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# Study on high-efficiency treatment of isophytol wastewater by micro-electrolysis combined with steam–air stripping

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**ABSTRACT**

The production process of chemical vitamin E intermediate is complex and involves a variety of long-chain organic compounds which are difficult to degrade. Furthermore, a large amount of $NH_4Cl$ is used as a solvent in the process of production. Therefore, the wastewater from isophytol production has the characteristics of high chemical oxygen demand (COD), high ammonia nitrogen, high salt and low biodegradability, which will increase the treatment load of the subsequent sewage treatment plant and affect the stable operation of the wastewater treatment plant. In this study, micro-electrolysis combined with steam–air stripping process was used to pretreat the wastewater from isophytol production in order to improve its biodegradability. The experimental results showed that the average COD and $NH_3$–N removal rate of this process could reach more than 93.5% and 97.8%, respectively. Furthermore, the destructive experimental results show that the two processes both can maintain high stability. The combined process also recovers ammonia from wastewater in the form of ammonium sulfate. Therefore, the combined process has the characteristics of high treatment efficiency, economic and environmental protection, and is very suitable for the effective treatment of isophytol production wastewater.

*Keywords:* Micro-electrolysis; Steam–air stripping; Isophytol; Wastewater treatment; Organic matter degradation

## 1. Introduction

In the process of vitamin E production, wastewater containing a high concentration of refractory organic matter and the inorganic solvent is discharged centrally. And isophytol is the main raw material during the production process. Because of its high chemical oxygen demand (COD), high ammonia nitrogen and high salt, this kind of wastewater is one of the typical difficult industrial wastewater [1–3]. Ammonium ions and a large amount of free ammonia are contained in the wastewater of isophytol production, and the concentration of ammonia nitrogen can reach 70,000 mg/L [4,5]. The large discharge of such high ammonia nitrogen wastewater will cause eutrophication and deterioration of water bodies, thus causing great harm to the water environment, which makes the production and life of human beings seriously threatened [6–8]. Therefore, how to treat this kind of high ammonia nitrogen wastewater economically and effectively has become a key issue in the field of environmental protection, which has been widely concerned by environmentalists.

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At present, the treatment methods of ammonia nitrogen wastewater mainly include a physicochemical method and biological method [9,10]. Physicochemical methods include blowing off, ion exchange, folding point chlorination, chemical precipitation, membrane separation, advanced oxidation, electrolysis, and soil irrigation. Biological methods include nitrification–denitrification, simultaneous nitrification and denitrification, short-range nitrification and denitrification, anaerobic ammonia oxidation, anoxic/oxic, anaerobic-anoxic-oxic, sequencing batch reactor and oxidation ditch [11–13]. High concentration (>500 mg/L) ammonia-nitrogen wastewater is usually treated by blowing off or chemical precipitation to recover ammonium products to compensate for operating costs. Low concentration (<500 mg/L) ammonia-nitrogen wastewater was usually treated by biological treatment methods.

Most ammonium in high concentration ammonia nitrogen wastewater exists in the state of ammonium ion ($NH_4^+$) and free ammonia ($NH_3$), and the two remain in balance [14]. The equilibrium of ammonium ions and free ammonium is affected by pH values. The principle of deamination by blowing off is to improve the wastewater pH to convert more ammonium ions into free ammonium, and then the free ammonium escape from the water by the physical action of heating up and aeration blowing off. The blowing off method generally includes air blowing off, steam blowing off and hypergravity blow-off. At present, the air blowing method is more used in the treatment of high concentration ammonia nitrogen wastewater and has the advantages of a high blowing rate and relatively low treatment cost. However, the air blowing method still has some shortcomings, such as the packing layer is easy to scale during the blowing process, which makes the flow of wastewater not smooth, thus affecting the normal operation of the equipment [15]. Therefore, the blowing method is usually used in conjunction with other methods. The steam blowing off is suitable for the treatment of high concentration ammonia nitrogen wastewater discharged continuously with a high ammonia nitrogen removal rate. But its operating conditions are difficult to control, and with high power consumption [16]. The large-scale industrial application of hypergravity-blowing ammonia nitrogen removal technology is less, mainly because the technology is not mature enough. Therefore, the effective treatment of high concentration ammonia nitrogen requires a combination of air or steam blowing methods and other treatment techniques.

Micro-electrolysis technology is a kind of water treatment technology that takes wastewater as electrolyte solution and forms a large number of small primary batteries with iron or aluminum and carbon to produce oxidation, flocculation, adsorption, replacement and other chemical reactions to remove pollutants efficiently [17–19]. This technology is widely used and has a better treatment effect on pharmaceutical intermediate [20,21], dye [22,23], landfill leachate [24–26], electroplating [27] and other wastewater. Isophytol is synthesized by a multi-step reaction of various organic materials. The composition of highly concentrated wastewater formed by the synthesis process is complex, and the organic impurities in water are mostly long-chain compounds. Because organic matter in this kind of wastewater does not have recovery value, a multi-dimensional electrolysis process can be used to continuously degrade macromolecular organic matter and chromogenic substances in wastewater to improve its biodegradability.

In this paper, micro-electrolysis combined with steam–air stripping process was used for the pretreatment of isophytol production wastewater. Considering the process conditions such as the production of a large amount of lime milk and low-pressure steam during the production of isophytol, the process combines steam stripping with air stripping, selects a high-temperature blowing method to remove ammonia nitrogen from high concentration wastewater intermittently to shorten the residence time of ammonia removal process. Synchronously, the ammonia was continuously absorbed by sulfuric acid, the recovery of ammonia was investigated and the recovery value of ammonia was estimated. After deamination, the wastewater enters the micro-electrolysis process for COD and chroma removal, the treatment effect of the micro-electrolysis process was investigated, and the mechanism of organic degradation was discussed. Furthermore, in order to investigate the stability of the combined process, a series of destructive experiments were carried out.

## 2. Materials and methods

### 2.1. Experimental equipment

Micro-electrolysis combined with steam–air stripping process equipment is shown in Fig. 1.

#### 2.1.1. Deamination process equipment

The fillers in the deamination tower and ammonia absorption tower are round plastic fillers. In order to facilitate the full contact of waste liquid and gas in the deamination tower, the device uses two-stage spray (two wastewater return pipes to transport wastewater from the bottom of the tower to two different heights of the deamination tower for spraying) besides filling the deamination tower with fillers. A foam remover is installed at the top of the ammonia removal tower to puncture the bubbles formed between the liquid and the gas in the tower, and nylon wire is used to prevent the bubbles not punctured by the foam remover from entering the ammonia absorption tower. Because there is no steam in the laboratory to increase the blowing temperature, the device temporarily installed two electric heating rods with a power of 4,000 W instead of steam.

#### 2.1.2. Micro-electrolysis process equipment

Four 12 V DC power supply is used as the electrolytic power supply in the multidimensional electrolytic cell. The graphite plate is connected to the positive electrode of the DC power supply as the anode of electrolysis, whereas the steel plate is connected to the negative electrode of the DC power supply as the cathode of electrolysis. The distance between each set of cathode and anode electrodes is 8 cm. Particle activated carbon located in each electrolytic cell is used as the third dimension medium of electrolysis, and some quartz sand (Φ = 1~2 mm, analytical reagent grade and purchased from Chongqing Chuandong Chemical Group Co., Ltd., Chongqing, China.) is installed in the outlet tank of the electrolytic cell.

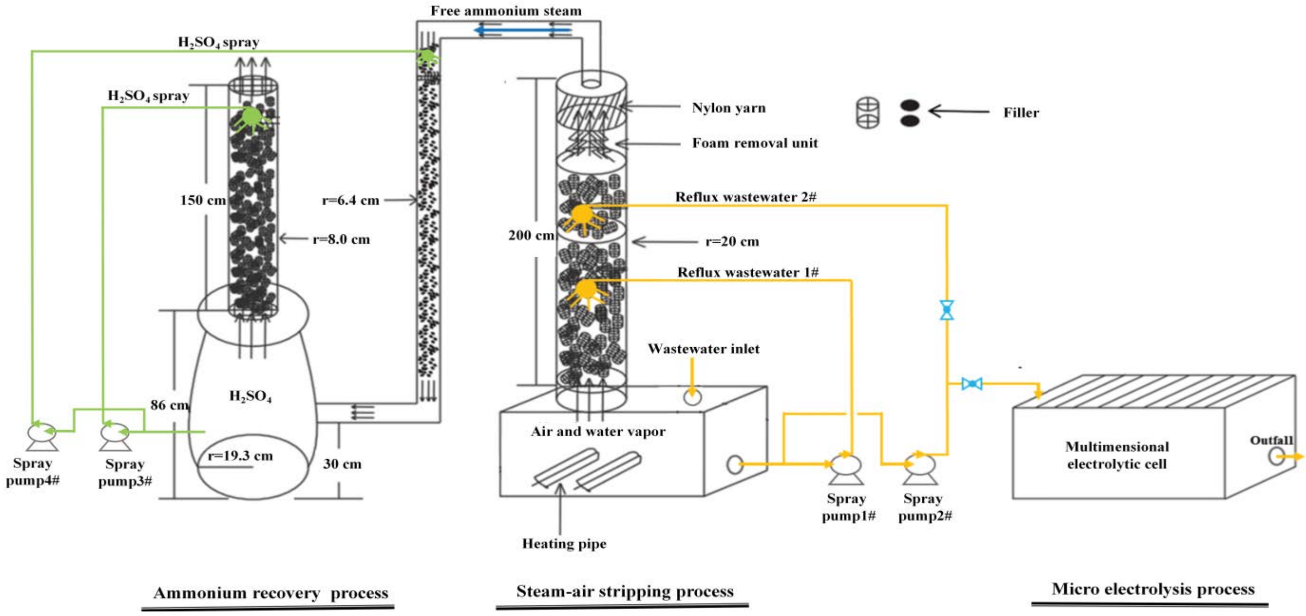


Fig. 1. Micro-electrolysis combined with steam–air stripping process equipment.

### 2.2. Experimental methods

#### 2.2.1. Raw wastewater

The isophytol production wastewater used in the experiment was derived from a raw material medicine production workshop in Sichuan and the experimental wastewater was prepared according to the daily discharge ratio of each post in the isophytol production workshop. The detailed ratio of about 180 L of experimental wastewater is shown in Table 1.

Table 1
Detailed ratio of about 180 L of experimental wastewater

| Wastewater production workshop | Volume of water (L) |
|---|---|
| Product washing water | 30 |
| Condensation reaction wastewater1# | 60 |
| Condensation reaction wastewater2# | 40 |
| Hydrogenation reaction wastewater | 5 |
| Rectification wastewater | 20 |
| Ethynylation reaction wastewater | 25 |

#### 2.2.2. Description of the wastewater treatment process

The calcium carbide slag was added to 180 L of the configured wastewater according to the proportion of 10,000 mg/L, and the pH value of the wastewater was adjusted to about 13.0, and the wastewater was fully mixed and static for 2 h. Then about 35 L sulfuric acid (50 wt%) was prepared into the absorber barrel at the bottom of the absorption tank. After that, the static wastewater was pumped into the bottom tank of the deamination tower, and two sulphuric acid spray pumps, two wastewater spray pumps, a blower and heating rods at the bottom of the deamination tower were started simultaneously. The temperature in the deamination tower was controlled between 60°C~65°C. Until the wastewater in the deamination tower had almost no ammonia flavor, the $COD_{Cr}$ and $NH_3$–N indexes of the effluent from the deamination tower were determined. Then the deamination wastewater was pumped into the intermediate storage tank. Each DC power supply of the electrolytic cell was started, and the wastewater in the intermediate storage barrel was pumped into the electrolytic cell with a pump to control the outlet flow speed of about 30 L/h. The $COD_{Cr}$ and $NH_3$–N indexes of the electrolyte tank effluent were determined according to the frequency of 1 sample per 30 min.

#### 2.2.3. Destructive experiments

Of all kinds of high concentration wastewater, the highest concentration of ammonia nitrogen in "condensation reaction wastewater 1#" wastewater could reach 72,643.5 mg/L. Therefore, in order to investigate the stability of the deamination process, in the destructive experiment of deamination process, 90 L "condensation reaction wastewater 1#" raw wastewater was selected as experimental water for deamination treatment, and the $NH_3$–N and $COD_{Cr}$ of deamination tower effluent was determined for the process stability analysis.

Similarly, of all kinds of high concentration wastewater, the highest concentration of $COD_{Cr}$ in "condensation reaction wastewater 2#" wastewater could reach 58,953 mg/L and no ammonia nitrogen was detected. Therefore, in order to investigate the stability of the micro-electrolysis process, in the destructive experiment of micro-electrolysis process, 120 L "condensation reaction wastewater 2#" raw wastewater was selected as experimental water for electrolytic treatment, and the $COD_{Cr}$ and $NH_3$–N of electrolyte tank effluent was determined for the process stability analysis.

*2.2.4. Analysis of ammonia nitrogen recovery value*

The free ammonium blown out by the deamination tower reacted with sulfuric acid in the absorbent to form ammonium sulfate. The recovery value of ammonium was evaluated by measuring the content of ammonium sulfate.

*2.2.5. Test method of the water quality indicators*

The $COD_{Cr}$, $NH_3$–N and biochemical oxygen demand ($BOD_5$) of wastewater were tested based on the Chinese Environmental Standards HJ 828-2017, HJ 535-2009 and HJ 505-2009, respectively.

All of the experiments in this study were performed with three repeated experiments and the data used in Figs. 3–7 were the average of the results of every three repeated experiments.

**3. Result and discussion**

*3.1. Steam–air stripping process*

The water quality of each batch of the ammonia removal process is shown in Table 2.

As shown in Table 2 and Fig. 2, the $COD_{Cr}$ values of the original mixed water were all below 20,000 mg/L, the $COD_{Cr}$ removal rate of experimental wastewater was between 50% and 65%, and the $COD_{Cr}$ value of effluent could be reduced to less than 9,100 mg/L. Apparently, the deamination process could reduce some of the $COD_{Cr}$ value of experimental wastewater. This part of $COD_{Cr}$ removed was derived from the volatile organic compounds in raw wastewater. This part of volatile organic substances in the deamination tower was blown out into the ammonia absorption device under the action of internal heating and blast, and some were absorbed by sulfuric acid, and the rest floated out of the top of the absorber. It also could be seen clearly from Fig. 2 that the $COD_{Cr}$ removal rate increased with the $COD_{Cr}$ of raw water. On the basis of this trend and the $COD_{Cr}$ removal mechanism, it was not difficult to judge that the increased $COD_{Cr}$ in the original wastewater most come from the volatile organic compounds with low boiling points.

As shown in Table 2 and Fig. 3, the $NH_3$–N concentrations of the original mixed water were all more than 20,000 mg/L, and some experimental mixed wastewater even reached more than 40,000 mg/L. The $NH_3$–N concentration of effluent could be reduced to less than 500 mg/L, the $NH_3$–N removal rate of experimental wastewater were all more than 98%, and the average removal rate could reach 99.0%.

This deamination process has the following advantages: (1) the waste lime milk produced by isophytol production process can be used instead of alkaline agent to adjust the pH value of the original wastewater to alkaline, which can reduce the cost of treatment agent; (2) the waste steam produced in the isobutanol production workshop can be used as both the heat source of the deamination tower and the gas source to blow off ammonia, which can not only shorten the deamination time but also improve the removal rate of deamination; (3) using dilute sulfuric acid to absorb the blown ammonium can achieve the purpose of recovering ammonium. Therefore, the steam–air stripping process

Table 2
The water quality of steam–air stripping process and micro-electrolysis process (mg/L)

| Process | | Batch 1 | | | Batch 2 | | | Batch 3 | | | Batch 4 | | | Batch 5 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ |
| Steam–air stripping | Influent | 18,653.6 | 26,956.8 | – | 19,763.2 | 24,907.4 | – | 15,492.2 | 40,323.5 | – | 18,641.3 | 39,594.0 | – | 19,005.6 | 34,659.8 | – |
| | Effluent | 9,054.3 | 242.6 | 996.0 | 7,659.6 | 174.4 | 1,378.7 | 6,950.5 | 443.6 | 1,668.1 | 8,976.5 | 395.9 | 897.7 | 6,946.7 | 483.9 | 486.3 |
| | Removal rate (%) | 51.0 | 99.1 | – | 61.0 | 99.3 | – | 55.0 | 98.9 | – | 52.0 | 99.0 | – | 63.0 | 98.6 | – |
| Micro-electrolysis | Influent | 9,054.3 | 242.6 | 996.0 | 7,659.6 | 174.4 | 1,378.7 | 6,950.5 | 443.6 | 1,668.1 | 8,976.5 | 395.9 | 897.7 | 6,946.7 | 483.9 | 486.3 |
| | Effluent | 371.2 | 289.7 | 315.5 | 497.9 | 201.6 | 368.4 | 507.4 | 519.4 | 339.9 | 466.8 | 439.5 | 336.1 | 521.0 | 554.9 | 338.7 |
| | Removal rate (%) | 95.9 | −19.4 | – | 93.5 | −15.6 | – | 92.7 | −17.1 | – | 94.8 | −11.0 | – | 92.5 | −14.7 | – |

The $COD_{Cr}$ and $NH_3$–N removal rate of each batch are shown in Figs. 3 and 4, respectively.

has the advantages of high deamination efficiency, environmental protection and energy-saving, thus it is very suitable for the deamination treatment of high ammonia nitrogen wastewater from isophytol production.

### 3.2. Micro-electrolysis process

Table 2 and Fig. 3 show that the $COD_{Cr}$ concentration of the wastewater treated by deamination process could be reduced to 9,100 mg/L, but it is $BOD_5/COD_{Cr}$ was less than 0.3, and thus the effluent from the deamination tower could not be treated directly by the biochemical process. It is necessary to further degrade the refractory organics in order to improve the biodegradability of wastewater to meet the influent water quality requirements of the biochemical process. In this study, the micro-electrolysis process was used to treat the effluent of the deamination tower to improve the biodegradability of wastewater. The water quality of each batch of the micro-electrolysis process is also shown in Table 2. The $NH_3$–N and $COD_{Cr}$ removal rate of each batch are shown in Figs. 4 and 5, respectively.

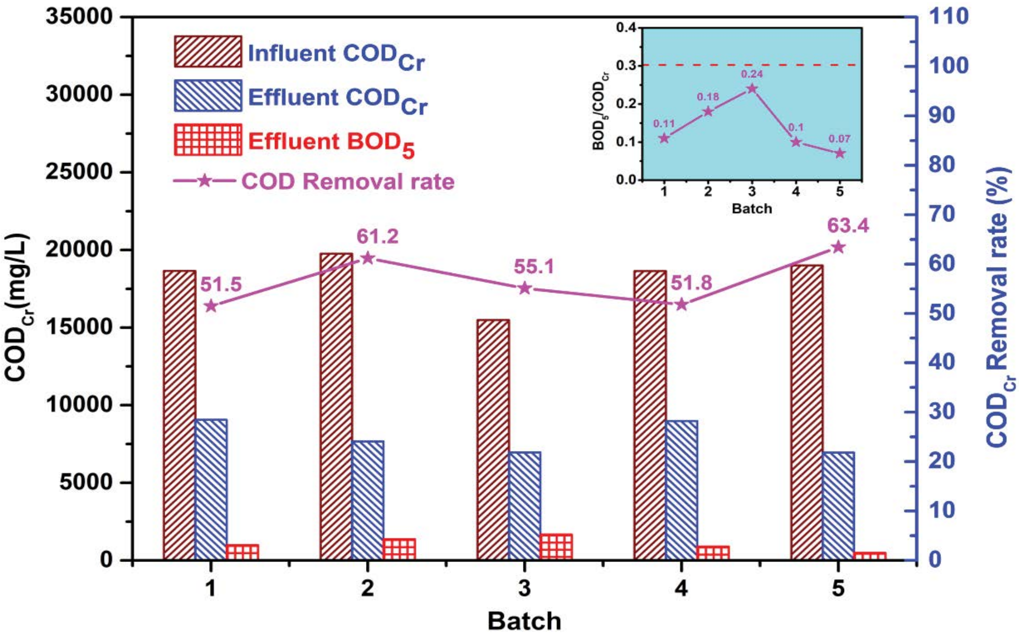


Fig. 2. The $COD_{Cr}$, $BOD_5$ and COD removal rate of each batch for steam–air stripping process.

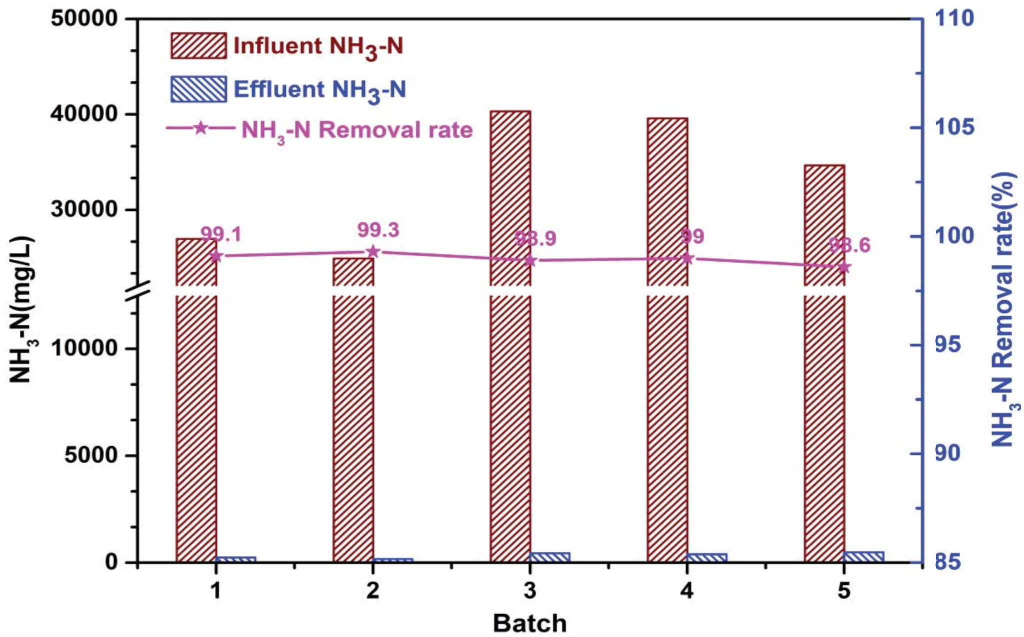


Fig. 3. The $NH_3$–N and $NH_3$–N removal rate of each batch for steam–air stripping process.

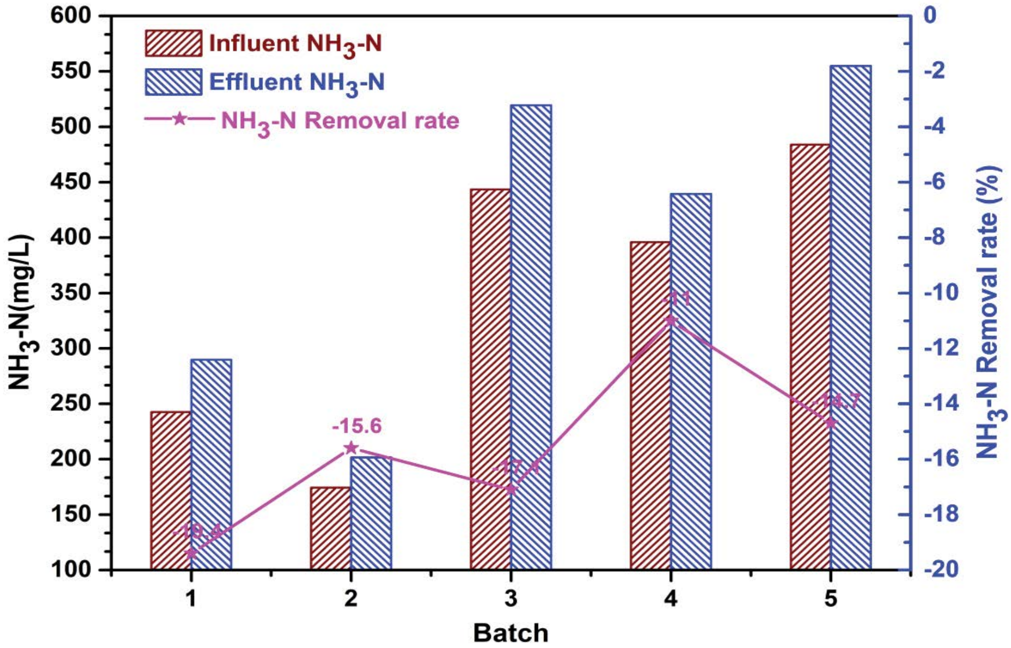


Fig. 4. The $NH_3$–N and $NH_3$–N removal rate of each batch for the micro-electrolysis process.

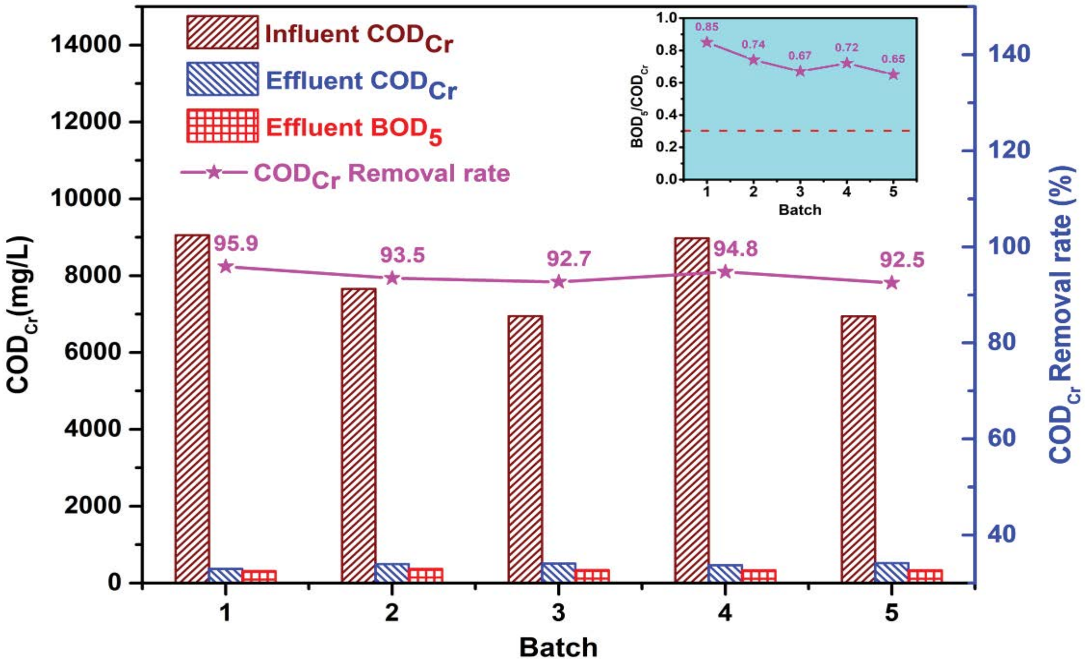


Fig. 5. The $COD_{Cr}$, $BOD_5$ and COD removal rate of each batch for the micro-electrolysis process.

As shown in Table 2, Figs. 2 and 4, after the electrolysis process, the concentration of ammonia nitrogen in the wastewater did not decrease but increased slightly. It is speculated that the reason may be that there was some organic ammonia in the wastewater, and its decomposition in the process of electrolysis led to the increase of ammonia nitrogen in the effluent [28,29]. The $COD_{Cr}$ concentration was decreased to below 550 mg/L, $COD_{Cr}$ removal rate was more than 92%. In the multi-dimensional electrolyzer formed by graphite, steel plate and powdered activated carbon, the refractory organic matter in wastewater was oxidized and degraded by electrons transferred between electrodes. Furthermore, powdered activated carbon was a kind of excellent adsorbent. When the wastewater flowed through the activated carbon in the electrolytic cell, some of the organic matter would also be adsorbed and removed. Therefore, under the dual action of oxidative degradation and adsorption, organic matter in wastewater was effectively removed. The biodegradability of wastewater treated by the micro-electrolysis process was greatly improved (As shown in Fig. 5, the highest $BOD_5/COD_{Cr}$ was 0.85).

### 3.3. Destructive experiments

Five destructive experiments of the two processes were carried out, respectively. The experimental data of destructive experiments in the deamination process and electrolysis process are shown in Table 3. Furthermore, Figs. 6 and 7 show the removal efficiency trends of $NH_3$–N and $COD_{Cr}$ in the destructive experiments of the two processes.

As shown in Table 3 and Fig. 6, the original wastewater used in the five experiments had a very high ammonia nitrogen concentration, the highest concentration was 72,643.5 mg/L. After the deamination process, the ammonia nitrogen concentration in the wastewater was rapidly reduced to less than 280 mg/L, and the average removal rate of ammonia nitrogen was as high as 99.6%. Compared with the previous deamination experiment, the destructive experiment had a higher ammonia nitrogen removal rate, which may be due to the fact that the wastewater used in the destructive experiment was a single component of "condensation reaction wastewater 1#" wastewater which contained less refractory organic amines.

Therefore, the results show that the deamination process could maintain high denitrification efficiency and high stability. As shown in Table 3 and Fig. 7, the original wastewater used in the destructive experiment of the electrolysis process had a relatively high $COD_{Cr}$ concentration and the highest concentration of 58,953 mg/L. The $COD_{Cr}$ of wastewater also dropped rapidly to below 5,800 mg/L after the electrolysis process with an average $COD_{Cr}$ removal rate of 91.5%. Furthermore, the biodegradability index ($BOD_5/COD_{Cr}$) of the effluent from the 5 experiments was above 0.6. Therefore, the micro-electrolysis process also had high removal efficiency of refractory organic matter and high stability.

### 3.4. Ammonia nitrogen recovery value

In this study, sulfuric acid was used to absorb ammonia from wastewater. The product is ammonium sulfate, which has a certain economic value. Ammonium sulfate is mainly used as fertilizer, suitable for various soils and crops, and can also be used in textile, leather, medicine and other fields. After analysis and calculation, 93 kg ammonium sulfate could be produced by treating 1-ton wastewater and the average purity of ammonium sulfate obtained in this study was 89.8% (19.03% of N). Usually, a market value of 19% ammonium sulfate is between 800~1,000 ¥ per ton, and thus each treatment of this kind of wastewater 1 ton can produce 74.4~93 ¥ economic value.

## 4. Conclusion

Effective treatment of high ammonia nitrogen isophytol wastewater has been one of the bottlenecks restricting the production of VE raw materials. It is of great practical significance to study the targeted and efficient treatment process of this kind of wastewater. In this study,

Table 3
The water quality of destructive experiments (mg/L)

| Process | | Batch 1 | | | Batch 2 | | | Batch 3 | | | Batch 4 | | | Batch 5 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ | $COD_{Cr}$ | $NH_3$–N | $BOD_5$ |
| Steam–air stripping | Influent | 1,024.7 | 69,463.9 | – | 894.6 | 72,643.5 | – | 669.7 | 64,532.3 | – | 765.9 | 60,372.2 | – | 876.5 | 61,287.6 | – |
| | Effluent | 591.3 | 264.0 | – | 548.4 | 305.1 | – | 417.9 | 238.8 | – | 472.6 | 181.1 | – | 539.0 | 214.5 | – |
| | Removal rate (%) | 42.3 | 99.6 | – | 38.7 | 99.6 | – | 37.6 | 99.6 | – | 38.3 | 99.7 | – | 38.5 | 99.7 | – |
| Micro-electrolysis | Influent | 51,260.2 | – | – | 58,953.0 | – | – | 56,463.2 | – | – | 53,628.4 | – | – | 53,429.0 | – | – |
| | Effluent | 4,100.8 | – | 3,063.3 | 5,777.4 | – | 3,478.0 | 5,194.6 | – | 3,309.0 | 4,022.1 | – | 2,968.3 | 4,381.2 | – | 3,215.8 |
| | Removal rate (%) | 92.0 | – | – | 90.2 | – | – | 90.8 | – | – | 92.5 | – | – | 91.8 | – | – |

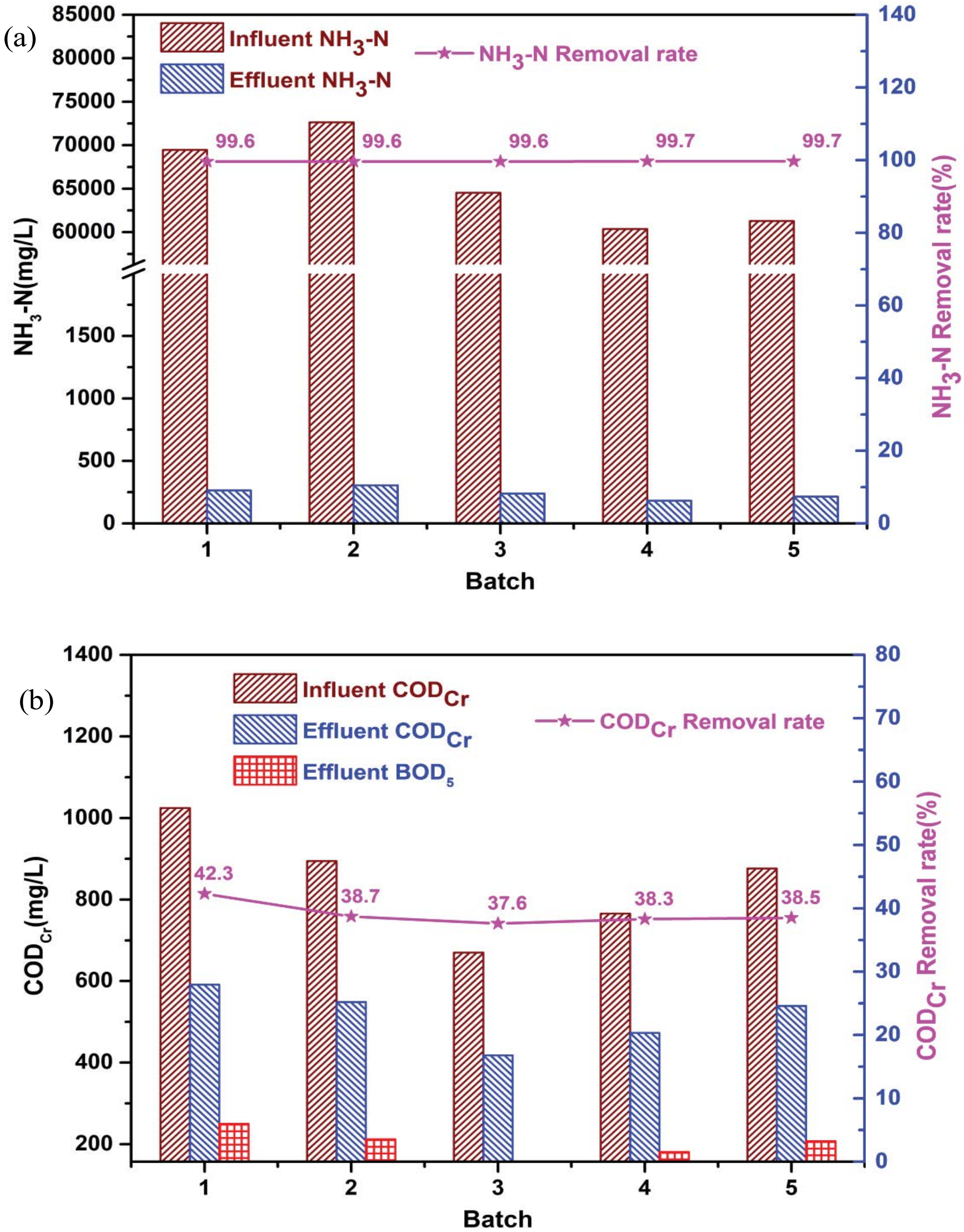


Fig. 6. The removal efficiency trends of $NH_3$–N (a) and $COD_{Cr}$ (b) in the destructive experiments of steam–air stripping process.

micro-electrolysis combined with steam–air stripping process was used to treat this kind of wastewater, the treatment effect and stability were investigated. Experimental results show that the average removal rate of ammonia nitrogen in steam–air stripping process can reach 97.8%, the $COD_{Cr}$ average removal rate of the micro-electrolysis process can reach 93.5%, and the destructive experimental results show that the two processes both can maintain high stability. In addition, the process can make full use of waste calcium carbide slag and waste steam produced in isophytol production workshop in wastewater treatment, which can not only save energy but also improve treatment efficiency. The process also recovers ammonia from wastewater in the form of ammonium sulfate, a valuable product. Therefore, the combined process has the characteristics of high treatment efficiency, economic and environmental protection, and is very suitable for the effective treatment of isophytol production wastewater. This research can provide new ideas for the effective treatment of isophytol production wastewater and other high ammonia nitrogen and high COD wastewater with similar properties.

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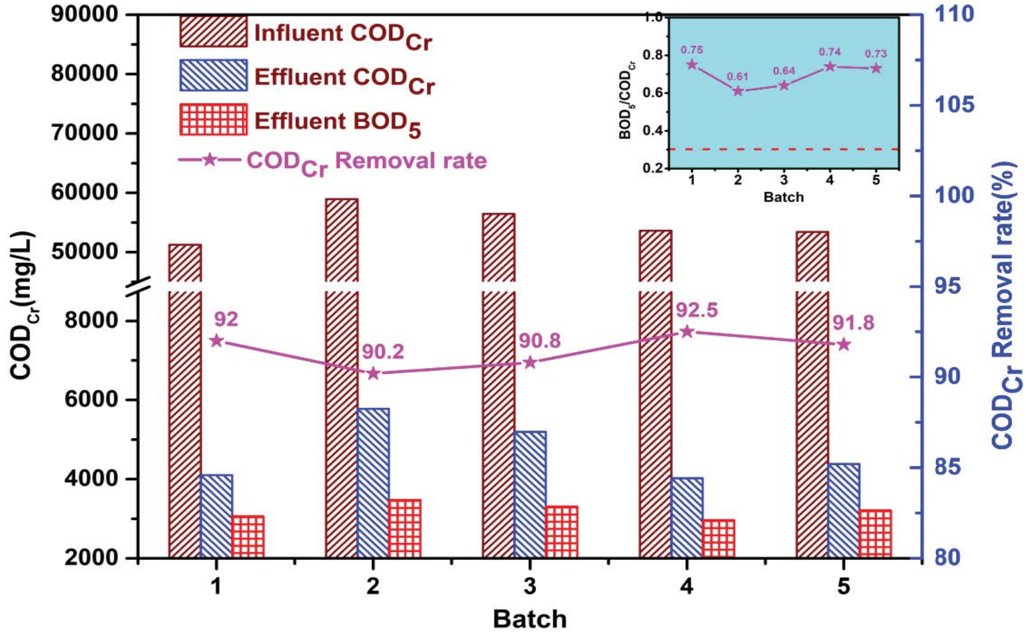


Fig. 7. The removal efficiency trends of $COD_{Cr}$ in the destructive experiments of the micro-electrolysis process.

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