Electrochemical redox treatment of denitrification in coastal secondary effluent using Ti/IrO\textsubscript{2} anode

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Abstract
In northern coastal industrial park, inlet of the wastewater treatment plant (WWTP) had the characteristics of low carbon source and high chloride ion concentration, which resulted in its poor biodegradability. In this case, the experiment explored an electrochemical method to remove nitrogen. Cathodic potential, Ti/IrO\textsubscript{2} was confirmed as the anode and −1.6 V was taken as the potential in order to remove nitrate-N. The findings include: when the initial chloride ion was 2000 and 3000 mg/L, the effect on the removal difference of nitrogen was slight. When the electrolysis time was 60 min, ammonia-N was removed completely, nitrite-N concentration kept 1 mg/L approximately. The ammonia-N removal efficiency went up with the increasing cathodic potential, and was completely removed in different water samples, but nitrate-N removal showed an opposite result. The production amount of nitrite-N was the least at −1.6 V. As the pH increased, ammonia-N and nitrate-N’s removal efficiency went up first and then down, the removal effect was the best at pH being 9, Nitrite-N was less influenced by pH. After optimizing the raw water sample, Nitrate-N and TN removal efficiency were significantly increased, but the nitrite-N almost kept constant.

Keywords Electrochemistry · High chloride ion · Nitrate-N · Ammonia-N · Low carbon source

Introduction
China has pulled the discharge standard of pollutant from WWTP up to a higher level. The discharge standard upgraded from Level 1B to Level 1A of Discharge standard of pollutants for municipal wastewater treatment (GB18918-2002) (Chinese national standard). Now many WWTPs in north china are required to meet a higher level to quasi-four standard. Therefore, they are facing the promotion of quasi-four standard. To meet the standard, there are a number of technical barriers to be tackled, one of which is how to lower the concentration of total nitrogen (TN) to 15 mg/L. Many WWTPs are facing the fact that they are lack in carbon sources (an indispensable substance), which causes the TN discharge hard to meet the discharge standard. Compared with municipal WWTPs, costal industrial park plants in north China are confronted with such complicated problems as complex influent quality, low concentration of carbon source and high nitrate-N and ammonia-N concentration. All of this makes it very difficult to meet the new discharge standard.

There is little research on denitrification of influent from coastal industrial park WWTPs. The groundwater level in coastal areas is high, leaking to sewage pipelines, which leads to a high chloride concentration in influent of WWTP, especially in rainy seasons. Its chloride ion concentration can reach up to 10000 mg/L. It becomes a subject for many engineers and scholars to make full use of the existing high chloride-ion concentration instead of carbon source to remove nitrogen. Current research mainly focuses on the configured sample in the laboratory. However, there is little experiment on the factual wastewater sample with high chlorine and low carbon from coastal industrial parks WWTPs.

Two methods are often employed to treat this kind of water, biological and electrochemical denitrification. Since
the former requires many carbon sources, the latter becomes a better alternative.

The transformation mechanism from ammonia-N and nitrate-N to nitrogen gas was not the same as different denitrification technology. Wang took photoelectrochemical technology to remove ammonia-N, (Wang et al. 2014), which was absorbed onto the electrode and then electrolyzed to nitrogen gas. For photocatalytic fuel cell (PFC), ammonia-N tended to be oxidized to nitrate-N instead of nitrogen gas (Zhang 2018). In neutral solution, ammonia-N is oxidized by hydroxyl radicals produced by dissolved oxygen in water instead of adsorbed on the electrode, but the reaction process is very slow (Chun 2018). When chloride ion exists in the solution, ammonia-N can be oxidized to nitrogen gas or nitrogen oxides (Garcia-Segura 2019; Kamai 2017). In the study of nitrate-N electrochemical reduction process, the nitrogen-containing substances could be removed well (Teng 2018). Co3O4/T (Choi 2018), Pd–Cu/γ-Al2O3 (Yao et al. 2019) was taking as the electrode. For Pd-coated was used as the alumina pellets oxidation electrode (Beltramea 2020). Each above electrode has its own advantages and disadvantages, and its application field is narrow. For the electrochemical denitrification of wastewater with high chloride concentration, the chosen of cathode was especially important. Ti/RuO2 as the anode has a good corrosion resistance, conductivity, long service life and lower chlorine evolution overpotential (Yue 2017); SnO2 and IrO2 was always used to improve the stability of RuO2 anode and prepared coated Ti electrode for wastewater degradation (Kaur 2019) and has been considered as promising electro-catalytic electrode (Zhu 2021). IrO2 electrodes have good conductivity, stability, corrosion resistance, low oxygen evolution overpotential and good dispersion performance (Coster 2017). So, Ti/IrO2 was always taken as the anode for removing nitrate by electrolysis and got a good result (Miao 2009). In addition, many researchers explored much redox experiment (Zhang 2013), under the optimal conditions, had better nitrate-N reduction effect (Kim 2019; Liu 2019). So in the experiment, Ti/RuO2-SnO2, Ti/RuO2-IrO2 and Ti/IrO2 were all taken as alternative electrode materials.

Some researchers conducted the experiments with the existence of chloride ion in solution, which can accelerate oxidizing ammonia-N (Zhou 2016) and reducing nitrate-N and produce less nitrogenous byproducts (Yao et al. 2019). So during the electrochemical reaction, chloride ion concentration in solution cannot be neglected. The chloride ion not only increased the conductivity of solution, but also participated in electrochemical reaction. This could oxidize the intermediate product (Zöllig 2016) and nitrogen-containing substances (Ma 2016; Raz 2016; Santos 2019). For those experiments, many were conducted in lab with configured solution. However, there were few denitrification experiments on complex, high chloride and low carbon source in wastewater of WWTP in industrial park.

The experiment conducted the denitrification test by electrochemical technology, and made full use of the characteristics of high chloride and low carbon source in tailwater from costal WWTP, and studied the rules of electrochemical denitrification under different electrode materials, cathode potential, chloride ion concentration and pH conditions. The findings provided references for application of electrochemical denitrification of high chloride and low carbon source tailwater from costal industrial park WWTP.

**Materials and methods**

**Electrodes and apparatus**

The experimental apparatus diagram was shown as Fig. 1 and adopted three-dimensional electrode. Cu (Shenzhen Yunding Metal Material Co., Ltd) was taken as cathode (Jiang 2020), Ti/IrO2 (Baoji Longsheng Nonferrous Metals Corporation) was the anode, and saturated calomel electrode (Shanghai Leici Electronic Technology Co., Ltd.) as the referenced anode. The electrodes size were all 5 cm × 6.5 cm × 0.1 cm, the number of cathode is 2 and anode is 1, the effective chemical reaction area was 65 cm² and 32.5 cm² for cathode and anode electrode respectively. Cu electrode was set between the double Ti/IrO2 electrodes and the space between the electrodes was 10 mm. The equipment was made by polymethyl methacrylate with the size 5 cm, 6 cm and 12 cm. Electrochemical work station (Tianjin Lanlike chemical and electricity high technology Co., Ltd) was used to adjust the cathode potential.
Experimental method and conditions

In the test, main monitoring items included TN, nitrate-N, nitrite-N, ammonia-N and pH. TN adopted the ultraviolet spectrophotometric method of potassium perchlorate oxidation. Nitrate-N and nitrite-N took ion chromatography methods. Nessler’s reagent spectrophotometry method was used to test ammonia-N. Chloride ion was prepared by sodium chloride, and KNO₃ and NH₄Cl were used for corresponding solution. All the chemicals were analytically pure and the experimental water was pure water. pH meter was Hach pH meter (HQ11d) (Fig. 2).

Sampling location

Raw tail water sampled from WWTP in Caofeidian new city of Tangshan Hebei China, and it is away 3 km from Bohai bay. The water samples were taken in May dry season. In the dry season, it is less rainfall, and the groundwater leaks less into underground pipes. The chloride ion concentration in in tail water was relatively low. The chloride ion concentration in the sampled water this time was 1171 mg/L. In contrast, groundwater level in rainy season is high, more groundwater leaks into pipes, and the chloride ion in secondary effluent was about 2000 ~ 3000 mg/L or even plus in daily monitoring. Due to the limitation of sampling time and space, the recording high value was about 10000 mg/L. The sampled water quality parameters were as Table 1.

Experimental procedures

Linear volt-ampere curve

This experiment conducted in the solution with 25 mg/LNO₃⁻N and 0.1 mol/L Na₂SO₄. Uniformly negative linear voltage was applied between − 2.5 V and 0 V by an electrochemical workstation. The obtained curve is the linear volt-ampere curve.

Chloride electrolysis

In the experiment, cathode potential was set − 1.6 V, and the initial pH value of electrolyte was 7. In the configured water samples, the initial nitrate-N and ammonia-N was 20 mg/L and 10 mg/L, initial chloride ion was configured as 1000 mg/L, 2000 mg/L, and 3000 mg/L respectively in different solutions. The aim was to investigate the effect of initial chloride ion concentration on denitrification.

Nitrogen removal test under different voltages

In order to explore the nitrogen removal effect, a proper cathodic potential should be got. Known from the linear volt ampere curve in Fig. 3, the electrolytic current increased with applied voltage starting from 0 V. The inflection point appeared when the cathode potential was about − 1.1 V and then the current suddenly dropped. When the cathodic

![Linear volt ampere curve](image)

![Oxygen/chlorine evolution polarization curves of three electrodes](image)

Table 1 Water quality of wastewater treatment plant

| Parameters       | COD₇₆ (mg/L) | pH    | Chloride ion (mg/L) | Ammonia-N (mg/L) | Nitrite-N (mg/L) | Nitrate-N (mg/L) |
|------------------|-------------|-------|---------------------|------------------|------------------|------------------|
| Value            | 66          | 7.3   | 1171                | 1.18             | 0.39             | 8.08             |
potential was −1.2 V, a strong reduction appeared and it was the reduction peak of NO₃⁻. Then, with voltage increasing from −1.2 V, the current increased again. According to the characteristics of the linear volt-ampere curve, the proper potential should be chosen between −1.8 V and −1.2 V and which would be taken as the cathodic potential of denitrification.

Results and discussion
Comparison of electrode materials
In the experiment, oxygen evolution was the main competitive side reaction and it affected the current efficiency. For chlorine evolution, the chlorine evolution over potential determined the efficiency of chlorine evolution reaction, during the process, free chlorine generated with strong oxidation and which oxidized ammonia-N. So the oxidation/chlorine evolution should all be considered in the experiment. Oxygen/chlorine evolution polarization curves of three different anode materials were measured in 0.5 mol/L sodium sulfate and saturated sodium chloride solution, separately. Figure 3 described the oxygen evolution and chlorine evolution polarization curves of the three electrodes under the above conditions. Among them the imbedded figure is the chlorine evolution curve, the large figure was the oxygen evolution curve. The polarization curve in Fig. 3 was composed of two parts: a weak polarization zone and a strong polarization zone, that was, the gentle zone where the oxygen evolution (chlorine evolution reaction) was not obvious in the first stage and the obvious reaction zone in the latter stage. The abscissa of the intersection of the tangents of the two regions is the potential for oxygen evolution (chlorine evolution). For related detailed methods, please refer to the literature (Wu 2010; Li 2018). The difference between oxygen evolution and chlorine evolution was calculated from the difference between the electrode’s oxygen evolution potential and chlorine evolution potential. According to the above analysis, the processes for selecting proper electrodes were stated below.

Three electrodes were used to conduct the experiment. Known from Fig. 3, Ti/RuOₓ-SnO₂ has the smallest difference value between oxygen/chlorine evolutions comparing with that of other two materials. And the value was 0.07 V. The oxygen evolution was the lowest. In the electrochemical reaction, competitive adsorption of OH⁻ and Cl⁻ happened on electrode surface. The smaller of the potential difference, the more the oxygen evolution side reaction occurred and the more current was used for oxygen evolution. This reduced the current efficiency for ammonia-N removal. The oxygen/chlorine evolution of Ti/RuOₓ-IrO₂ (Zhi 2020) ranked the second with value 0.11 V, and the chlorine evolution was the highest 1.13 V. For instance, if the chlorine evolution was too high, it would increase the voltage of reaction tank and energy consumption would increase accordingly. The voltage difference of Ti/IrO₂ was the biggest 0.19 V with the lowest chlorine evolution. The oxygen evolution was the highest among two other electrodes. When the voltage difference of oxygen/chlorine evolution was large and it would reduce the generation of side reaction. The lower the chlorine evolution was, the easier the free chlorine being produced. Thus, the ammonia-N removal efficiency improved. So taking Ti/IrO₂ as anode material was more suitable than the other two ways in this research. Ti/IrO₂ became the most appropriate anode with these advantages, such as highly conducting, physically hard and chemically stable and inert.

Nitrogen removal under different chloride ion concentration
(The initial nitrate-N and ammonia-N concentration in Fig. 4 were 10, and 20 mg/L, respectively. In each three-data-group in the seven time points, ammonia-N, nitrite-N and nitrate-N were tested with the chloride ion 1000, 2000, 3000 mg/L from left to right.)

Seen from Fig. 4, nitrate-N and ammonia-N concentration decreased gradually with the prolonging of electrolysis time. Nitrite-N came to appear within 10 min. Ammonia-N was not detected for the first time at the chloride ion concentration 3000 mg/L and the time 40 min. Electrolysis time 50 min and chloride ion concentration 2000 mg/L, ammonia-N was all removed. All the ammonia-N was removed when the time lasted 60 min. The removal effect of ammonia-N was mainly caused by free chlorine. With the promoting of chloride ion in solution, the producing amount of free chlorine would increase accordingly, so the oxidation efficiency.

![Fig. 4 Effect of different chloride ion concentration on nitrogen removal](image-url)
Effect of cathode potential on nitrogen removal

(In Fig. 5, the initial ammonia-N and nitrate-N concentration was 10, and 20 mg/L respectively. In seven time points, the test was conducted. Four sets of data formed a time point group, and in each group, ammonia-N, nitrate-N and nitrite-N were measured under the cathodic potential −1.2, −1.4, −1.6, −1.8 V from left to right.)

Known from Fig. 5, nitrite-N and ammonia-N concentration decreased gradually with time prolonged. Nitrate-N turned up for the first time in the four water samples within the time 10 min. In the following process, there was no obvious concentration difference under different cathodic potential.

With the increase of cathodic potential, removal efficiency of ammonia-N increased gradually and the best removal efficiency appeared at −1.8 V. When it was 20 min, ammonia-N could not be detected. The followed potential order was −1.6 V and −1.8 V. But for the potential −1.2 V and −1.4 V, the removal efficiency was lower and removed completely within 50 min. The main reason was that the increasing cathodic potential allowed cell voltage to increased, which led to increased current density on unit area, more electrons transferred, more free chlorine. Then the free chlorine reacted with ammonia-N, which showed faster removal efficiency. The result was similar to Guo’s research (Guo 2016).

Fig. 5 Effect of cathode potential on nitrogen removal
In terms of nitrate-N removal, the removal efficiency was the best when the cathodic potential was \(-1.2\) V and it was 71.4\% within 60 min. This was consistent with the linear volt-ampere curve above. The reduction peak occurred at \(-1.2\) V. Next, when the cathodic potential was \(-1.6\) V and \(-1.4\) V, the removal efficiency was 67.4\% and 53.8\% respectively; but for \(-1.8\) V, the efficiency was the lowest with the removal efficiency 52.8\%. This could be explained as the fact that the negative potential shift intensified the occurrence of hydrogen evolution reaction. And the increased hydrogen evolution reaction produced more bubbles, which not only reduced the adsorption area, but also led to the competitive adsorption of H\(^+\) and NO\(_3^−\). This, therefore, reduced the current efficiency and affected the removal efficiency.

The formation of nitrite-N as depicted in Fig. 5, under different cathodic potential, nitrate-N concentration increased faster within the first 10 min, and reached 2 ~ 4 mg/L, but it fluctuated up and down in a certain range after 10 min. when the cathodic potential was \(-1.6\) V, it produced relatively little nitrite-N. Which as the intermediate produce produced during the reduction process from nitrate-N to nitrogen gas, the cathodic potential was less selective to its formation. It also showed that, nitrite-N was a noteworthy factor affecting the removing of nitrate-N.

An interesting phenomenon could be seen from the above analysis and Fig. 5. During the electrolysis process, when the cathodic potential was \(-1.6\) V, the three nitrogen-N substances—ammonia-N, nitrate-N and nitrite-N all achieved the highest removal efficiency 82.3\% and the selectivity of nitrogen generation was higher at this time. Simultaneously, the value was better than that in the reduction peak potential \(-1.2\) V and the removal efficiency was all the same for \(-1.4\) V and \(-1.8\) V cathodic potential and lower. So, it was very important to choose appropriate cathode potential for electrochemical denitrification. If the cathode potential was too low, the current density was relatively low, the catalytic effect was not satisfactory, and the removal efficiency also decreased. If the cathode potential was too high, the hydrogen evolution reaction would be violent, affecting the nitrate-N removal efficiency. So, in the following experiment, \(-1.6\) V was chosen as the cathodic potential.

**Nitrogen removal under different pH conditions**

Seen from Fig. 6, initial pH value in solution had great affect the on the removal efficiency of ammonia-N. When the pH was 3, the removal efficiency of ammonia-N was the lowest with the time 60 min and the value was 48.8\%. The removal efficiency increased with the increase in initial pH value. When the initial pH equaled 9 and the time was 50 min, ammonia-N could not be detected in the solution. Then, with the increasing pH value, the removal efficiency slowed down. When pH was 12 and the time was 60 min, ammonia-N was removed completely, and the efficiency was better than that when the pH value was 3, 5 and 7, respectively. The phenomenon was that, the removal efficiency of ammonia-N gradually increased and then slowed down with the pH value increasing.
This suggested that the removal efficiency of ammonia-N under weak alkali condition was higher than that under strong alkali and acid conditions (Yao 2016). Under the acidic conditions, ammonia-N mainly exists in the form of NH$_4^+$, which is not easy to be oxidized compared with NH$_3$. Moreover, Cl$_2$ overflows easily and free chlorine concentration reduces. Under the condition of strong alkali, there is a large amount of OH$^-$ in solution, which will diffuse to the anode during electrolysis process and adsorb competitively with Cl$^-$. In addition, OH$^-$ reacts with free chlorine forming ClO$_3^-$, which hinders the oxidation of ammonia-N. This is consistent with the conclusion of Qin Jingjing (Qin 2016).

When initial pH was 3, the removal efficiency of nitrate-N was the lowest, only 33.6%. When the initial pH was 9, the best removal efficiency reached 52.1%. However, with pH 12, the efficiency dropped. This was, to some extent, different from Ding Jing and Zhang Dongmei’s research. Dingjing thought the effect of pH on the removal of nitrate-N was not obvious (Ding 2015). Differently, Zhang Mei believed it was beneficial to nitrate-N reduction when the initial pH was low. Their different opinions consisted in the decreasing H$^+$ concentration gradually with the increasing pH. According to formula (1), when the pH value was lower, for example, the pH value was 3, and there is a dynamic equilibrium between $OCl^-$ and H$^+$. When the $OCl^-$ concentration went down, weakening the oxidation of $OCl^-$ on ammonia-N and other nitrogen-containing substances. At this time, all the oxidized chlorides exist in the solution in the form of HClO (Yan 2016). At this time, because HClO has strong oxidizing properties and is not charged, it is easier to approach the cathode surface, and it is the intermediate product nitrous acid produced in the nitrate reduction process. Salt and other nitrogen-containing substances are oxidized. So, The removal efficiency of nitrate-N went down. When the pH was higher, such as the pH value 12, the competent absorption between OH$^-$ or other substances and H$^+$ occurred on the electrode, which hindered the removal of nitrate-N, and was consistent with Zhang’s conclusion (Zhang 2013). The removal efficiency of nitrate-N decreased. The higher removal efficiency appeared at the pH being 9 and 7 with the removal efficiency was 52.1% and 47.1% respectively. That was to say nitrate-N removal was the best when the initial pH value is neutral and slightly alkaline.

To some extent, the production of nitrite-N was affected by pH value, but the effect was slight. In the first 10 min, the nitrite-N concentration went up quickly, and then fluctuated within a certain range, and the concentration value kept between 0.8 and 1.6 mg/L. The lower pH, the less the produced nitrite-N. When the initial pH was 9, the amount of nitrite-N was the least. Under weak alkali condition it was lower than that under strong alkali and acid condition. The effect of nitrite-N removal rule was familiar to that of ammonia-N. Further research is expected in the effect of pH on nitrite-N removal.

### Nitrogen removal efficiency in raw and optimized raw water

Figure 7 depicted the denitrification effect of electrolyzed wastewater samples under different conditions. After 60 min’ electrolysis, it could be seen that the concentration of nitrogen-containing substances showed a downward trend, and the removal efficiency of the optimized wastewater was significantly higher than that of the wastewater without optimization. Under the optimal conditions, the reduction rate of nitrate-N was more quickly, and the removal efficiency was 56.93% and 71.41% respectively before and after optimization. Under the condition of optimal raw water parameters, the org-N removal efficiency was much higher than that of raw water. The nitrite-N concentration increased and stayed at a certain level. In contrast, the concentration of the optimal raw water maintained and showed no obvious change under the optimized operating parameters. This might attribute to the faster oxidation of nitrite-N caused by increasing Cl$^-$ concentration. Ammonia-N was not detected during the electrolysis, because its concentration was rather low, 1.18 mg/L and it had been completely oxidized in a short time. This fact was contrary to the research whose conclusion was that ammonia-N increased first and then decreased while nitrite-N was not detected (Shen 2020). The TN decreased quickly under the optimal parameters with its concentration 3.87 mg/L in 60 min and the removal efficiency being 71.2%. In summary, the optimal operational parameters were better than that or raw water and better electrolysis denitrification. It was consistent with the prepared
wastewater results. That is to say, the chloride ion concentration in rainy season is much higher than other seasons, the electrochemical denitrification efficiency is better, and the tests is supposed to be carried out in the future experiments.

Conclusion

The research conducted the electrochemical denitrification in the tailwater, simulated and optimal tailwater. Tailwater sampled from WWTP of costal industrial park and it had the characteristics of high chloride and low carbon resource. The following conclusions were drawn.

The concentration of initial chloride ion concentration 2000 and 3000 mg/L made no obvious difference to the nitrogen removal. Ammonia-N was completely removed in 60 min, and the nitrate-N concentration was about 1 mg/L. In the course of experiment, with the increasing cathodic potential, the removal efficiency of ammonia-N went up gradually, ammonia-N in different water samples were completely removed completely in 50 min. by comparison, the conclusion on nitrate nitrogen was opposite. When the cathodic potential was −1.2 V, the highest efficiency was 71.4% and the nitrite-N amount was the least at −1.6 V. For −1.6 V, pH had great influence on the removal of ammonia-N: with the increase in pH, the removal of ammonia-N and nitrate-N increased firstly and then decreased. The best result appeared at pH being 9, nitrite-N was less affected by pH value. By comparing the raw and optimized raw water samples, the reduction efficiency of nitrate-N in optimized raw water increased from 56.9 to 71.4%, nitrite-N concentration kept constant, and TN removal efficiency also increased significantly.

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Declarations

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