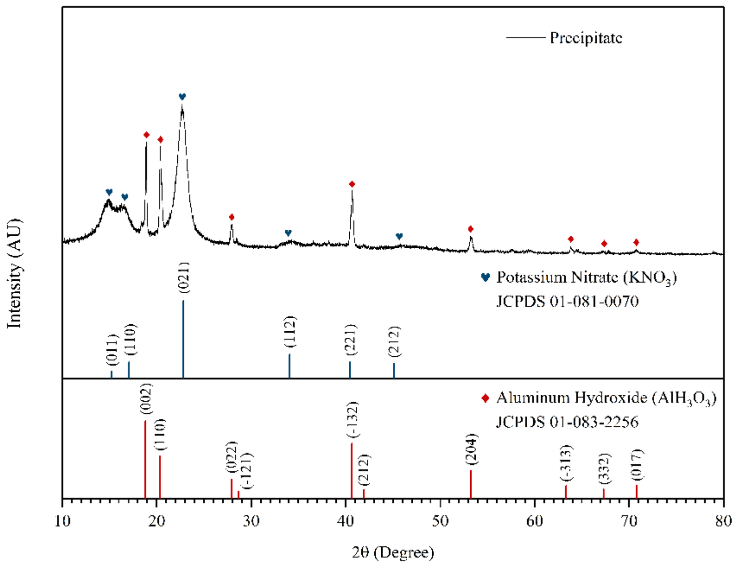

**Figure 8** XRD patterns of precipitate produced.

## Conclusions

The study on the efficiency of nitrate removal from synthetic wastewater using a pulse electrolysis process with aluminum electrodes found that the optimal conditions for this process are a pulse width of 80% and a frequency of 10 kHz. Under these conditions, nitrate reduction efficiency reaches 90.73%, nitrogen gas removal efficiency was 80.08%, and the energy consumption was 0.0020 kW.h/mg$_{\text{nitrate}}$.

In comparison, the direct current electrolysis process consumes 0.0113 kW.h/mg$_{\text{nitrate}}$. Thus, the pulse electrolysis process demonstrates significantly lower energy consumption. Additionally, the weight loss after treatment with the pulse process is 0.0801 g/m$^3$ which is 0.8%, compared to the DC process where the weight loss is 0.1469 g/m$^3$ or 1.5% of the initial aluminum sheet weight. Therefore, the pulse electrolysis process is a better alternative for nitrate removal as it is more energy-efficient compared to the DC electrolysis process.

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