Recovery of copper and water from copper-electroplating wastewater by the combination process of electrolysis and electrodialysis

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ABSTRACT

In this paper, a laboratory-scale process which combined electrolysis (EL) and electrodialysis (ED) was developed to treat copper-containing wastewater. The feasibility of such process for copper recovery as well as water reuse was determined. Effects of three operating parameters, voltage, initial Cu^{2+} concentration and water flux on the recovery of copper and water were investigated and optimized. The results showed that about 82% of copper could be recovered from high concentration wastewater (HCW, >400 mg/L) by EL, at the optimal conditions of voltage 2.5 V/cm and water flux 4 L/h; while 50% of diluted water could be recycled from low concentration wastewater (LCW, <200 mg/L) by ED, at the optimal conditions of voltage 40 V and water flux 4 L/h. However, because of the limitation of energy consumption (EC), LCW for EL and HCW for ED could not be treated effectively, and the effluent water of EL and concentrated water of ED should be further treated before discharged. Therefore, the combination process of EL and ED was developed to realize the recovery of copper and water simultaneously from both HCW and LCW. The results of the EL–ED process showed that almost 99.5% of copper and 100% of water could be recovered, with the energy consumption of EL $\approx$3 kW h/kg and ED $\approx$2 kW h/m^3. According to SEM and EDX analysis, the purity of recovered copper was as high as 97.9%.

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1. Introduction

Copper-electroplating wastewater is generated from electroplating and acid–alkali washing process [1]. The maximum level of copper that discharged into the environment is 2 mg/L which was established by the Environmental Protection Agency of China [2]. Copper is toxic to animals, algae and plants. Besides the environmental impact, due to the inefficient use of heavy metals resources and the discharge of wastewater, the pollution control legislation is regulating and controlling electroplating operations by increasing the economic penalties. In response to all these, electroplating industries try to develop various separation processes to reuse materials from the rinse water and thus realize the reuse of both the heavy metals and the water.

Chemical precipitation is the most common method for the removal of dissolved metals from wastewater. However, it produces precipitation sludge which leads to secondary pollution [3]. The optional processes include reverse osmosis (RO), electrodialysis (ED), ion-exchange (IE) and electrolysis (EL). ED is one of the most recent technologies that applied to the recovery of plating chemicals from rinse water [4]. Advantages of ED are that the low concentration of heavy metals can be concentrated and the other part of effluent water can be diluted for reuse. However, it does not fit for high concentration due to the restriction of membrane pollution. In addition, the EL process differs from ED with the advantages of the recovery of heavy metals and the feasibility for high concentration of heavy metals wastewater. Over thirty kinds heavy metals can be recovered from aqueous solutions by EL, including many precious metals and heavy metals [5]. There are many researches about copper recovery by EL [6–9]. Meanwhile the ED for acidic aqueous solution that contains copper ions has been studied by several workers [10–12]. However, no study has been conducted for copper-electroplating wastewater by such combined techniques.

With an aim at copper recovery and water reuse from copper-electroplating wastewater, this paper presented a combination process of EL and ED. By adopting such a strategy, the wastewater is converted into two valuable products: copper and water. Experimental variables include: voltage, water flux and initial Cu^{2+} concentration. Additionally, properties of recovered copper and water have also been analysed.

2. Materials and methods

2.1. Materials and apparatuses

Copper-containing wastewater was prepared by diluting copper sulfate ($CuSO_4 \cdot 5H_2O$) with tap water (the conductivity of tap water $\approx$300 μs/cm). Sulfuric acid (H_2SO_4) was used for pH adjustment

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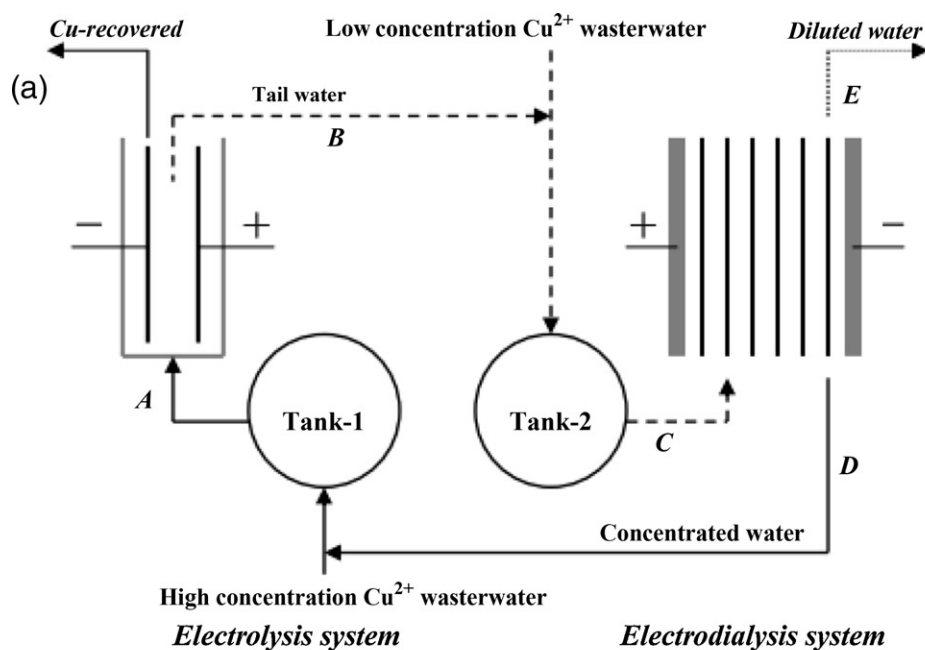*E-mail address:* cspeng@ouc.edu.cn (C. Peng).

Fig. 1. Scheme of the EL–ED combination process: (a) basic scheme and (b) real photo.

in order to approach the actual wastewater. Copper concentration was monitored by atomic absorption spectrometry (Solaar M6, Thermo). Other parameters of the water samples were determined by a pH meter (PH-3D) and a conductivity meter (DDS-307). The morphology and elements distribution of recovered copper were analysed by SEM, EDX (XL-30, Philips) and Stereo Microscope (SMZ660, Nikon). All chemicals used here were analytical grade.

Before all experiments, we analysed 36 samples of copper-containing wastewater from 12 electroplating enterprises around our city. The results showed that the concentration of Cu^{2+} ranged largely from less 100 mg/L to almost 1000 mg/L, while pH ranged gently from 2.5 to 5.0. So, in our experiments, wastewater prepared for EL had no pH adjustment, because copper sulfate is a little acidic; while wastewater prepared for ED adjusted pH to 2.5 ± 0.3, because the effluent of EL kept the pH in this range, no matter the pH of feed

was 2.5 or 5.0. When EL and ED working together, the feed pH of both EL and ED could be stabilized at 2–3.

As shown in Fig. 1, the combination process of EL and ED was made from two independent systems: electrolysis system and electrodialysis system. If the effluent water of EL was put into Tank-2 and concentrated water of ED was put into Tank-1, the two independent systems were merged into an EL–ED combination process. The main parts of EL system included an electrolytic cell, a DC power, a pump and a tank. The electrolytic cell (3.7 L) contained four anode plates and three cathode plates with an area of 294 cm². The anode plates made from titanium grid covered with iridium and ruthenium oxides, and the cathode plates made from AISI 304 stainless steel. During the first stage of EL experiments, all electrodes were smooth plates without any holes, but all electrodes were punched dozens of holes on plates during the second stage of

EL experiments which could increase current efficiency greatly. An air pump was installed at the bottom of the electrolytic cell in order to stir. The main parts of ED system included a membrane stack, a DC power, a pump and a tank. The ion-exchange membranes used in ED was characterized as low permeability and high acid tolerability, with an area of 350 cm² and 200 pairs. Two power supplies (PS-303D) were used to provide constant DC power for EL and ED, respectively.

2.2. Methods and analysis

As shown in Fig. 1, the EL–ED combination process was designed for treating copper-electroplating wastewater with different copper concentration. If high concentration copper wastewater (HCW), for instance, Cu^{2+} concentration was >300 mg/L. The HCW was firstly fed into Tank-1, and then pumped into electrolytic cell of EL for copper recovery, the effluent water from electrolytic cell was sent to Tank-2 for further treatment by ED. Since most of copper was recovered in EL system, the Cu^{2+} concentration in Tank-2 was usually <300 mg/L, so this effluent water was called low concentration copper wastewater (LCW). The LCW was pumped into membrane stack of ED for copper and water separation, diluted water from ED was reused and concentrated water was sent back to Tank-1 for the next circulation. If LCW, the wastewater was firstly fed into Tank-2 for ED treatment, and then the concentrated water was sent to Tank-1 for EL treatment. If LCW and HCW should be treated simultaneously, LCW was fed into Tank-2 and HCW was fed into Tank-1, respectively. Five Sampling points marked A, B, C, D and E were located at in and out of EL, in and out (including concentrated and diluted water) of ED. Conductivity, pH, Cu^{2+} concentration and water flux of the samples from this five points were analysed. The voltage and current of EL and ED were respectively recorded and integrated automatically by current meters (UT60E) connected to a computer.

Copper recovery rate (R_C), water recycle rate (R_W), energy consumption of EL (EC_1) and energy consumption of ED (EC_2) were calculated as Eq. (1)–(4):

$$R_C = \frac{Q_A \cdot (C_A - C_B)}{Q_A \cdot C_A} \tag{1}$$

$$R_W = \frac{Q_E}{Q_L + Q_H} \tag{2}$$

$$EC_1 = \frac{U \cdot I}{Q_A \cdot (C_A - C_B)} \tag{3}$$

$$EC_2 = \frac{U \cdot I}{1000 \cdot Q_E} \tag{4}$$

where, R_C: copper recovery rate, %; R_W: water recycle rate, %; EC_1: energy consumption of EL, kW h/kg; EC_2: energy consumption of ED, kW h/m³; U: voltage, V; I: current, mA; Q: water flux, L/h; C: concentration of copper, mg/L.

3. Results and discussion

3.1. Electrolysis (EL)

3.1.1. Cell voltage

Cell voltage is one of the most important operational parameters, which can affect copper recovery and energy consumption strongly. Cell voltage is constituted of three parts: decomposition voltage, excess voltage and Ohmic voltage drops. Decomposition voltage provides the standard Gibbs energy for the overall reaction, excess voltage due to polarization of the electrode interfaces, and Ohmic voltage drops due to the resistance of various zones of the cell. High voltage may lead to high copper recovery with

high energy consumption, while low voltage may lead to low copper recovery and low energy consumption. So, an optimum voltage must be a balance voltage between high copper recovery and low energy consumption.

In this experiment, wastewater was pumped into the electrolytic cell from Tank-1 for copper recovery. The initial Cu^{2+} concentration of raw water at sampling point-A was kept at a stable range of 450 ± 20 mg/L for this experiment, and the inlet water flux was 4 L/h. Table 1 lists the main results of the experiment.

It can be seen from Table 1 that the Cu^{2+} concentration of effluent water at sampling point-B gradually decreased from 134.92 mg/L at 1.5 V/cm to 48.62 mg/L at 2.5 V/cm, and then became 21.77 mg/L at 4.0 V/cm. While the copper recovery gradually increased from 68.80% at 1.5 V/cm to 89.34% at 2.5 V/cm, and then became 95.36% at 4.0 V/cm. It seemed that higher voltage made more effective copper recovery, because higher voltage forced the ions to electro-migrate and electro-deposit more quickly. However, energy consumption (EC) gradually increased from 2.57 kW h/kg at 1.5 V/cm to 7.71 kW h/kg at 2.5 V/cm, but sharply increased from 7.71 kW h/kg at 2.5 V/cm to 50.60 kW h/kg at 4.0 V/cm. Due to high current density resulted from high voltage, the concentration polarization happened as the severe shortage of Cu^{2+} ions near the electrode plate, which lead to the H_2 gas generation. As shown in Fig. 2, there was no appearance of H_2 bubble at 1.5 V/cm, but a few small H_2 bubbles on the electroplates at 2.5 V, and many big H_2 bubbles on the plates and in solution at 4.0 V. It was the generation of H_2 that might decrease the current efficiency and increase the EC. Taking into account the balance between copper recovery and EC, 2.5 V/cm was chosen as a suitable voltage in the following experiments.

3.1.2. Initial Cu^{2+} concentration of wastewater

Since the Cu^{2+} concentration of real wastewater from different electroplating workshops vary greatly from less than 100 mg/L to more than 1000 mg/L, five kinds of wastewater with concentrations of 150 mg/L, 350 mg/L, 450 mg/L, 800 mg/L and 1050 mg/L were used for this experiment. The voltage and water flux of this experiment were 2.5 V/cm and 4 L/h, respectively.

The main results of the experiment are listed in Table 2. It can be seen that the Cu^{2+} concentration of effluent water at sampling point-B gradually increased from 14.81 mg/L to 96.23 mg/L with the increasing of initial Cu^{2+} concentration from 150 mg/L to 1050 mg/L. While the copper recovery maintained at a stable range from 90.13% to 93.15% in this experiment. EC decreased from 11.08 kW h/kg to 5.29 kW h/kg with the increasing of initial Cu^{2+} concentration from 150 mg/L to 1050 mg/L. Considering the EC of 11.08 kW h/kg was still too high for real application, the Cu^{2+} concentration of raw wastewater should be >350 mg/L.

3.1.3. Water flux

Water flux can directly decide the capacity of wastewater treatment, so it is a very important parameter for real application. In this part, effect of water flux on wastewater treatment by EL was investigated, under the conditions of 500 mg/L initial Cu^{2+} concentration and 2.5 V/cm voltage. The results are listed in Table 3.

It can be seen from Table 3 that the Cu^{2+} concentration of effluent water at sampling point-B increased with the increasing of water flux. Maybe the bigger the water flux, the shorter time the Cu^{2+} ions stayed in the electrolytic cell, so some Cu^{2+} ions were flowed out before deposited. The copper recovery was 93.02% when water flux was 1 L/h, but its EC was 5.71 kW h/kg due to its long electrolytic time. Compared with it, the EC was about 3 kW h/kg when ran at high water flux of 4–8 L/h. For high water flux, there were huge amounts of Cu^{2+} ions which had ready for deposition in the electrolytic cell. Consequently, the generation of hydrogen on plates was restrained and resulted in a high current efficiency. Taking cop-

Table 1
Effect of cell voltage on copper recovery and energy consumption.

Voltage (V/cm)	Cu²⁺ concentration (mg/L)		Copper recovery (%)	EC (kW h/kg)
	Point-A	Point-B		
1.5	432.47	134.92	68.80	2.57
2.0	446.95	90.52	79.75	4.61
2.5	455.97	48.62	89.34	7.71
3.0	463.19	40.41	91.28	19.45
4.0	469.44	21.77	95.36	50.60
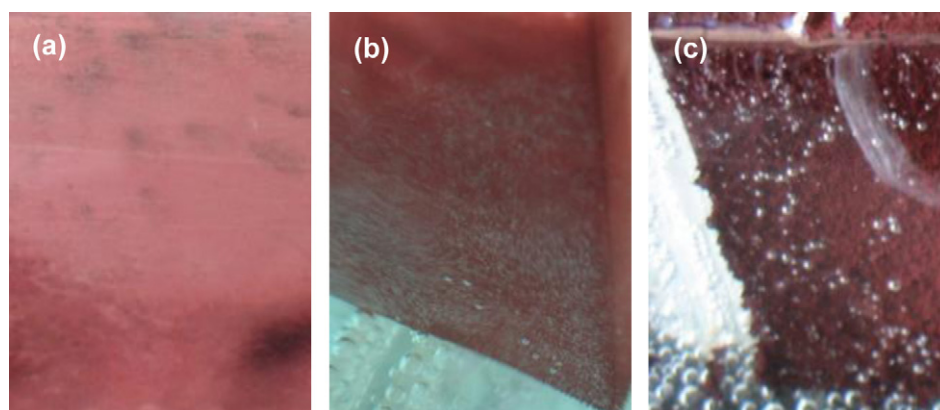
Fig. 2. Photos of hydrogen generation at different cell voltage: (a) 1.5 V/cm, (b) 2.5 V/cm, (c) 4.0 V/cm.

Table 2
Effect of initial Cu²⁺ concentration on copper recovery and energy consumption.

Voltage (V/cm)	Cu²⁺ concentration (mg/L)		Copper recovery (%)	EC (kW h/kg)
	Point-A	Point-B		
2.5	150	14.81	90.13	11.08
2.5	350	23.97	93.15	9.01
2.5	450	34.89	92.25	7.35
2.5	800	59.10	92.61	5.71
2.5	1050	96.23	90.86	5.29

Table 3
Effect of water flux on copper recovery and energy consumption.

| Water flux (L/h)	Cu²⁺ concentration (mg/L)		Copper recovery (%)	EC (kW h/kg)
	Point-A	Point-B		
1	500	34.89	93.02	5.71
2	500	43.29	91.34	3.77
4	500	89.65	82.65	3.32
6	500	170.98	55.13	3.25
8	500	179.25	46.33	3.19

per recovery, EC and treatment capacity into consideration, 4 L/h was a suitable water flux.

3.2. Electrodialysis (ED)

3.2.1. Effect of cell voltage

Cell voltage is the driving force of the ED process due to it determines the migration speed of Cu²⁺ ions across the membranes. In this experiment, a simulated copper-containing wastewater (200 mg/L) was treated at different cell voltages as following: 20 V, 30 V, 40 V, 50 V, 60 V. Both the diluted water and concentrated water flux were 4 L/h.

It can be seen from Fig. 3 that the Cu²⁺ concentration of concentrated water increased gradually from 337.8 mg/L to 450 mg/L with the cell voltage increasing from 20 V to 60 V, while the Cu²⁺ concentration of diluted water decreased sharply from 2.90 mg/L at 20 V to 0.83 mg/L at 40 V and slightly from 0.83 mg/L at 40 V to
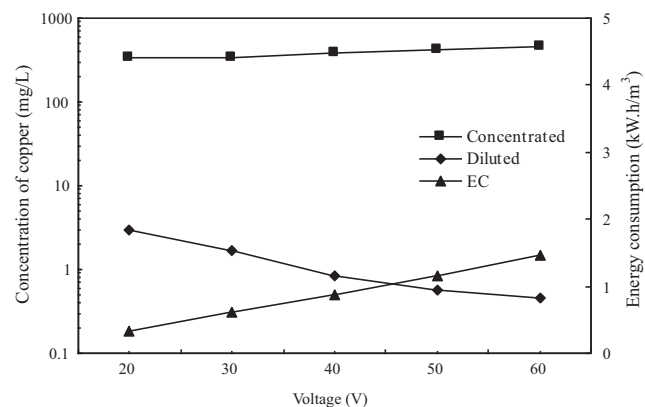
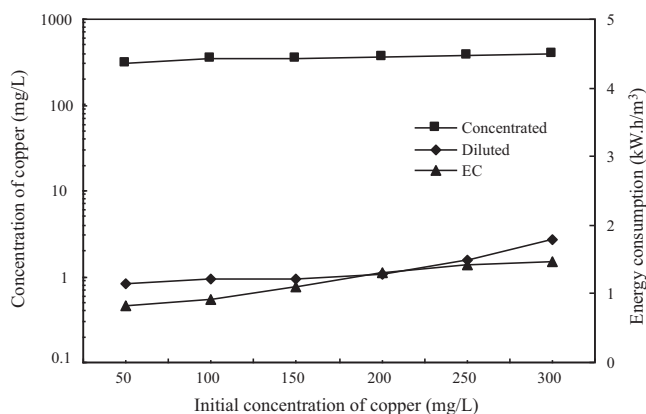**Fig. 3.** Effect of voltage on Cu²⁺ ions separation from wastewater and energy consumption.

Fig. 4. Effect of initial concentration on Cu^{2+} ions separation from wastewater and energy consumption.

Fig. 5. Effect of water flux on Cu^{2+} ions separation from wastewater and energy consumption.

0.46 mg/L at 60 V. At the same time, the conductivity of diluted water decreased from 452.5 μs/cm at 20 V to 132.0 μs/cm at 40 V rapidly but slightly from 132.0 μs/cm at 40 V to 81.6 μs/cm at 60 V. It seems that higher voltage was better for water quality, but the EC increased linearly from 0.33 kW h/m³ to 1.47 kW h/m³ with the voltage increasing from 20 V to 60 V. So, taking water quality and energy consumption into consideration, 40 V would be the optimal voltage for ED process, since the Cu^{2+} concentration of 0.8 mg/L and conductivity of 132.0 μs/cm at 40 V were good enough to meet the water reuse requirement. Under the condition of 40 V, the EC was only 0.87 kW h/m³.

3.2.2. Effect of initial Cu^{2+} concentration

Six kinds of wastewater with the initial Cu^{2+} concentrations ranging from 50 mg/L to 300 mg/L were used for this experiment, under the conditions of operating voltage at 40 V and the water flux of diluted and concentrated water at 4 L/h and 4 L/h, respectively. The results are presented in Fig. 4.

It can be seen from Fig. 4 that the Cu^{2+} concentration of concentrated water increased slightly from 312 mg/L to 399 mg/L with the initial Cu^{2+} concentration increasing from 50 mg/L to 300 mg/L, while the Cu^{2+} concentration of diluted water increased also slightly from 0.82 mg/L to 1.07 mg/L at the range from 50 mg/L to 200 mg/L of the initial Cu^{2+} concentration, but increased quickly from 1.07 mg/L to 2.70 mg/L with the Cu^{2+} concentration increasing from 200 mg/L to 300 mg/L. In addition, the rising curve in Fig. 4 demonstrated that with the increasing of initial Cu^{2+} concentration from 50 mg/L to 200 mg/L, and then to 300 mg/L, the energy consumption also increased slowly from 0.83 kW h/m³ to 1.31 kW h/m³, and then to 1.46 kW h/m³. From the three curves of Fig. 4, we could find that the Cu^{2+} concentration of diluted water limited the concentration range of Cu^{2+} in feed water. In order to make sure the Cu^{2+} concentration of diluted water below 1 mg/L, the initial Cu^{2+} concentration must be <200 mg/L for ED process.

3.2.3. Effect of water flux

In this experiment, the water flux of diluted water and concentrated water were kept the same at six levels as 1 L/h, 2 L/h, 4 L/h, 6 L/h, 8 L/h and 10 L/h. The cell voltage and initial Cu^{2+} concentration were fixed at 40 V and 200 mg/L, respectively. The results are presented in Fig. 5.

It can be seen from Fig. 5 that the Cu^{2+} concentration of concentrated water kept at a stable range of 550 ± 20 mg/L. But the curve of diluted water shows that the optimal range of water flux would be 4–6 L/h, in which the Cu^{2+} concentration of diluted water kept the minimum level at about 0.35 mg/L. As for EC, it declined gradually from 3.50 kW h/m³ to 0.85 kW h/m³ with the increasing
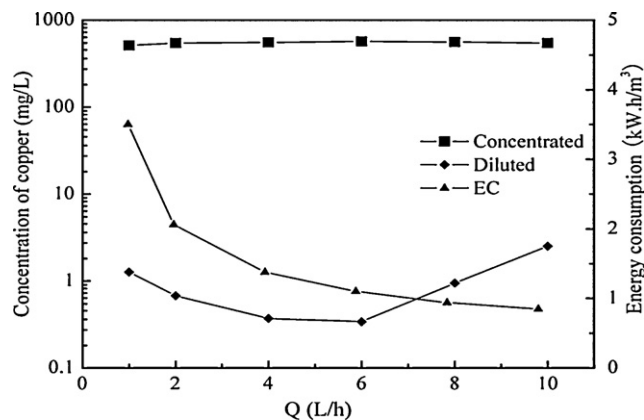
water flux. According to the experiment results, if the ED process was operated at the water flux range of 4–6 L/h, the Cu^{2+} concentration of diluted water would be under 0.5 mg/L, the conductivity of diluted water would be about 100 μs/cm, and the EC would be less than 1.5 kW h/m³ of diluted water. All the results demonstrated that the diluted water of ED could be reused.

3.3. Combination of electrolysis and electrodialysis

According to the experiment results mentioned above, the EL process can recover copper from wastewater but only effective for HCW, while the ED process can recover water from wastewater but only effective for LCW. So, neither EL nor ED can treat the real copper-electroplating wastewater alone, due to the Cu^{2+} concentration of real wastewater from electroplating workshops vary greatly from less than 100 mg/L to more than 1000 mg/L. Thus, the combination process of EL and ED was developed, as shown in Fig. 1, to investigate the feasibility of copper and water recovery for all kinds of copper wastewater, no matter the concentration was high or low. If >350 mg/L, the wastewater must be fed into EL first, and then for all EL–ED process, energy consumption is lowest; if <200 mg/L, the wastewater must be fed into ED first, and then for all EL–ED process, energy consumption is lowest; if 200–350 mg/L, the wastewater is fed into ED first, or EL first, or ED and EL simultaneously, energy consumption is almost the same.

The combination process was investigated in three situations: only HCW, only LCW, HCW and LCW simultaneously. The operating conditions were kept the same of the three situations: 2.5 V/cm cell voltage and 4 L/h water flux for EL; 40 V membrane voltage, 4 L/h diluted water flux and 4 L/h concentrated water flux for ED. The results of the three situations are listed in Table 4, Table 5 and Table 6, respectively.

It can be seen from the three tables that the combination process could be operated stably in all three situations. In all situations, the conductivity of diluted water at sampling point-E was less than 170 μs/cm and Cu^{2+} concentration was less than 1.8 mg/L, which meant the diluted water was suitable for reuse. All water flowed into the combination system could flow out from diluted water only, and all diluted water was suitable for reuse. Therefore, water recovery of the combination system could be 100%. As for copper, almost 99.5% could be recovered from the combination system, except for less than 0.5% of copper lost from diluted water. Moreover, the energy consumption of electrolysis (EC_1) was less than 3.0 kW h/kg for copper recovery, and the energy consumption of electrodialysis (EC_2) was less than 2.0 kW h/m³ for water recovery.

The recovered copper, as shown in Fig. 6, was analysed in morphology and element distribution. A real photo of the recovery

Table 4
Operating results of the EL–ED combination processes for high Cu^{2+} concentration wastewater.

		Operating time (min)								
		0	30	60	90	120	150	180	210	240
Concentration of Cu^{2+} (mg/L)	Point-A	504.31	438.23	428.42	413.75	439.28	469.30	421.96	439.57	423.76
	Point-B	–	174.88	135.82	126.89	118.32	111.56	109.03	112.34	108.29
	Point-C	–	270.75	273.98	269.88	219.28	197.10	199.82	203.62	191.85
	Point-D	–	296.61	336.71	445.26	425.17	464.11	498.08	524.66	561.11
	Point-E	–	0.21	0.27	0.21	0.28	0.48	0.33	0.41	0.38
Conductivity (μs/cm)	Point-E	–	16.0	27.0	64.0	57.9	53.8	57.2	54.9	69.5

Table 5
Operating results of the EL–ED combination processes for low Cu^{2+} concentration wastewater.

		Operating time (min)								
		0	30	60	90	120	150	180	210	240
Concentration of Cu^{2+} (mg/L)	Point-A	–	483.77	497.65	512.36	451.36	458.06	460.11	552.00	499.28
	Point-B		215.70	170.03	158.6	157.67	185.06	152.72	156.75	154.22
	Point-C	281.16	284.06	249.36	267.75	282.78	269.67	261.11	250.11	251.24
	Point-D	–	645.68	598.11	622.59	637.7	627.43	625.45	670.04	678.18
	Point-E	–	0.89	0.99	1.31	1.09	1.54	1.76	1.27	1.25
Conductivity (μs/cm)	Point-E	–	124.7	144.3	124.7	136	147.4	142.2	167.9	144.5

Table 6
Operating results of the EL–ED combination processes for high and low Cu^{2+} concentration wastewater simultaneously.

		Operating time (min)								
		0	30	60	90	120	150	180	210	240
Concentration of Cu^{2+} (mg/L)	Point-A	446.51	470.37	462.32	450.77	449.35	425.60	509.32	495.29	479.59
	Point-B	–	385.66	307.83	265.75	228.21	213.32	232.32	217.76	210.48
	Point-C	165.33	224.87	213.79	223.04	218.93	229.77	224.44	232.52	222.73
	Point-D	–	379.28	450.66	410.44	460.22	475.34	504.67	517.49	567.53
	Point-E	–	0.46	0.56	0.64	0.72	0.51	0.48	0.76	0.72
Conductivity (μs/cm)	Point-E	–	93.4	87.7	92.2	115.6	93.2	95.5	96.7	95.6
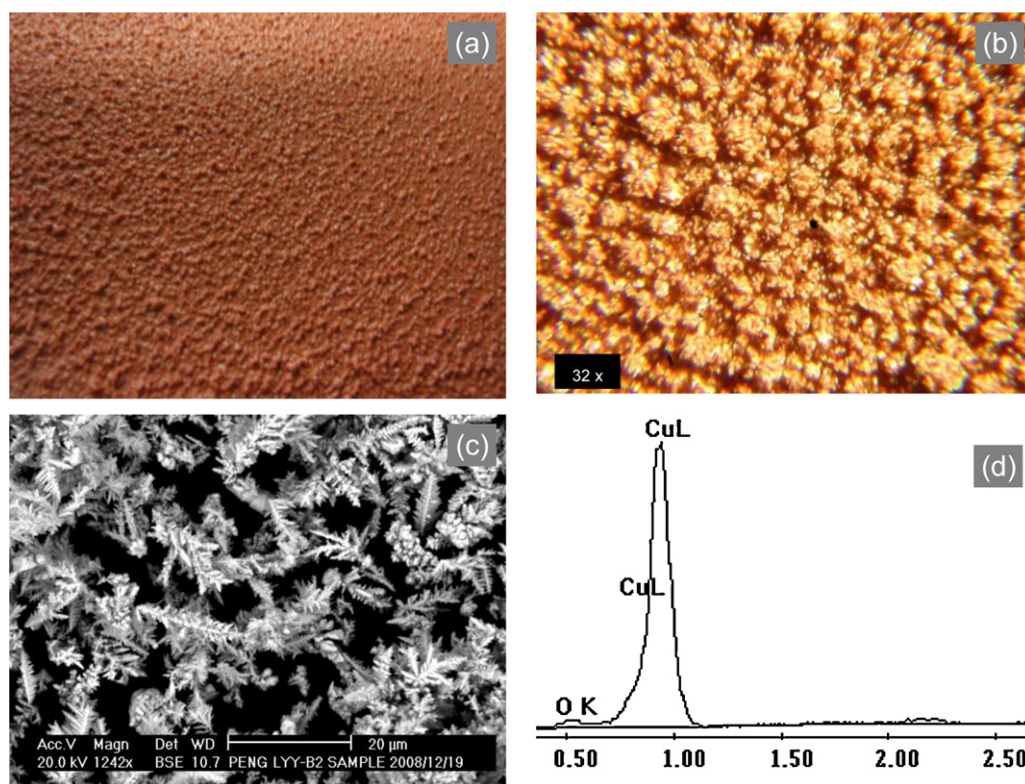
Fig. 6. Morphology and element distribution analysis of the recovered copper: (a) optical camera, (b) Stereo Microscope, (c) SEM and (d) EDX.

copper by an optical camera is shown in Fig. 6(a), a 32 times magnification picture by Stereo Microscope is shown in Fig. 6(b), and a SEM image is shown in Fig. 6(c), while the element distribution by EDX is shown in Fig. 6(d). According to the EDX determination, the purity of the recovered copper was 97.9%, while 2.1% of oxygen might be from the oxidation of sample in the air.

4. Conclusions

A series of experiments were done to investigate the feasibility of copper-electroplating wastewater treatment by EL process, ED process and EL–ED combination process in this paper. On the basis of the experiments results, some conclusions could be deduced as following:

(1) EL process could effectively recover copper from high concentration wastewater (HCW, >400 mg/L). About 82% of copper could be recovered from the wastewater at the conditions of 500 mg/L Cu^{2+} concentration, 2.5 V/cm voltage and 4 L/h water flux.

(2) ED process could effectively separate Cu^{2+} ions from low concentration wastewater (LCW, <200 mg/L). At the conditions of 200 mg/L Cu^{2+} concentration, 40 V voltage, 4 L/h diluted water flux and 4 L/h concentrated water flux, 50% of water could be reused with its conductivity <150 μs/cm and Cu^{2+} concentration <1.0 mg/L.

(3) Due to the limitation of energy consumption (EC), LCW for EL and HCW for ED could not be treated effectively, so the effluent water of EL and concentrated water of ED should be further treated before discharged. Therefore, neither EL nor ED can treat the real copper-electroplating wastewater alone, since the Cu^{2+} concentration of real wastewater from electroplating workshops vary greatly from less than 100 mg/L to more than 1000 mg/L.

(4) The EL–ED combination process could realize the recovery of copper and water simultaneously from both HCW and LCW. Almost 99.5% of copper and 100% of water could be recovered, with the energy consumption of EL <3.0 kW h/kg and ED <2.0 kW h/m^3.

(5) According to SEM and EDX analysis, the purity of recovered copper was as high as 97.9%.

Acknowledgements

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