Simultaneous Desalination and Removal of Recalcitrant Organics from Reverse Osmosis Leachate Concentrate by Electrochemical Oxidation

Chaoqun Yan,§ Yijuan Tian,§ Zhiliang Cheng,§ Zejun Wei, Xuan Zhang, and Xuejun Quan*

1. INTRODUCTION

Incineration and landfill are the mainstream treatments of municipal solid waste (MSW) in China.1 However, a large amount of leachate that accounts for 10–25% mass weight of MSW would be produced during its storage stage because of the inadequate MSW classification in China.2 The MSW leachate is a kind of dark gray, foul-smelling organic wastewater, which contains high concentrations of organic matter, ammonia, dissolved solids, and heavy metals.3–6 It is a considerable threat to the ecological environment and human beings.5,6 Reverse osmosis (RO) technology is often used to treat the biologically treated MSW leachate due to its advantages of excellent effluent quality, smaller footprint, automatic control, and low cost.8 However, 20% and even up to 50% volume of the treated wastewater is concentrated as RO leachate concentrate (ROLC) because of the high concentration of recalcitrant organic compounds in the RO concentrate. As a result, the RO leachate concentrate (ROLC) is extremely hard to be treated. In practical wastewater treatment, recirculating spraying and combustion treatment are often used in landfills and MSW incineration plants in China, respectively. However, the above-mentioned treatments of ROLC are costly, have low efficiency, and exhibit adverse side effects. Especially for the landfill, recirculating spraying treatment is the temporary disposal of ROLC, causing the salt and recalcitrant organic compounds to accumulate continually. As a result, the whole landfill system faces a threat of collapse, and treatment technology for ROLC is urgently needed.

To develop a new efficient treatment technology of ROLC, researchers around the world have turned to advanced oxidation processes (AOPs) for a solution. The AOPs are characterized as producing strong oxidation species such as $\cdot{\text{OH}}/\cdot{\text{O}}_2^{{\text{•}}} /\cdot{\text{HO}}_2^{{\text{•}}}$ during the treatment process, exhibiting superiority in recalcitrant organic removal and biodegradability improvement.11 The AOPs commonly used in ROLC include electrochemical oxidation,12,13 Fenton-active persulfate-coupling oxidation,14,15 coagulation–flocculation Fenton method,16,17 coagulation–ozonation method,18,19 electro-Fenton oxidation,20–22 and photoelectro-Fenton oxidation.23 However, many kinds of research studies on AOPs are still at the laboratory stage because of the high treatment cost, complicated operation, large generation of sludge, and secondary pollution.

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Compared with the other AOPs, electrochemical oxidation is considered to be an eye-catching technology for the treatment of ROLC owing to no sludge generation, no addition of chemical oxidation reagent, convenient operation, versatility, and low cost.\(^\text{13,24-27}\) Furthermore, the high concentration of inorganic ions, particularly the chloride ions (\(\text{Cl}^+\)) in ROLC, can improve the conductivity of wastewater and benefit the formation of active chlorine species (\(\text{Cl}_2/\text{OCl}^-\)).\(^\text{26,28}\) As active chlorine has been proved to be a good intensifier to remove organic substances,\(^\text{28}\) leading to an anticipation of a highly efficient but low-energy-consuming treatment of ROLC by electrochemical oxidation. Last but not the least, desalination, which is vital for the treatment of ROLC, is also expected during the electrochemical treatment process because of the consumption of \(\text{Cl}^-\).

In this work, ROLC was electrochemically oxidized in a homemade multichannel flow reactor (MCFR) with an electrode gap of 5 mm using a RuO\(_2/\text{IrO}_2\)-coated titanium plate (RuO\(_2/\text{IrO}_2\)-Ti) as the anode and a 304 stainless steel plate as the cathode. The effects of current density (\(I_\text{A}\)), wastewater circulating velocity (\(U_\text{L}\)), and treatment capacity per electrode area (\(C_\text{E}\)) on the chemical oxygen demand (COD) removal efficiency (RE) were studied. Then, the average energy consumption (EC) of this electrochemical oxidation process was comprehensively evaluated and compared with other works. Besides, gas chromatography–mass spectrometry (GC–MS) was employed to determine the original and final treated products of ROLC. Finally, the precipitate generated in the treatment process was collected and analyzed, and a new mechanism of the simultaneous removal of COD and desalination by electrochemical oxidation was proposed correspondingly.

2. RESULTS AND DISCUSSION

2.1. Effects of Process Parameters on Organics Removal. 2.1.1. Effect of \(I_\text{A}\). The effect of \(I_\text{A}\) on the COD removal of ROLC by electrochemical oxidation was investigated and the results are shown in Figure 1. As can be seen in the figure, the increase of \(I_\text{A}\) from 4.39 to 10.96 mA/cm\(^2\) accelerated the COD elimination rate but no significant influence of \(I_\text{A}\) on COD removal was observed when \(I_\text{A}\) exceeded 13.16 mA/cm\(^2\). The anode of RuO\(_2/\text{IrO}_2\)-Ti plates in this work is the so-called chlorine evolution electrode. The mechanism of the electrochemical oxidation of the recalcitrant organic compounds in the ROLC can be described by the following eqs 1–2:\(^\text{29,30}\)

\[
\begin{align*}
2\text{Cl}^- & \rightarrow \text{Cl}_2 + 2e^- \\
\text{Cl}_2 + \text{H}_2\text{O} & \rightarrow \text{ClO}^- + 2\text{H}^+ + \text{Cl}^- \\
\text{active chlorine (Cl}_2/\text{ClO}^-\text{)} & \rightarrow \text{CO}_2 + \text{H}_2\text{O}
\end{align*}
\]

Higher current densities (4.39–10.96 mA/cm\(^2\)) promoted the generation of oxidants of active chlorine at the surface of the electrode, resulting in the rapid removal of COD,\(^\text{33}\) which is also confirmed by the detection of residual active chlorine as shown in Figure 1c. As illustrated in Figure 1c, a higher concentration of residual active chlorine was detected with a higher current density. However, further increasing \(I_\text{A}\) to 13.16 mA/cm\(^2\) did not significantly affect the removal rate of COD may due because the control steps of the electrochemical oxidation reaction depended on the mass transfer between the oxidants and organic pollutants.\(^\text{32,33}\) This phenomenon is also consistent with the concentration changes of residual active chlorine produced in the electrochemical oxidation process shown in Figure 1c. In the first 80 min of the electrochemical treatment, oxidant species of \(\text{Cl}_2/\text{OCl}^-\) reacted rapidly with the organsics due to the high content of organic compounds. As a result, the content of the residual active chlorine in the reaction system is particularly low, and the limiting factor of the treatment is the production of oxidants. However, when the treatment time exceeds 80 min, the residual active chlorine content in the system increased greatly with the enhancement of current density. Thus, at this stage of the treatment, the limiting factor of the electrochemical reaction changed into the mass-transfer process.

The decolorization of ROLC by the electrochemical oxidation process was also investigated, and the result is shown in Figure 2. It can be noted that the color of ROLC was nearly completely removed after 180 min treatment by the electrochemical process. This is mainly due to the production of active chlorine (reactions 1–2) that oxidizes the colored substances.

2.1.2. Effect of \(U_\text{L}\). The \(U_\text{L}\) affects the residence time, solubility of chlorine, mix and mass transfer of organic compounds, inorganic ions, and oxidant (\(\text{Cl}_2/\text{OCl}^-\)) in the electrochemical treatment process.\(^\text{33,34}\) The effect of \(U_\text{L}\) on the
The electrochemical oxidation efficiency of the recalcitrant organic pollutants was studied and presented in Figure 3. As illustrated in Figure 3, the efficiency of COD removal decreased continuously, and the RECOD at 180 min decreased from 94.9 to 77.4% with the increase of $U_L$ from 0.29 to 0.73 cm/s. This result is a little different from that reported by the previous literature,29,35,36 probably because a higher $U_L$ led to a shortening of the residence time of the wastewater in the multichannel electrolytic unit. Besides, increasing $U_L$ also improves the convection effect of the solution between the electrode plates, which promotes the mass transfer of $\text{OCl}^-$ to the cathode plates and strengthens the cathode reaction of the consumption of oxidant ($\text{OCl}^-$),37 which results in a decrease of the COD removal rate. Finally, excessive $U_L$ can harm the solubility of electrogenerated chlorine,$^{29}$ which may not benefit the electrochemical degradation of organics in the ROLC.

2.1.3. Effect of $C_E$. The $C_E$ (mL/cm²) is defined as the corresponding wastewater treatment capacity per electrode area of the electrochemical oxidation process for the first time, which is a good index that can be used for the evaluation to scale up an electrochemical reactor. The effect of $C_E$ on the electrochemical degradation efficiency of recalcitrant organic pollutants is indicated in Figure 4. The figure shows that the COD elimination rate increased first with an increase in $C_E$ from 0.88 to 1.10 mL/cm² but decreased rapidly and sharply with a further increase in $C_E$ from 1.10 to 1.54 mL/cm². Thus, the optimal $C_E$ was 1.10 mL/cm². The results possibly imply that for a certain electrode area, there may exist a relatively good wastewater treatment capacity to match. For a smaller $C_E$, maybe the electrode plates were not in their best conditions to produce chlorine, and a little larger $C_E$ may improve the generating efficiency of chlorine. However, extremely larger $C_E$ means that an excessive organic matter needs to be treated with limited active chlorine because of the fixed anode plate area, which also agrees with our previous research studies.$^{38,39}$ Moreover, a larger $C_E$ also causes a reduction in the treatment cycles of wastewater in the electrolytic cell, which can significantly affect the electrochemical treatment efficiency.

2.2. Energy Consumption Evaluation. The energy consumption of the electrochemical treatment of ROLC was evaluated under a better condition of $I_A$ of 10.96 mA/cm², $U_L$ of 0.44 cm/s, and $C_E$ of 1.10 mL/cm², and water quality of the treated ROLC by electrochemical oxidation process for 3 h is listed in Table 1. As shown in Table 1, the MCFR exhibited excellent performance of simultaneous desalination and removal of recalcitrant organic compounds from ROLC, and the REs of the COD, total organic carbon (TOC), Cl⁻, and color could reach as high as 96.5, 73.9, 96.7, and 99.6%, respectively.

The $EC$ (kWh/(kg COD)) of the treatment process was calculated by eq 16, and the result is presented in Figure 5. As noted in Figure 5, $EC$ of the electrochemical oxidation of ROLC increased from the initial 9.15 kW/(kg COD) to 40.98 kW/(kg COD) gradually when the electrochemical treatment process continued from 0.5 to 3 h. At the first stage of electrochemical oxidation of 30 min, the $EC$ was as low as only 9.15 kW/(kg COD). The reason for this is not difficult to understand that the initial COD, TOC, and Cl⁻ were as high as 3414.7, 1987.5, and 15396.4 mg/L, respectively, which are easy to electrolyze and oxidize, and the COD sharply decreased to 1791.4 mg/L of RECOD of 47.5% as a result of a lowest $EC$ of the treatment process. However, with the prolongation of the treatment time, the degradation rate of recalcitrant organics in the ROLC was slowed down, resulting

![Figure 2](https://doi.org/10.1021/acsomega.1c01916)

Figure 2. Decolorization of the ROLC by electrochemical oxidation treatment. Reaction conditions: wastewater volume = 2.5 L, $I_A$ = 10.96 mA/cm², $U_L$ = 0.44 cm/s, $C_E$ = 1.10 mL/cm², initial Cl⁻ concentration = 15 396 mg/L, pH = 8.1−9.6, wastewater temperature 25.3−28.6 °C.

![Figure 3](https://doi.org/10.1021/acsomega.1c01916)

Figure 3. Effect of $U_L$ on the COD concentration (a) and RECOD (b) in the electrochemical treatment of ROLC. Reaction conditions: wastewater volume = 2.5 L, $I_A$ = 10.96 mA/cm², $C_E$ = 1.10 mL/cm², initial Cl⁻ concentration = 15 396 mg/L, pH = 8.1−9.6, and wastewater temperature 25.3−28.6 °C.
in a correspondingly sharp increase in the energy consumption of the electrochemical oxidation process. This phenomenon was also observed by many other researchers. However, it is worth mentioning that the COD degradation rate was much quicker than that of the TOC, which indicated that a larger proportion of organics in the ROLC may be volatile compounds and were difficult to degrade by the electrochemical treatment process. This hypothesis is also confirmed by the GC–MS results of a large number of esters, alkanes, etc. To exhibit the energy-saving and advanced organics removal performance of the MCFR, a comparison of energy consumption of different electrochemical oxidation processes was made and is presented in Table 2. As can be seen in the table, the MCFR reveals higher COD removal efficiency, good energy-saving performance, and larger wastewater treatment capacity for the electrochemical oxidation process of recalcitrant organic wastewater of landfill leachate and ROLC.

### 2.3. GC–MS Analysis

The organic compounds of the original ROLC and the treated ROLC for 3 h were detected by GC–MS under the conditions of \( I_A = 10.96 \text{ mA/cm}^2, U_L = 0.44 \text{ cm/s}, \text{initial Cl}^- \text{ concentration} = 15396 \text{ mg/L}, \text{pH} = 8.1–9.6, \) and wastewater temperature 25.3–28.6 °C. Compared with the GC–MS results of the original ROLC sample, we found that after 3 h of electrochemical oxidation treatment, the organics in the treated ROLC were greatly reduced with their content from 46.8% to 17.1%. Besides, the species of organics (credibility of ≥60% and percentage content of ≥0.1%) were significantly eliminated from 33 kinds (10 alkanes, 10 esters, 3 ketones, 2 alcohols, and 8 others) of the original wastewater compared to 13 kinds (7 chlorinated organics, 2 alkanes, 1 ester, 1 alcohol, 1 organic acid, and 1 alkene) of the treated ROLC. These results can be explained because of the electrochemical oxidation degradation of macromolecular organics into small molecular substances, chlorinated organic intermediates, and even completely oxidized into CO2 and H2O. The results also agree with RECOD and RETOC of the electrochemical oxidation process of recalcitrant organic wastewater of landfill leachate and ROLC.

### 2.4. Mechanism Analysis

In addition to the excellent elimination performance of recalcitrant organics in the ROLC, the electrochemical treatment with MCFR also showed a commendable ability of desalination. During the electrochemical oxidation process, white precipitates were observed to be generated in the treated ROLC solution and on the surface of cathode plates, which were separated for further X-ray diffraction (XRD) analysis. The phase compositions of the white precipitates were analyzed by XRD, and the results are
Table 2. Comparison of Average Energy Consumption (EC) of This Study with the Other Electrochemical Oxidation Processes

| Reactor Type                                      | Electrode/Oxidant Species                  | Water Treatment Capacity (L) | Electrochemical System Characteristics | RE and EC                                      |
|--------------------------------------------------|--------------------------------------------|-----------------------------|---------------------------------------|-----------------------------------------------|
| Differentially charged columns (DCC)              | anode: boron-doped diamond (BDD), cathode: Ca (0.5 mg/L) | 0.4                         | initial COD = 600 mg/L, flow velocity = 56 mL/min, electrode area = 50 cm², current density = 1.08 mA/cm² | RE = 98.2%, EC = 13.1 kWh/(kg COD) |
| Joint electrochemical system (JES)                | anode: Pt, cathode: 304 stainless steel plates | 2.0                         | initial COD = 2800 mg/L, flow rate = 500 L/h, reaction time = 4 h, electrode area = 6 cm², current density = 83 mA/cm² | RE = 94%, EC = 5.5 kWh/(kg COD) |
| Flow sequencing batch reactor (FSBR)              | anode: Ti/BDD, cathode: Pt | 600                          | initial COD = 3414.7 mg/L, circulating velocity = 0.44 cm/s, reaction time = 3 h, electrode area = 70 cm², current density = 4.3 mA/cm² | RE = 96%, EC = 11.0 kWh/(kg COD) |
| Single-chamber flow reactor (SCFR)                | anode: RuO₂/IrO₂ coated Ti plates, cathode: 304 stainless steel plates | 10                           | initial COD = 3308 mg/L, flow rate = 50 L/h, reaction time = 8 h, electrode area = 380 cm², current density = 10.96 mA/cm² | RE = 90.2%, EC = 7.9 kWh/(kg COD) |
| Multichannel flow reactor (MCFR)                  | anode: BDD, cathode: carbon-felt | 35                           | initial COD = 9900 mg/L, circulating velocity = 0.44 cm/s, reaction time = 3 h, electrode area = 380 cm², current density = 10.96 mA/cm² | RE = 90.2%, EC = 7.9 kWh/(kg COD) |
| RO leachate concentrate/0.5 RO leachate/0.05 RO leachate/10 | | | | RE = 95%, EC = 4.5 kWh/(kg COD) |
| RO leachate concentrate/10 | | | | RE = 94%, EC = 5.5 kWh/(kg COD) |

Note: This study: RE = 95.5% and EC = 3.5 kWh/(kg COD).
3. CONCLUSIONS

An easy scale-up multichannel flow reactor (MCFR) with electrode gaps of 5 mm was developed to simultaneously desalinate and eliminate recalcitrant organic compounds from RO leachate concentrate (ROLC) by the electrochemical oxidation process. High current density ($I_A$) favors high organic substance removal efficiencies, but higher superficial circulating velocity ($U_L$) and treatment capacity per electrode area ($C_E$) restrict pollutant removal efficiency. The better conditions for the electrochemical oxidation of ROLC with MCFR are $I_A$ of 10.96 mA/cm$^2$, $U_L$ of 0.44 cm/s, $C_E$ of 1.10 mL/cm$^2$, and the removal efficiency of COD, TOC, $\text{Cl}^-$, and color as high as 96.5, 73.9, 96.7, and 99.6%, respectively, for 3 h of electrochemical oxidation treatment. The MCFR also shows an energy-saving property with an average energy consumption of only 40.98 kW/(kg COD). After the electrochemical oxidation treatment, the organic species in ROLC reduce from 33 kinds to 13 kinds with relatively higher credibility ($\geq 60\%$) and content ($\geq 0.1\%$). The desalination mechanism of the electrochemical treatment process is mainly the precipitation of NaCl and CaCO$_3$ and the consumption of $\text{Cl}^-$. Therefore, MCFR provides a promising solution for the treatment of ROLC and exhibits prospects of industrial application.

4. MATERIALS AND METHODS

4.1. Water Quality of ROLC. ROLC is a dark-colored and poisonous organic wastewater, which is classified as a hazardous waste in China. The ROLC used in this paper...
was collected from a local MSW incineration plant in Banan district, Chongqing City. The main physicochemical characteristics of the ROLC are summarized in Table 3. As illustrated in the table, the ROLC has a high color of 2500 times, high salt content with Cl\(^-\) of 15 396.4 mg/L, total alkalinity (CO\(_3^{2-}\)/HCO\(_3^-\)) of 11 500.0 mg/L, high concentration of organic compounds with COD and TOC of 3414.7 and 1987.5 mg/L, respectively, and extreme refractory biodegradation with BOD\(_5\)/COD of 0.06.

### 4.2. Experimental Setup and Operation

The electrochemical oxidation of ROLC was conducted in MCFR, and the experimental setup is shown in Figure 9. The MCFR consisted of the main body of an acrylic multichannel electrolytic cell (190 mm × 50 mm × 320 mm, L × W × H), a direct current (DC) power supply (KXX-6050D, Zhaxiong Co.), a circulation pump (HQB-2200, Sensen Co.), a water tank (10 L), and a rotameter. The electrodes were placed in the main body with three RuO\(_2\)/IrO\(_2\)-Ti plates as the anode and four 304 stainless steel plates as the cathode (20 cm × 19 cm, L × W; thickness of 1 mm), and the distance between the electrode plates was fixed at 5 mm.

During experiments, a certain volume (2.0, 2.5, 3.0, 3.5 L) of ROLC was added into the water tank. Then, it was circulated in the system by a circulation pump for 10 min, and 10 mL of sample was collected and labeled as the zero-time sample to detect the original water quality. After that, the DC power supplier was turned on to start the electrochemical treatment process. The electrooxidation process was carried out at a constant ambient temperature (25.0 ± 1.0 °C), different \(I_A\) (4.39, 6.58, 8.77, 10.96, and 13.16 mA/cm\(^2\)), \(U_L\) (0.29, 0.44, 0.58, and 0.73 cm/s), and \(C_E\) (0.88, 1.10, 1.32, and 1.54 mL/cm\(^2\)). The samples were taken out from the reactor after a fixed time interval to detect the water quality. Each experiment was performed in duplicate, and the deviation of the results between the same experiments was controlled at ±3%.

### 4.3. Analytical Method

The COD of the ROLC was analyzed according to the Chinese water quality determination of the COD of the fast digestion spectrophotometric method. The absorbance was measured using a UV–vis spectrophotometer (TU-1901, Beijing Puxi Analyzing Devices Ltd. Co., China). The TOC of the wastewater was detected using a TOC analyzer (TOC-LCPH, Shimadzu, Japan). The concentrations of active chlorine (Cl\(_2\)/ClO\(_2\)) and chloride ions (Cl\(^-\)) were measured by iodimetry\(^{29}\) and silver nitrate titration methods, respectively. The color and alkalinity were determined by multiple dilution and acid–base titration methods, respectively.

An X-ray diffractometer (XRD-7000, Shimadzu, Japan) with Cu Kα radiation (\(λ = 1.5418\) Å, 40 kV, 30 mA) at a scan step of 0.02° and a scan speed of 2°/min was used to determine the phase compositions of the precipitate generated and collected during the electrochemical oxidation process.

Gas chromatography–mass spectrometry (GC–MS, Agilent 7890A-5975C) was used to determine the organic compounds of the ROLC before and after electrochemical treatment with an OV1701 capillary column (30 m × 0.25 mm × 0.25 μm). Before the GC–MS test, 500 mL of ROLC or treated ROLC sample was extracted with trichloromethane (CHCl\(_3\), high-performance liquid chromatography (HPLC) grade) under the original pH of 8.1, then under acidic condition (pH 2.0), and at last under alkaline condition (pH 12.0) using a separating funnel. Each extraction of the same pH was performed three times with 25 mL of CHCl\(_3\), respectively. The combined extract organic phases were concentrated to 2–3 mL at 40 °C with a rotary evaporator and then dehydrated with anhydrous Na\(_2\)SO\(_4\) for GC–MS analysis. During the test, the sample injection volume was 0.5 μL, and high-purity helium (He, 99.999%) was used as the carrier gas with a flow rate of 1.0 mL/min. The heating program of the GC–MS test is kept at 40 °C for 5 min, then heated at a rate of 4 °C/min to 250 °C, and then kept for 5 min. The voltage of the electron multiplier is 1341 eV, and the emission electron energy is 69.9 eV.\(^{30,47}\)

### 4.4. Calculation of RE and EC

The REs of COD, TOC, and Cl\(^-\) by the electrochemical oxidation process were calculated as eq 15

\[
\text{RE} = \frac{C_0 - C_t}{C_0} \times 100\%
\]

where \(C_0\) and \(C_t\) (mg/L) are the concentrations of COD, TOC, and Cl\(^-\) at the initial and \(t\) time of the electrochemical treatment, respectively.

The EC of the electrochemical oxidation process can be calculated with eq 16\(^{42}\)

\[
\text{EC} = \frac{1000Ut}{V(C_0 - C_t)}
\]

where \(\text{EC} \ (\text{kWh/} \text{kg COD})\) is the average energy consumption of electrochemical treatment process; \(U \ (\text{V})\) is the DC voltage; \(I \ (\text{A})\) the is the DC current; \(t \ (\text{h})\) is the electrochemical oxidation time; \(V \ (\text{L})\) is the volume of the treated wastewater; and \(C_0\) and \(C_t\) (mg/L) are the COD of ROLC at the initial and \(t\) time of the electrochemical treatment, respectively.
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