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Study on the pretreatment of high concentration polyester resin wastewater

Penghao Tian, Chao Wang, Mingyang Yang, Junbo Zhou*

College of Mechanical and Electrical Engineering, Beijing University of Chemical Technology, No. 15, Beisanhuan East Road, Chaoyang District, Beijing 100029, China, emails: zhogab@163.com (J.B. Zhou), bucttph@163.com (P.H. Tian), wangchaobuct@163.com (C. Wang), yangmingyangbuct@163.com (M.Y. Yang)

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ABSTRACT

Unsaturated polyester resin (UPR) wastewater is highly acidic with poor biodegradability, which is difficult to treat. In this paper, the UPR wastewater was treated by distillation and cooling to solve these problems. And the effects of distillation and cooling on the parameters, such as conductivity, total dissolved solids, chemical oxygen demand $(COD)_{Cr}$, biochemical oxygen demand $(BOD)_5$ and organic acid content of the UPR wastewater were studied. Through experiments, it was found that there is no obvious effect on the treatment of UPR wastewater by distillation or cooling. But through combination of distillation and cooling, the COD_{Cr}, conductivity and the BOD_5 of the wastewater decreased by 22.4%, 24.6% and 30.9%, respectively, and the pH was increased from 2.23 to 4.03. A mass number of white flocs were precipitated while the irritating odor is significantly reduced, and the biodegradability of the UPR wastewater was improved. The experiment results showed that the distillation or cooling could not affect the parameters above of the UPR wastewater, but the method designed in this paper can easily and effectively treat UPR wastewater, which can be used in industry.

Keywords: UPR wastewater; Pretreatment; Distillation; Cooling; Maleic anhydride

1. Introduction

Unsaturated polyester resin (UPR) is one of the most commonly used thermosetting resins that is used in many production processes, such as automobile, anti-corrosion, construction, electric engineering and other industries [1]. UPR can be cured at normal temperature, formed under normal pressure, which has flexible process properties and stable physical properties. There are a large number of UPR factories in China. The UPR wastewater discharged during the productive process is a typical high concentration organic industrial wastewater. There are many problems in UPR wastewater treatment, which is seriously restricts the development of the industry. The average chemical oxygen demand $(COD)_{Cr}$ of UPR wastewater is 20,000 mg/L and the pH is around 1–2. The composition of polyester resin wastewater is complex. The properties of the resin wastewater are varied that produced by different production materials and processes. In general, the UPR wastewater is difficult to treat that cannot degrade under environmental self-purification easily. If the polyester resin wastewater is directly discharged into environment, it will accumulate in natural media such as soil, water and organisms, which can cause huge damage to the environment [2].

The methods commonly used to treat high-concentration organic wastewater such as UPR mainly include Fenton [3], micro-electrolysis [4] and physical methods such as distillation concentration [5] and cooling crystallization. The Fenton method is suitable for treating the high concentration, hard degradation and high toxicity organic wastewater [6]. The COD_{Cr} removal efficiency of UPR wastewater was above 80% after

* Corresponding author.

treatment by Fenton method [7,8]. But Fenton reagents are expensive and H_2O_2 is highly likely to explode when exposed to light or at temperatures above 40°C [9]. Therefore, some plants with poor storage capacity are not suitable for using the Fenton method to treat the wastewater. Micro-electrolysis is an advanced redox technology [10,11], it has made good achievement in various fields [12]. And the COD_{Cr} removed efficiency of organic wastewater can reach about 70% [13,14]. However, the precipitates generated during the micro-electrolysis process and it will deposit on the surface of the iron filings to passivated it. The large amount of floc produced during the neutralization with alkali will increase the burden of the dehydration stage [15]. And using Micro-electrolysis to treat wastewater will produce so much sludge that causes secondary pollution.

There have been many researches on the pretreatment of organic wastewater by distillation concentration and cooling crystallization. Zhao et al. [16] was pretreated the UPR wastewater by azeotropic distillation. The biochemical oxygen demand (BOD)/COD of UPR wastewater was increased from 0.021 to above 0.3. The biodegradability of the wastewater was improved effectively. Ming et al. [17] extracted avilamycin from 4-methyl-2-pentanone by distillation concentration and cooling crystallization, and the extraction efficiency can reach above 85%, and the organic solvent of avilamycin can be reused with nonpolluting. The physical method of distillation concentration and cooling crystallization not only have a high removed efficiency of organic macromolecules in organic wastewater, but also can separate inorganic salts [18,19] to reduce the conductivity of organic wastewater [20], which is an efficient method to improve the biodegradability of organic wastewater. The organic matter and inorganic salts separated could be reused to reduce the production costs of UPR [21]. Meanwhile, physical methods react quickly, and the treatment process is safe without secondary pollution [22].

In this paper, (1) distillation concentration and cooling crystallization were used to pretreat the UPR wastewater. (2) The effects of temperature changes on conductivity, total dissolved solids (TDS), COD_{Cr}, BOD_5 and acidity of UPR wastewater were studied. (3) And the mixing process that was distillation–cooling, was used to pretreat the UPR wastewater. The organic matter was separated, and the biodegradability of the UPR wastewater was improved.

2. Materials and method

2.1. Materials

The UPR wastewater sample used in the experiment was collected from a chemical industry in Langfang, China. Potassium dichromate (>99.8%, AR), mercuric sulfate (>99%, AR), and concentrated sulfuric acid (>98%, AR) were purchased from Beijing Weiss Chemicals (No. 2, Shangdi Information Road, Haidian District, Beijing 100089, China). All chemicals used in the experiments were without any further purification. And the experimental solutions were prepared in the deionized water (3.09 μS/cm).

2.2. Experimental apparatus and procedure

The experimental set-up is shown in Fig. 1. It mainly consists of five parts: temperature measuring apparatus (TTI-22, ISOTECH, Britain), heating equipment, distillation tower, condenser and distillate receiving bottle.

According to Table 1, the experiment was divided into three groups. Firstly, took 400 mL untreated UPR wastewater (crystal-free clear solution). The temperature, conductivity, TDS, COD_{Cr}, BOD_5 and pH of the UPR wastewater were examined and recorded before adding to the distillation tower. The first experiment was distillation. 400 mL of untreated UPR wastewater (crystal-free clear solution) was heated to boil at 99.2°C. After boiling, the temperature was kept constant for 5 min. After the UPR wastewater was cooled, the conductivity, TDS, COD_{Cr}, BOD_5 and pH of the wastewater were examined and recorded. The second experiment is cooling. 400 mL untreated UPR wastewater

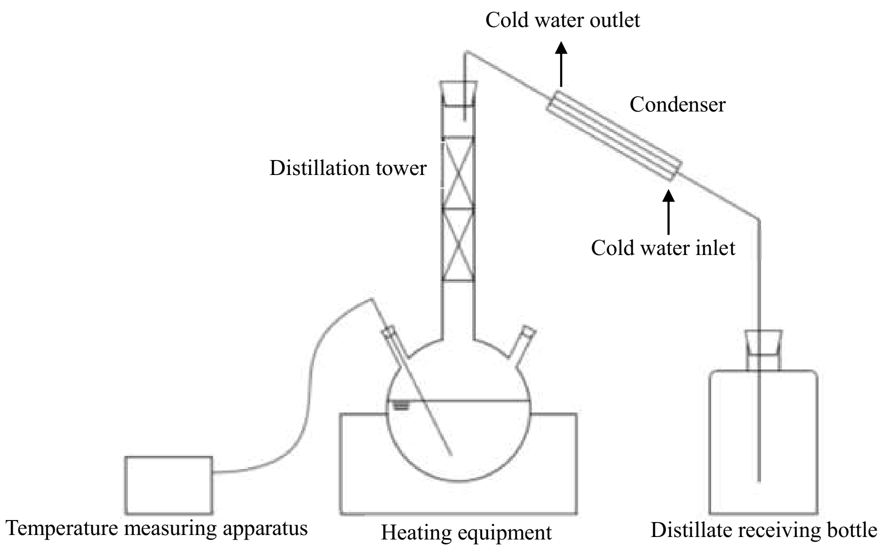

Fig. 1. Distillation experiment apparatus.

Table 1
Experiment process

	Distillation	Cooling
Experiment one	√	
Experiment two		√
Experiment three	√	√

(crystal-free clear solution) was placed in the freezer for cooling. When the UPR wastewater was at 15°C, 10°C, 5°C and 0°C, respectively, it was taken out and the temperature, the conductivity, and the pH were recorded. After the temperature of UPR wastewater was returned to normal, the conductivity, TDS, COD_{Cr}, BOD_5 and pH of the wastewater were examined and recorded. The third experiment is distillation–cooling. Repeat the first experiment of distillation. Then, the distilled wastewater was placed in the freezer for cooling. When the UPR wastewater was at 15°C, 10°C, 5°C and 0°C, respectively, it was taken out and the temperature, the conductivity and pH were recorded. After the temperature of UPR wastewater was returned to normal temperature, the conductivity, TDS, COD_{Cr}, BOD_5 and pH were examined and recorded. After all the experiment was over, the three groups of treated wastewater were sealed with sealing films and stored in a dark place.

2.3. Analysis methods

The initial composition of UPR wastewater was analyzed using the PY-GC/MS at a cracking temperature of 350°C (ISQ 7000 GC-MS, Thermo Fisher, America), the analytical results are given in Table 2. The conductivity was examined using the conductivity meter (COM-100, HM, Korea). The pH was measured by the pH meter (TDS-PH-EC, Ispring, America). The measurement method of TDS was followed by the Chinese national standard, GB/T 5750.4-2006 [23]. The measurement of BOD followed the Chinese environmental standards, HJ 505-2009 [24]. The measurement of COD followed the Chinese environmental standards, HJ 828-2017 [25]. Since the initial COD_{Cr} of the UPR wastewater was too high, it was diluted 100 times to ensure the accuracy before the measurement. The removal efficiency of TDS was calculated according to the following formula:

$$\eta\left(\text{TDS},\%\right)=\frac{\rho_1-\rho_2}{\rho_1}\times 100\% \quad (1)$$

where η(TDS, wt.%) is the removal efficiency of TDS in the UPR wastewater. ρ_1 is TDS of initial UPR wastewater, mg/L. And ρ_2 is TDS of treated UPR wastewater, mg/L. Similarly, the removal efficiency of conductivity is η(conductivity,%), COD_{Cr} η(COD_{Cr},%) and BOD_5 η(BOD_5,%) can be obtained.

3. Results and discussions

3.1. Treatment by distillation

There was no obvious phenomenon found during the distillation experiment. According to Table 3, the conductivity of the wastewater after distillation increased from 8,050 to 9,260 μS/cm that increased by 15%. And the TDS, COD_{Cr}, BOD_5 and pH was changed slightly. According to Table 2, the boiling point of the main components except water in the UPR wastewater is higher than 100°C. But the wastewater could boil continuously at 99.2°C. The main solvent, water and 1,2-propanediol, of the UPR wastewater can form a binary azeotrope with a mixing ratio of 1:1, and its specific boiling point varies with different mixing ratios [26,27]. Therefore, the main solvent of the UPR wastewater, water and 1,2-propanediol, were distilled off at 99.2°C, and the conductivity of TDS, COD_{Cr} and pH were increased after the experiment. Therefore, it can be concluded that the treatment of distillation concentration has no obvious effect on the UPR wastewater.

3.2. Treatment by cooling

A small amount of floc appeared in the polyester resin wastewater at 14°C. As shown in Fig. 2, there was no obvious change about the conductivity of the polyester resin wastewater above 10°C. When the temperature changed below 5°C, the conductivity of the water was decreased by 500 μS/cm, and there was no significant change between 5°C and 0°C. When the temperature of the UPR wastewater returned to normal temperature, the conductivity, TDS,

Table 3
Parameters of UPR wastewater by distillation

Conductivity (μS/cm)	TDS (mg/L)	COD_{Cr} (mg/L)	BOD_5 (mg/L)	pH
9,260	2,573	1.2×10^5	62,500	2.25

Table 2
Main substances in initial UPR wastewater and their physical properties

Materials	Peak area (%)	Melting point (°C)	Boiling point (°C)
Water	27.42	0	100
1,2-propanediol	25.07	–59	188.2
Maleic anhydride	24.42	52.8	202.2
Ethylene glycol	5.59	–12.9	197.3
Phenol	3.05	41	181.9

Conductivity (μS/cm)	TDS (mg/L)	COD_{Cr} (mg/L)	BOD_5 (mg/L)	pH
8,050	2,550	1.19×10^5	65,130	2.23

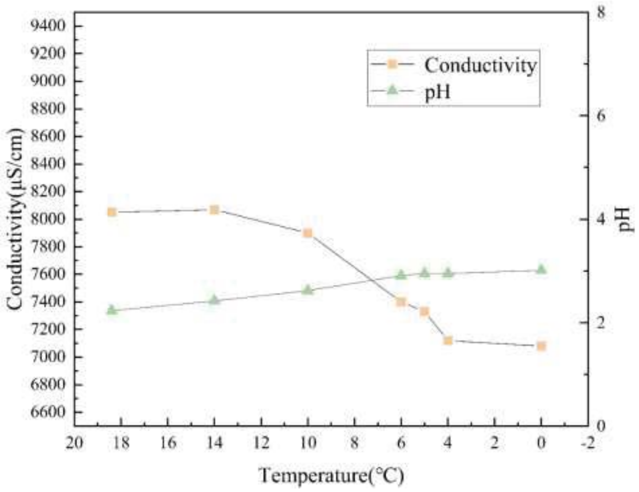

Fig. 2. Effect of temperature on conductivity and pH by cooling.

COD_{Cr}, BOD_5 and pH of treated wastewater were shown as in the Table 4. The conductivity of the UPR wastewater decreased from 8,050 to 7,080 μS/cm that decreased by 12%. The TDS of the wastewater decreased from 2,550 to 2,043 mg/L that decreased by 19.9%. The pH of the wastewater increased from 2.23 to 3.23. And the COD_{Cr}, BOD_5 only changed slightly. Therefore, it can be concluded that the treatment of cooling crystallization has no obvious effect on the UPR wastewater.

According to Fig. 3, the particles in the wastewater were analyzed by stokes' law model [28]. The dissolved solids could be modeled by homogeneous pellets with same weight and size, and the process of a pellet precipitated in a stationary viscous fluid environment. During the cooling process, when the particles in the wastewater were moved downward by gravity F_g, they were subjected to a viscous force direction, which was opposite to the movement. The formula for this force is [29]:

$$F_d = 6\pi\eta Rv \tag{2}$$

where F_d is the viscous force of the particles in the liquid phase; η is the viscosity of the liquid phase; R is the radius of the particle (which equivalent to a spherical object of same volume); v is the velocity of the particle relative to the liquid phase. The viscosity η of the liquid phase increases when the temperature is lowered, and the viscous force F_d of the particles increases. Therefore, even if the temperature promotes the precipitation of the dissolved solids in the wastewater, only a few dissolved solid particles will form flocs and suspend in the solution under the influence of the viscous force F_d.

Table 4
Parameters of UPR wastewater by cooling

Conductivity (μS/cm)	TDS (mg/L)	COD_{Cr} (mg/L)	BOD_5 (mg/L)	pH
7,080	2,043	1.19×10^5	63,300	3.23

3.3. Treatment by distillation-cooling

According to Fig. 4a, the UPR wastewater treated by distillation was sealed and cooled to normal temperature. After kept for a while at normal temperature, a small amount of white floc is floated in the UPR wastewater. As seen in Fig. 4b, a large number of white flocs appeared at 0°C compared with those in other experiments, and the odor of the UPR wastewater became significantly smaller. As shown in Fig. 4c, there was still white precipitation in the UPR wastewater after 12 h. As shown in Fig. 5, when the temperature was above 10°C, the conductivity of the UPR wastewater did not change significantly. When the temperature decreased to 5°C, the conductivity was decreased by 710 μS/cm, which had no significant difference compared with that in the 3.2 section where the temperature decreased to 5°C. But the conductivity decreased significantly from 5°C to 0°C that decreased by 1,200 μS/cm. And the changing trend of the pH was a significant rise when the temperature changes from 5°C to 0°C. According to Table 5, after distillation-cooling, the conductivity of the UPR wastewater decreased from 9,260 to 6,980 μS/cm that decreased by 24.6%. The TDS was reduced from 2,550 to 1,060.88 mg/L that decreased by 58.4%. The COD_{Cr} was reduced from 1.19×10^5 to 9.23×10^4 mg/L that decreased by 22.4%. The BOD_5 was reduced from 65,130 to 45,000 mg/L that decreased by 30.9%. The pH was increased from 2.23 to 4.03. In the end of the experiment, the flocs were filtered and weighed. The weight of flocs was 0.35 g, and the concentration of flocs in the wastewater was 875 mg/L.

According to Table 2, the main components of the UPR wastewater were water, maleic anhydride and 1,2-propanediol, which accounted for 27.42%, 24.42% and 25.07%, respectively. Other components in the UPR wastewater were too small, and there were few irritating substances in them, so ignore. The mixture of 1,2-propanediol and water was azeotrope. And there were many white floc precipitates at the bottom of the initial wastewater, so the UPR wastewater sample provided by the factory was supersaturated. In the first step of distillation, water and 1,2-propanediol in the wastewater were heated to azeotrope. The solvent

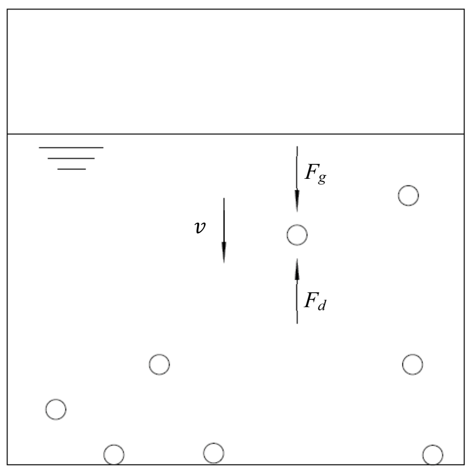

Fig. 3. Particle force diagram in liquid phase.

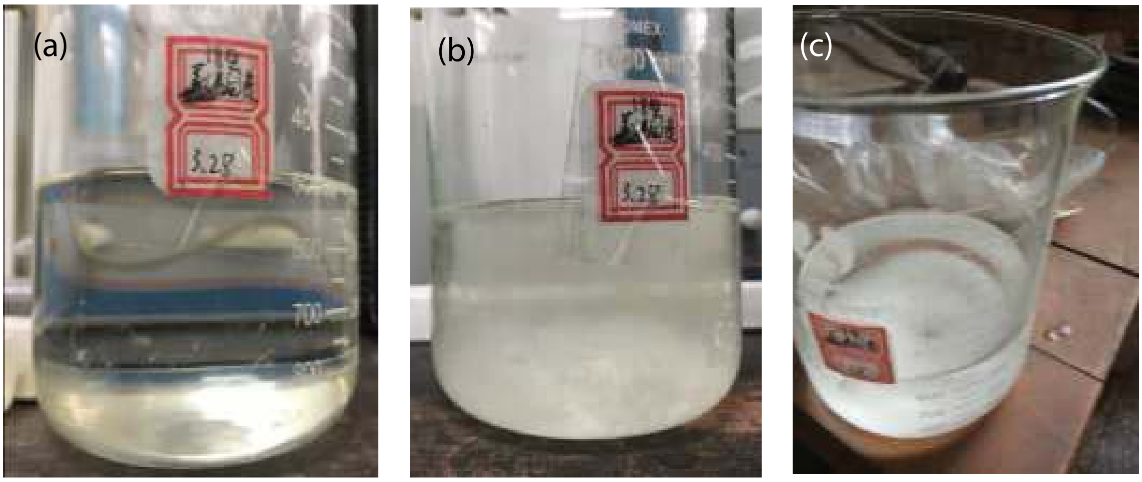

Fig. 4. (a) Wastewater by distillation, (b) Wastewater by distillation-cooling and (c) Wastewater treatment by distillation-cooling after 12 h.

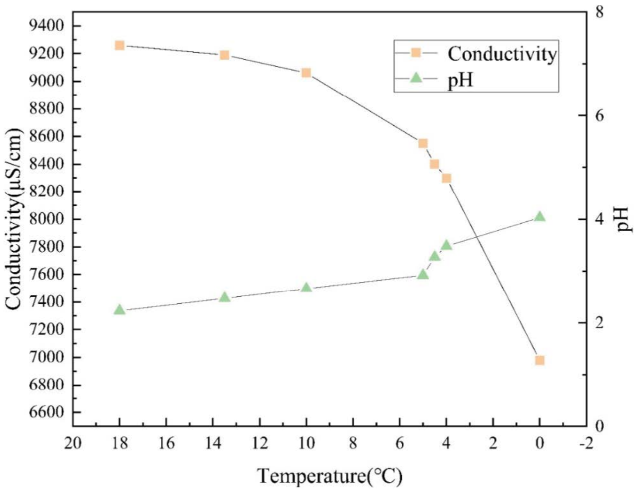

Fig. 5. Effect of temperature on conductivity and pH by distillation-cooling.

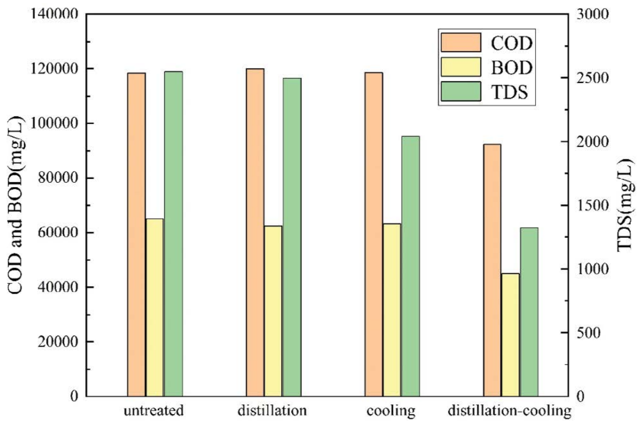

Fig. 6. Comparison histogram of TDS, COD_{Cr} and BOD_5.

Table 5
Parameters of UPR wastewater by distillation-cooling

Conductivity (μS/cm)	TDS (mg/L)	COD_{Cr} (mg/L)	BOD_5 (mg/L)	pH
6,980	1,060.88	9.23×10^4	45,000	4.03

of the maleic anhydride, which was water and 1,2-propanediol, was reduced due to the distillation. According to the experiment of Jiang [27], the solubility of maleic anhydride in the aqueous phase decreased with the decrease of temperature. So maleic anhydride precipitated in a large amount at around 0°C, which was also consistent with the phenomenon that large amount of white floc precipitates appeared at 0°C.

There was no obvious experiment phenomenon in the first group of distillation experiments, and the conductivity increased by 15%. The TDS, COD_{Cr}, BOD_5 and pH almost did not change. Although there was a small amount of floc in the second group of cooling experiments, the conductivity of the wastewater only decreased by 12%. And the TDS, COD_{Cr}, BOD_5 and pH almost did not change. Therefore, single distillation or cooling is not suitable for treating this UPR wastewater. But in the third group of distillation-cooling experiments, there was a large number of white flocs, which did not appear in other groups of experiments after treated. The flocs appeared in the UPR wastewater when distilled for 5 min and cooled to 0°C. According to Figs. 5 and 6, conductivity, TDS, COD_{Cr}, BOD_5 were reduced 24.6%, 58.4%, 22.4%, 30.9% and 4.03, respectively. And the pH increased from to 4.03. Therefore, compared with the other two processes, the reduction of conductivity, TDS, COD_{Cr} and BOD_5 in UPR wastewater after distillation-cooling pretreatment was more obvious. The organic and inorganic matter in the UPR wastewater could be removed efficiently. The increase of pH indicated that the high acidity of the UPR wastewater was improved [31], and the organic acid was removed. Therefore, its toxicity was lower with the improved biodegradability, which was helpful to the subsequent process [32].

4. Conclusions

In summary, the process of distillation-cooling for UPR wastewater was convenient, effective and nonpolluting. After distillation at 99.2°C for 5 min and then cooled to 0°C, the removal efficiency of conductivity, TDS, COD_{Cr}, and BOD_5 could reach about 24.6%, 58.4%, 22.4% and 30.9%, respectively. And the pH increased from 2.23 to 4.03. Namely, the organic acids, organic matter and inorganic matter in UPR wastewater were efficiently treated, which meant that the toxicity of UPR wastewater was reduced, and the biodegradability was improved. Moreover, the maleic anhydride, one of the raw materials of UPR, was separated by distillation-cooling. The experiment results indicate that the distillation-cooling method is more adaptive than single distillation or cooling for the pretreatment of UPR wastewater.

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