

Article

Efficiency of Advanced Oxidation Processes for Treating Wastewater from Lithium-Ion Battery Recycling

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Abstract

A treatment process was developed for effluents from direct physical lithium-ion battery (LIB) recycling with a focus on the removal of organic contaminants. The high chemical oxygen demand to biological oxygen demand ratio (COD/BOD₅) of 3.9–4.6 indicates that biological treatment is not feasible. Therefore, three advanced oxidation processes were evaluated: UV/H₂O₂ oxidation, the Fenton process and electrochemical oxidation. Two target scenarios were considered, namely compliance with the limit for discharge into the sewer system (COD = 800 mg/L) and compliance with the stricter limit for direct discharge into surface waters (COD = 200 mg/L). Under the investigated conditions, UV/H₂O₂ oxidation and the Fenton process did not meet the required discharge limits and exhibited high chemical consumption. In contrast, electrochemical oxidation achieved both discharge criteria with a lower energy demand, requiring 32.8 kWh/kgCOD_{removed} for sewer discharge and 95.3 kWh/kgCOD_{removed} for direct discharge. An economic assessment further identified electrochemical oxidation as the most cost-effective option, with treatment costs of EUR 6.63/m³, compared to EUR 17.31/m³ for UV/H₂O₂ oxidation and EUR 28.66/m³ for the Fenton process. Overall, electrochemical oxidation proved to be the most efficient and environmentally favorable technology for treating wastewater from LIB recycling, enabling compliance with strict discharge regulations while minimizing the chemical and energy demand.

Keywords: lithium-ion battery recycling; wastewater treatment; advanced oxidation process; electrochemical oxidation; Fenton process; UV/H₂O₂ oxidation; energy assessment



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1. Introduction

To preserve critical raw materials and to reduce dependency on external sources, the European Union (EU) is actively working to develop a comprehensive lithium-ion battery (LIB) recycling infrastructure within Europe. This initiative aims to enhance resource efficiency, minimize environmental impacts and support the transition to a circular economy by recovering and reusing key materials such as lithium, cobalt and nickel from end-of-life batteries. Kampker et al. predict a volume of recycled batteries corresponding to a storage capacity of 38.8 GWh in 2030 in the EU [1]. Consequently, it is critical to assess

and mitigate the environmental impact of recycling processes by reducing emissions and optimizing energy efficiency [2]. To achieve this, the development of holistic recovery and treatment methods is essential. Within this context, a water-based direct physical recycling process of LIBs was developed at Fraunhofer IWKS [3–5], resulting in the recovery of a high-quality black mass in addition to metallic components (Cu, Al and steel) and plastic foils as value-added byproducts. However, this process requires water. As the process water becomes mainly contaminated with the electrolytes of LIBs, which consist of aprotic organic solvents and conducting salts, a procedure to remove the contaminants from the process water was developed in the current work. In particular, the degradation of these poorly biodegradable organic carbonates by using advanced oxidation processes (AOPs) was investigated and assessed [6].

Figure 1 schematically shows the developed LIB recycling procedure, where the LIBs are opened and cathode and anode foils are delaminated in a reactor filled with water by using electrohydraulic fragmentation (EHF) [3–5]. The various battery components are subsequently separated by a combination of skimming and sieving (SuSi) processes. The resulting process water possesses high concentrations of phosphorus and fluorine, as well as a high chemical oxygen demand (COD) [7]. Table A1 in Appendix A shows the nominal composition of the process water. Three methods have been considered in the present study to reduce the COD: UV/H₂O₂ oxidation, the Fenton process and electrochemical oxidation. During UV/H₂O₂ oxidation, hydroxyl radicals (OH radicals) are generated by the homolytic cleavage of H₂O₂ by UV irradiation [8]. Depending on the cleaning objective and the type of wastewater, between 0.0025 and 2 kg/kgCOD_{removed} are typically used [9–13]. The classic Fenton process is a catalytic conversion that uses Fe²⁺ as the catalyst and H₂O₂ as the oxidizing agent [14]. The principal reactions are shown in Equations (1) and (2):

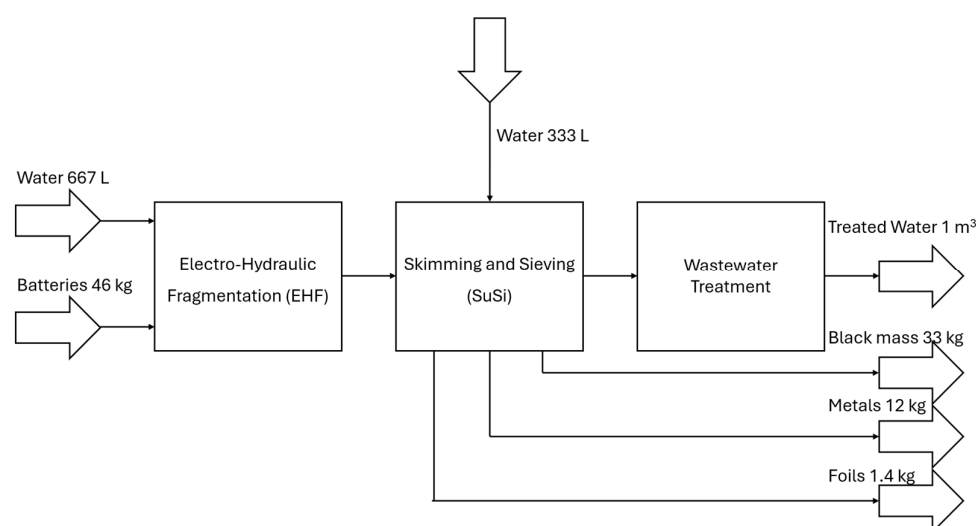
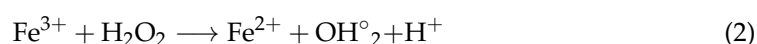
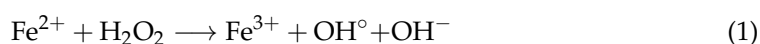


Figure 1. Flow chart of the recycling route, including a mass assessment for the treatment of 1 m³ of wastewater.

A literature review has shown that between 0.96 and 2 kg/kgCOD_{removed} H₂O₂ is used and that the mass ratio of H₂O₂/Fe²⁺ is between 0.32 and 290 [15–21]. During electrochemical oxidation, organic material is mainly degraded by OH radicals, but there is also direct oxidation at the electrode surface and indirect oxidation by other oxidative species such

as H_2O_2 [22]. Typical current densities reported in the literature are 6.6–66 mA/cm^2 , with energy demands of 17–534 $\text{kWh}/\text{kgCOD}_{\text{removed}}$ [23–30]. It is difficult to draw meaningful comparisons based on the values in the literature, as the cleaning targets vary widely. They range from 31.4 to 100% COD removal [9,23,27]. There are differences in input concentrations. Here, the COD ranges between 397 and 60,500 mg/L [10,19]. In addition, the types of wastewaters and their composition vary significantly, e.g., wastewater from textile manufacturing, pulp production and petrochemicals. Therefore, direct comparisons under similar conditions are essential for drawing meaningful conclusions.

The AOPs were evaluated and qualified for two cases:

- (i) Discharge of the treated process water into the sewer system to be further treated in a municipal wastewater treatment plant with a COD limit value of 800 mg/L [31].
- (ii) Direct discharge of industrial wastewater into a water body. According to the German legal framework, the COD needs to be reduced to at least 200 mg/L to comply with the limit values defined in Annex 27, “treatment of waste by chemical-physical plants” [32].

The wastewater is currently treated in a chemical–physical plant because it exceeds the sewer system’s limits for COD, fluoride and total phosphorus. Since, at the time of publication, there is no annex to the wastewater ordinance that deals with the recycling of batteries, one aim of this work is to provide recommendations for the future preparation of such an annex. Based on this background, the following objectives have been set for this publication:

- (i) Investigate whether the recovery of organic carbonates is an option for wastewater treatment.
- (ii) Evaluate the treatment performance of three AOPs: UV/ H_2O_2 oxidation, the Fenton process and electrochemical oxidation.
- (iii) Compare the AOPs in terms of energy demand, chemical requirements and discharge compliance, considering two scenarios: discharge into the sewer system and direct discharge into a water body according to German wastewater ordinance limits.
- (iv) Provide the leveled treatment costs per m^3 for the three AOPs.
- (v) Derive recommendations for the most suitable treatment option for LIB recycling process water and for potential regulatory parameters in future wastewater legislation.

2. Materials and Methods

2.1. Analytical Methods

All samples were filtered using a 0.45 μm polyvinylidene fluoride (PVDF) filter prior to analysis. The COD and biological oxygen demand (BOD_5) were analyzed with cuvette tests (LCK114 and LCK555) (Hach Lange GmbH, Düsseldorf, Germany) by measuring absorbance at wavelengths of 605 nm and 620 nm with the Hach Lange (Düsseldorf, Germany) DR3900, respectively. The COD/ BOD_5 ratio was used to evaluate the biodegradability of the process water.

Total organic carbon (TOC) was measured as non-purgeable organic carbon (NPOC) using an multiN/C 3100 TOC Analyzer (Analytik Jena, Jena, Germany). To ensure that only organic carbon was measured, CO_2 was stripped by adding two drops of 2 mol/L hydrochloric acid to each 5 mL cuvette.

The concentration of organic carbonates was determined using an AZURA Compact high-performance liquid chromatography (HPLC) system. An Eurospher III 150 $\times$ 4 mm 100-5 C 18 column (Knauer Wissenschaftliche Geräte GmbH, Berlin, Germany) was used in the chromatograph. The carbonates were analyzed by a diode array detector (DAD) (Knauer Wissenschaftliche Geräte GmbH, Berlin, Germany) at 194 nm. Organic carbonates

have a comparatively high limit of detection and limit of quantification [33–35]. To increase the concentration of organic carbonates, a battery cell was opened and immersed in 35 mL of ultrapure water.

To check how much of the COD was still present as organic carbonates, the measured COD was compared with the calculated COD. The COD was calculated using the following Equation (3), which assumes the complete oxidation of carbon to CO₂ (3).

$$\text{COD}_{\text{cal.}} = \frac{c_{\text{org.carbonate}} \cdot x_{\text{Carbon}} \cdot \tilde{M}_{\text{O}_2}}{\tilde{M}_{\text{org.carbonate}}} \quad (3)$$

$c_{\text{org.carbonate}}$ Concentration of organic carbonates [mg/L]

x_{Carbon} Number of carbon atoms in the carbonate molecule [-]

$\tilde{M}_{\text{org.carbonate}}$ Molar mass of organic carbonates [g/mol]

$\tilde{M}_{\text{O}_2}$ Molar mass of oxygen molecules [g/mol]

Using a DR6000 spectrophotometer (Hach Lange GmbH, Düsseldorf, Germany), a spectrum was measured over the spectral range from 200 to 800 nm. An inductively coupled plasma optical emission spectrometer (ICP-OES) 3800 (Perkin Elmer, Waltham, MA, USA) was utilized to quantify the content of aluminum, calcium, magnesium, phosphorus, sulfur and silicon in the precipitate. The sample was digested using aqua regia at 240 °C for 1 h in a Turbowave (MLS-MWS GmbH, Leucht kirch im Allgäu, Germany).

Phase analysis of the precipitates was conducted using powder X-ray diffraction (PXRD) with an Empyrean diffractometer (Malvern Panalytical, Malvern, UK) operated in the Bragg–Brentano geometry. The diffraction patterns were recorded at 40 mA and 40 kV using a cobalt X-ray source ($\lambda = 1.78901 \text{ \AA}$) over a 2θ range from 10 to 75°, with a scan speed of 0.006°/s and a step size of 0.013°. Phase identification was performed using HighScore Plus version 4.9 software (Malvern Panalytical, Malvern, UK) in combination with the Inorganic Crystal Structure Database (ICSD; FIZ Karlsruhe, Karlsruhe, Germany).

2.2. LIB Recycling Route

The input material was end-of-life Bosch e-bike battery systems, each containing 40 cells (type 18650) with NMC811 (LiNi_{0.8}Mn_{0.1}Co_{0.1}O₂) cathode material. In the first step, the battery systems were dismantled manually. For safety reasons, the batteries were discharged to a 10% state of charge. The EHF -400 plant (ImpulsTec GmbH, Radebeul, Germany) with the dimensions width = 1940 mm; height = 2430 mm and depth = 1400 mm was used to open the battery cells and to delaminate the anode and cathode foils. The EHF system is shown in Figure 2a and its operating scheme is illustrated in Figure 2b. Three electrodes were immersed in a reactor containing 30 battery cells and 20 L of water. The high-voltage (40 kV) discharge of the electrodes led to the formation of pressure waves in the water. The pressure waves opened the battery cells at the material interfaces. This process had an energy demand of 1.78 kWh/kg_{Batteries} [2,3]. Afterwards, the SuSi process was used to separate the EHF slurry into separate fractions of black mass, plastics and metals, which required an additional 10 L of water.

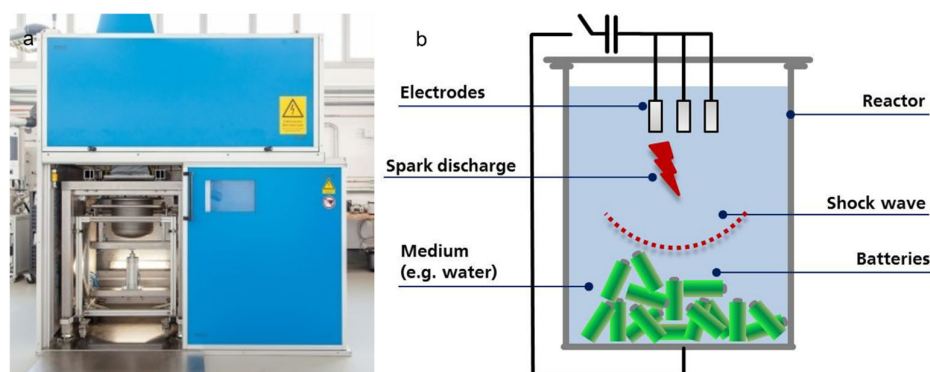


Figure 2. (a) Front view of EHF plant and (b) schematic diagram of EHF showing the three electrodes in the water-filled reactor with batteries [3,36].

2.3. Advanced Oxidation Processes

UV/H₂O₂ oxidation experiments were conducted on a 500 mL laboratory scale. A 15 W low-pressure mercury vapor lamp with a radiation maximum at 254 nm was used. The system was cooled using water. The H₂O₂ concentration was between 3 and 9 g/L and the pH value was set at 4.

The Fenton experiments were also carried out at laboratory scale (50 mL). The process water was prepared with FeSO₄ · 7 H₂O and 30% hydrogen peroxide solutions. The concentrations of FeSO₄ · 7 H₂O and H₂O₂ solutions were varied in the range of 1–12 g/L and 1–16 g/L, respectively. COD removal was tested at different reaction times of 30 min, 1 h, 2 h, 6 h and 24 h.

The electrochemical oxidation experiments were carried out at laboratory scale (200 mL). The experiments were performed with a boron-doped diamond (BDD) anode and two steel cathodes. The area of each electrode was 40 cm², and the distance between the anode and cathodes was 0.5 cm. The system was operated at a constant direct current of 25–100 mA/cm² with the corresponding voltages. A schematic illustration of the experimental setup is provided in Figure A1 in the Appendix A.

2.4. Economic Analysis

For the economic analysis, upscaling was performed to treat 16.7 m³/d of wastewater. The period of 2024/2025 was considered. These conditions were chosen because a battery recycling plant of this scale was planned. The upscaling was carried out according to a three-step method described by Weyand et al. [37]. The service life was determined using official amortization tables and listed in Table A2 in Appendix A. A time horizon of 20 years was considered. Since all three processes had comparable maintenance and personnel costs, these costs were not taken into account.

The capital expenditures (CAPEX) are the summed up investment costs of the plant parts, and operational expenditures (OPEX) are energy, chemical and disposal costs. These costs are listed in Table A2 in Appendix A as well. The investment costs were based on supplier quotations. Equation (4) shows the calculation of the levelized treatment costs (LTCs) to treat 1 m³.

$$LTC = \frac{1}{\dot{V}} \left(\frac{CAPEX}{t} + OPEX \right) \quad (4)$$

$\dot{V}$ Volume flow (in this case 16.7 m³/d)

t Time

3. Results and Discussion

3.1. Process Water Composition

The process water composition is described in detail in our previous publication, and the results are summarized in Table A1 [7]. Several wastewater samples were analyzed, with COD ranging from 916 to 2270 mg/L and TOC from 452 to 687 mg/L. The present work focuses on the organic components. First, it was investigated whether the recovery of the organic carbonates is a viable option.

Because of the high limit of detection of the organic carbonates, a highly concentrated sample was prepared. Here, the COD was measured to be 108,000 mg/L. Table 1 lists the concentration of the detected organic carbonates that were found in the HPLC/DAD analysis. The COD of the organic carbonates was then calculated according to Equation (1). Comparing the measured and the calculated COD, it was found that only 39.6% of the COD was present as pure organic carbonates. The remainder were degradation products such as methanol, ethanol, fluorobenzene and cyclohexylbenzene [35]. This was mostly due to the fact that the batteries were at the end of their lives. During the use phase, the electrolyte is degraded [35,38]. Additionally, the recycling process can also lead to further degradation of the organic components. In the case of unused batteries, e.g., from surplus production, there would likely be fewer degradation products. In this case their recovery should be evaluated separately. Thus, since the organics are mainly present as degradation products of the organic carbonates due to the recycling of end-of-life batteries, the organic carbonates themselves are not recovered. Their recovery is complex and would involve cost-intensive processes such as distillation. Moreover, highly pure organic carbonates are required for marketing and reuse in LIBs.

Table 1. Concentration of organic carbonates in concentrated process water samples and the calculated COD.

Organic Carbonate	Ethylene Carbonate (EC)	Propylene Carbonate (PC)	Dimethyl Carbonate (DMC)	Ethyl Methyl Carbonate (EMC)	Total
Concentration [mg/L]	3760	4670	23,990	5920	-
COD _{cal} [mg/L]	4099	5855	25,567	7279	42,800

3.2. UV/H₂O₂ Oxidation

In general, two main approaches are available for the degradation of organic compounds: biological treatment and AOPs. A COD/BOD₅ ratio below 2 indicates that biological treatment may be effective. Wastewater from LIB recycling has a COD/BOD₅ ratio of 3.9–4.6. Therefore, AOPs were chosen for the removal of the organic load [7].

Figure 3a shows the TOC removal for different H₂O₂ concentrations. The experiment with 3 g/L was aborted prematurely. Figure 3b shows the COD removal over time of the sample using 6 g/L H₂O₂.

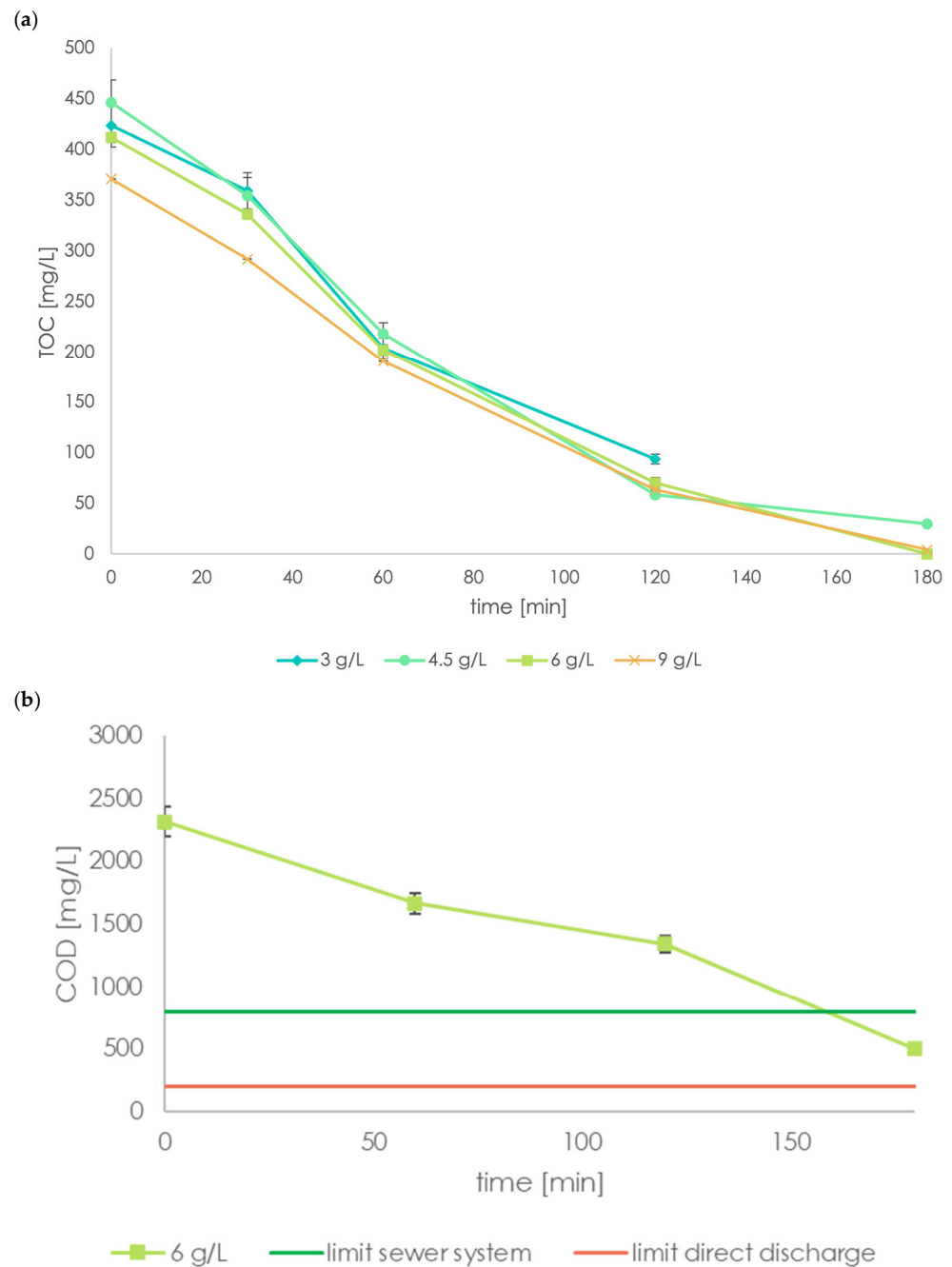


Figure 3. (a) TOC removal for different H_2O_2 concentrations ranging from 3 to 9 g/L over a time period of 180 min at a pH value of 4. (b) COD removal of the 6 g/L H_2O_2 concentration. Limit value in green (sewer system) and limit value in red (direct discharge). The error of the analytical method has been included.

Only the COD limit value for discharge into the sewer system was reached. The limit value was reached after 158.5 min. The direct discharge limit might have been achieved if the reaction time were extended. A linear regression yielded a reaction time of 221 min. Extending the reaction time by 23% also increased the energy demand by 23% and the process became less economically feasible. A further increase in concentration of the oxidation agent showed the same degradation rates. However, lower H_2O_2 concentrations led to slightly lower degradation rates. Nevertheless, TOC removal reached 100% using a H_2O_2 concentration of 6 g/L beyond 180 min. Although a complete degradation of the TOC was observed (see Figure 3a), the COD value was only reduced by 78%. This may

be the case for three possible reasons. Firstly, H_2O_2 can lead to incorrect and increased measurement results in analyzing the COD [39]. According to Kang et al., the error can be as high as $16 \text{ mgCOD}/\text{mmolH}_2\text{O}_{2\text{residual}}$ [40]. Secondly, CO_2 was purged during the TOC measurement process, which can also lead to the removal of volatile carbon compounds, so-called purgeable organic carbon. To evaluate their influence, the COD was measured before and after purging of CO_2 , and it was found that the COD decreased by 9%. This is, however, not sufficient to fully explain the discrepancy between the TOC and COD removal.

Lastly, inorganic substances that are not oxidized during UV/ H_2O_2 treatment may subsequently be oxidized under the strongly oxidative conditions of dichromate-based COD digestion, resulting in elevated COD values. In particular, reduced phosphorus and sulfur species act as electron donors during dichromate digestion and are therefore detected as oxygen demand, despite not contributing to organic carbon. However, the discrepancy between TOC and COD raises the question of whether TOC is, for highly inorganically contaminated wastewater, a more appropriate sum parameter for the organic load when discussing necessary parameters for an annex in the wastewater ordinance for battery recycling.

An important comparison parameter is the energy requirement of the process. An initial COD of 1632 mg/L is assumed in order to be able to better compare the energy and chemical requirements of the three oxidation processes. The process requires $56.3 \text{ kWh/kgCOD}_{\text{removed}}$ or 43.3 kWh/m^3 to reach the COD limit value to allow for discharge into the sewer system after 158 min of treatment. Schwaickhardt et al. require $38.6 \text{ kWh/kgCOD}_{\text{removed}}$ to treat hospital laundry wastewater, whereas Babaei et al. require $131.1 \text{ kWh/kgCOD}_{\text{removed}}$ to treat petrochemical wastewater [9,13]. The experiments were performed on a similar experimental scale. Nevertheless, the energy requirements of the battery process water were of the same order of magnitude as the energy demands found in the literature for other process waters.

Another important factor was the amount of chemicals needed. This process had a high H_2O_2 requirement, at $7.8 \text{ kgH}_2\text{O}_2/\text{kgCOD}_{\text{removed}}$, compared to the values in the literature, which range between 0.0025 and $2 \text{ kgH}_2\text{O}_2/\text{kgCOD}_{\text{removed}}$ [10–13]. One factor that negatively influences the energy and chemical requirements of the process is the fact that the process water absorbs light at the wavelength of the UV lamp (254 nm). The absorption spectrum is shown in Figure A2 in Appendix A. Due to strong absorption of UV radiation by the wastewater matrix at 254 nm, not all energy emitted by the UV lamp was available for hydroxyl radical generation, as part of it is dissipated as heat. However, the reaction system was actively temperature-controlled and remained close to ambient conditions, indicating that removal efficiency was primarily limited by reduced photon availability rather than by temperature-dependent reaction kinetics.

Another disadvantage with regard to the maintenance of the UV/ H_2O_2 oxidation plant for this particular water is that the process water from battery recycling contains the anion hexafluorophosphate (PF_6^-). PF_6^- hydrolyzes to hydrofluoric acid (HF) and phosphoric acid (H_3PO_4) at low pH values [7]. Dissolved fluorine occurs in different species (F^- , HF or HF_2^-) depending on the pH. At higher pH values, fluoride is predominantly present as F^- , whereas decreasing the pH below 4 leads to a steep increase in the proportion of HF [7]. For this reason, the UV/ H_2O_2 experiments were conducted at pH 4, representing a compromise between favorable conditions for hydroxyl radical formation and operational constraints related to fluoride hydrolysis and material compatibility.

3.3. Fenton Process

First, the concentration of $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, which yielded the highest COD removal, was tested. The results are shown in Table 2. The experiment was carried out at pH 4 and with an H_2O_2 concentration of 4 g/L.

Table 2. COD removal at different $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ concentrations at pH 4, dosage of H_2O_2 4 g/L.

Concentration $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ [g/L]	1	3	6	9	12
COD removal [%]	38.9	42.7	58.1	58.3	57.9

COD removal increases until a concentration of 6 g/L. Thereafter, COD removal stagnates. Figure 4 shows the COD removal at different H_2O_2 concentrations and a constant $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ concentration of 6 g/L.

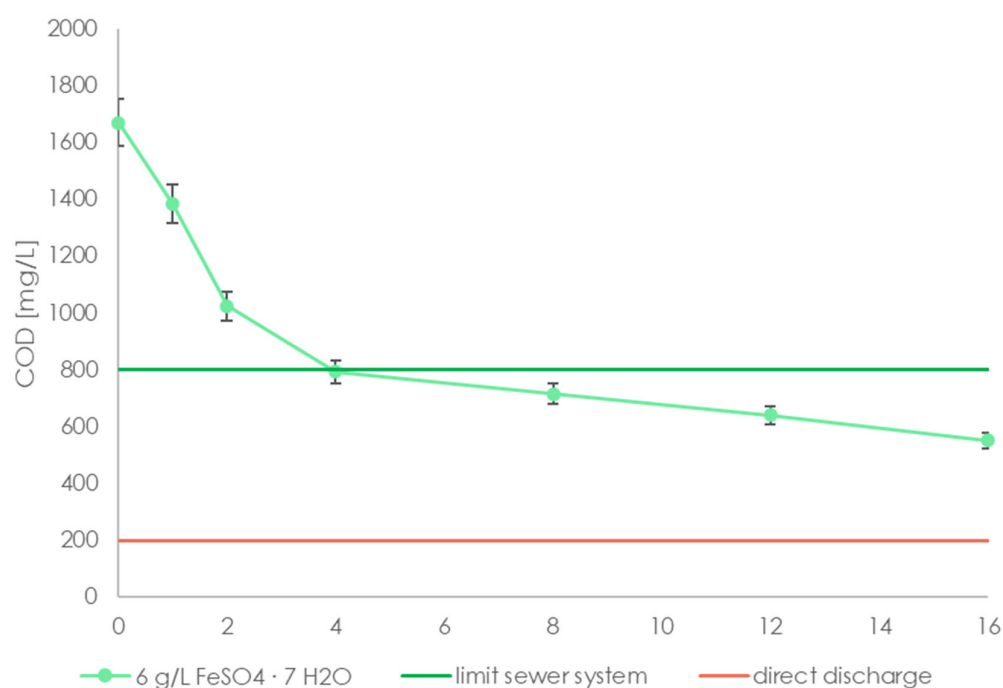


Figure 4. COD removal at various H_2O_2 concentrations (6 g/L $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, pH = 4, reaction time 24 h). The limit values for two discharge cases are indicated in dark green (sewer system) and in red (direct discharge). The error of the analytical method has been included.

At an H_2O_2 concentration below 4 g/L, a strong increase in residual COD can be seen. From a concentration of 8 g/L H_2O_2 , the COD removal rate increases only slightly. None of the samples met the requirement of COD removal to allow for discharge directly into a water body. However, removal rates of over 95.4% were achieved in the TOC measurements carried out for quality control.

The optimum pH value for Fenton processes is 2.5–4, since, at higher pH values, inactive iron oxohydroxides are formed and ferric hydroxide precipitates [41]. Due to the hydrolysis of the PF_6 anion and the resulting formation of HF, the pH value was not set below 4. It can be assumed that lower pH values would have yielded better results. COD degradation stagnated at around 67%, while TOC was almost completely removed. As with UV/ H_2O_2 oxidation, complete COD degradation does not take place with near-complete TOC degradation. Compliance with the limit values for direct discharge by increasing the reaction time is questionable, as the reaction is complete after 24 h. Figure A3 in Appendix A shows the COD removal over time. Table 2 and Figure 4 show that increasing

the concentration of the reagents does not result in a significantly higher degradation rate. Changes to the process design, e.g., through cascading, are also conceivable in order to maintain a constant concentration of the reagents.

Due to the high reaction time, the measurement error due to the presence of H_2O_2 can be excluded. As the loss of purgeable organic carbon was the same in all three oxidation processes, the only remaining reason is that inorganic components were oxidized in the cuvette test.

If the values achieved are compared with data in the literature for different industrial wastewaters, it is noticeable that there was a comparatively high oxidizing agent consumption of $8.06 \text{ kg}_{\text{H}_2\text{O}_2}/\text{kgCOD}_{\text{removed}}$. The values in other publications are between 0.96 and $2.00 \text{ kg}_{\text{H}_2\text{O}_2}/\text{kgCOD}_{\text{removed}}$ and, therefore, significantly lower [15,16,19,20]. The $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ ratio of 2.52 shows that the iron requirement is also comparatively high [15–21]. In summary, it can be stated that the chemical requirement for this process water was relatively high compared to other Fenton processes. One advantage of the Fenton process is the low energy demand, but it must be noted that the sulphate concentration is too high for discharge into the sewage system in the case of industrial implementation. This cannot be sufficiently reduced by a precipitation reaction. Therefore, for example, nanofiltration would have to be performed downstream, thus increasing the energy demand of the process.

3.4. Electrochemical Oxidation

Figure 5 shows COD degradation by electrochemical oxidation at currents of $25\text{--}100 \text{ mA}/\text{cm}^2$ over a period of 1 h.

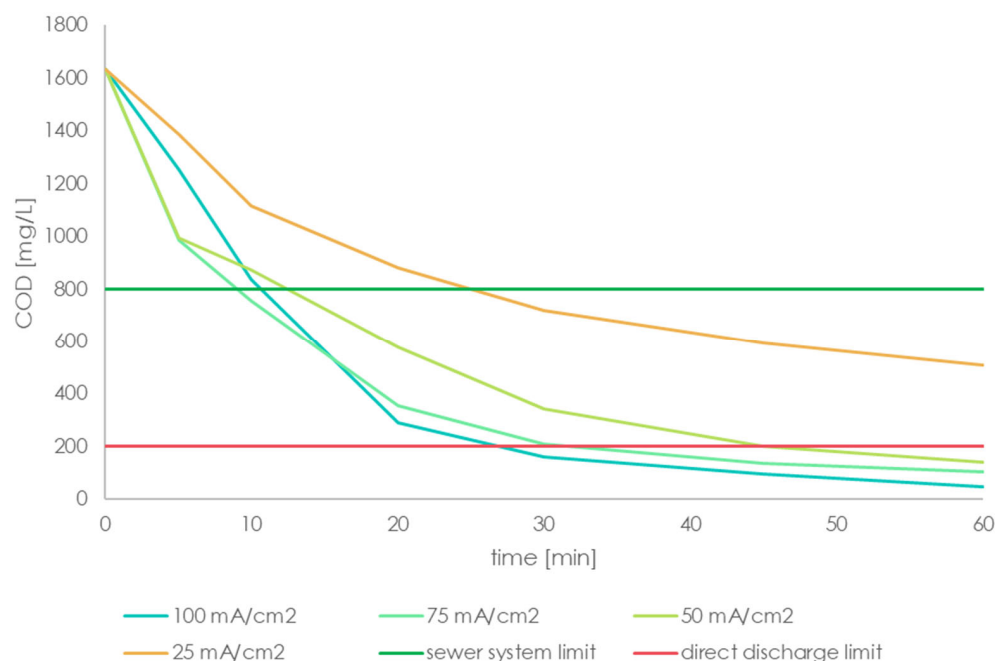


Figure 5. COD removal over 1 h at current densities between 25 and $100 \text{ mA}/\text{cm}^2$. Limit values for discharge cases are indicated by the dark green (sewer system) and red lines (direct discharge). The error of the analytical method has been included.

Electrochemical oxidation is the only AOP that achieves both discharge limits. With a current density of 25 or $50 \text{ mA}/\text{cm}^2$, the limit values for discharge into the sewage system or direct discharge were reached, respectively. It is noticeable that a plateau of $67\text{--}78\%$ of the removed COD was reached in previously investigated processes, as well as when using electrochemical oxidation at a current density of $25 \text{ mA}/\text{cm}^2$. Since electrochemical

oxidation also reaches these limits at 25 mA/cm² with a COD removal of 70%, this cannot be explained by measurement error due to the use of H₂O₂ as an oxidation agent, since no oxidation agent was used. This again indicates the oxidation of inorganic components. During the experiment, solids precipitated in the samples when current densities of 50, 75 and 100 mA/cm² were applied.

In terms of energy consumption, it is more efficient to use low current densities with longer reaction times, as otherwise, more waste heat is produced. To reach the limit value for discharge into the sewage system, 32.8 kWh/kgCOD_{removed} (reaction time: 22.9 min, current density 25 mA/cm²) is required, or 95.3 kWh/kgCOD_{removed} (reaction time: 58.7 min, current density 50 mA/cm²) for direct discharge into a water body. This means the energy demand per cubic meter would be 27.3 kWh/m³ for discharge into the sewer system and 136.5 kWh/m³ for direct discharge into a water body. A comparison with values in the literature (see Table A3) shows that the energy required to treat the process water from the present study is comparable to other types of industrial wastewater [23–30]. This means that the electrochemical oxidation process uses 41.7% less energy for the same COD removal result compared to UV/H₂O₂ oxidation. Due to the lower energy requirements and the fact that the process does not need the addition of chemicals, electrochemical oxidation is recommended for the treatment of battery recycling process water.

Analysis of the Solid Precipitate

The resulting precipitate was characterized via ICP-OES, with the results shown in Table 3, and with structural analysis using PXRD, with the results given in Figure 6. The elements were selected based on the wastewater composition and ICP-OES measurements before and after oxidation [7].

Table 3. Composition of the precipitation product after electrochemical oxidation for 1 h at 100 mA/cm², measured by ICP-OES.

Al (wt%)	Ca (wt%)	Mg (wt%)	P (wt%)	S (wt%)	Si (wt%)
0.27	32.79	1.93	1.48	0.33	0.04

PXRD analysis was carried out to determine the phase composition of the solid precipitate.

In accordance with the chemical analysis (Table 3), CaCO₃ with trigonal symmetry (PDF No. 98-016-4935) was detected by PXRD as the predominant Ca compound in the precipitate. In addition, CaCO₃ is known to be able to incorporate smaller amounts of Mg into its crystal lattice, a phenomenon known in both the minerals calcite and aragonite, as well as from cement studies [42,43]. Other elements such as Al, P, S and Si did not display a crystalline phase within the diffraction pattern, which could be due to their low volume fraction or to the formation of amorphous precipitates that could not be detected using this technique.

Operationally, CaCO₃ precipitation during electrochemical oxidation can cause scaling on electrodes or downstream piping, which may reduce process efficiency over time and necessitate regular cleaning or descaling. At higher current densities, pronounced local pH gradients and enhanced ion migration near the electrode surfaces promote supersaturation with respect to CaCO₃, particularly due to cathodic hydroxide formation and increased local concentrations of calcium and carbonate species, thereby favoring salt precipitation. However, in electrochemical systems, scaling on electrodes can be effectively mitigated by periodically reversing the current direction, which promotes the detachment or dissolution of mineral deposits and enables in situ electrode cleaning. While CaCO₃ is environmentally inert and non-toxic, it may still be relevant with respect to compliance with limit values for total solids (sewer system: sedimentable solids = 10 mL/L; direct discharge: n/a [31,32]).

Although the amount of sedimentable solids was below 1 mL/L in the present study, this limit value should be considered in future applications. In the current study, the precipitate was retained within the reactor and regular rinsing combined with polarity reversal effectively removed deposits from the electrodes and reduced operational risks.

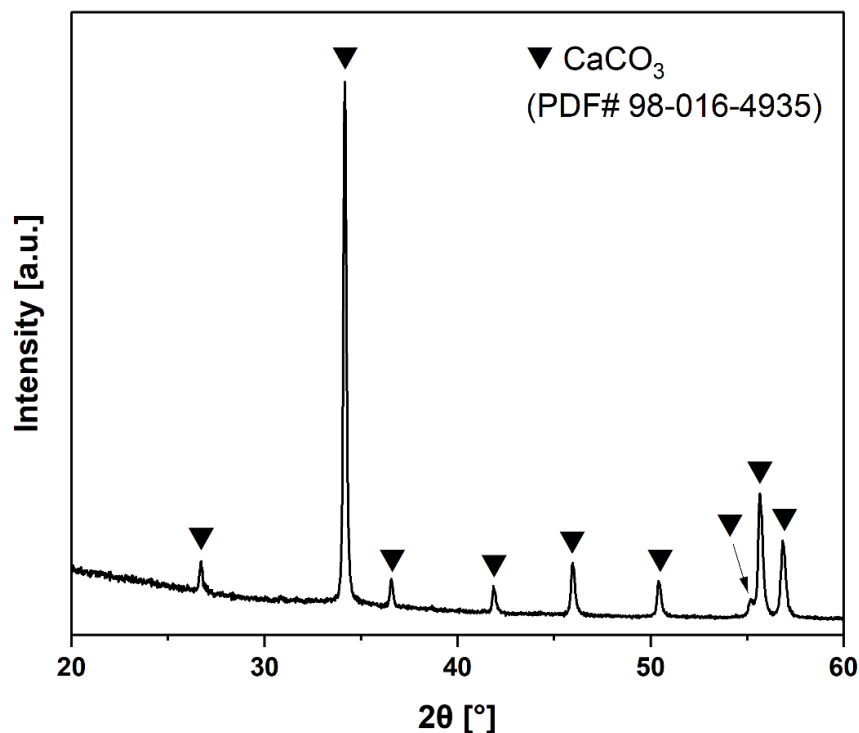


Figure 6. PXRD pattern of the solid precipitate obtained after electrochemical oxidation. All of the recorded diffraction peaks can be attributed to trigonal CaCO_3 (PDF# 98-016-4935). The diffraction pattern was recorded at 40 mA and 40 kV using a cobalt X-ray source ($\lambda = 1.78901 \text{ \AA}$).

A more detailed analysis of the process water composition suggests that several inorganic species are responsible for the discrepancy between TOC and COD. The ICP-OES results (Table 3) reveal the presence of phosphorus and sulphur in reduced forms, which were oxidized during electrochemical oxidation. For phosphorus these could be the intermediate products of the hydrolysis of the conducting salt phosphoryl fluoride, difluorophosphoric acid or monofluorophosphate, and for sulfur, these could be sulphites or thiosulfates. They act as reductants in the dichromate-based COD test. These compounds were therefore detected as ‘oxygen demand’, despite not containing organic carbon. Additionally, carbonate equilibria play a role: the precipitation of CaCO_3 observed during electrochemical oxidation suggests that some of the inorganic carbon was mobilized under the acidic digestion conditions of the COD test, in which carbonate and bicarbonate species could be partially oxidized. These inorganic contributions explain why COD removal stagnated at around 70% while TOC was almost completely degraded. Consequently, TOC appears to be the more reliable parameter for monitoring the organic load of LIB recycling wastewater.

In summary, it can be said that all three factors likely explain the discrepancy between the measured COD and TOC values. However, the precipitation of inorganic compounds had the greatest influence. In this case, considerable energy is consumed in carbonate precipitation. In addition to the organic load, the process water was also contaminated with the anions of the conducting salt from the electrolyte of the LIBs. A byproduct of this work is the finding that less than 4% of the conducting salt anions were converted to fluoride or phosphate in the specified time frame for both UV/ H_2O_2 oxidation and electrochemical

oxidation. Therefore, an oxidative solution to hydrolyze the conducting salts in wastewater was ruled out due to the very high energy requirement. Muschket et al. have shown that the anions of the conducting salt were not retained in a municipal wastewater treatment plant and were released into the environment [44].

Economic Analysis

The following section will clarify how the costs of the three AOPs behave when the processes are scaled up. The approach by Weyand et al. was chosen for upscaling [37]. This follows a three-step system:

Upscaling definition:

Upscaling was carried out based on the results obtained in the laboratory. The aim of the upscaling was to treat 16.7 m³/d of wastewater. The technology was scaled up from technology readiness level 4 to 8 to reach the limit values of the sewer system.

Upscaling leap:

The process was scaled up in consultation with UV oxidation system manufacturers. Since no system with the same ratio of lamp surface area to wastewater volume was available, it was assumed that reaction times might need adjustment. For the economic analysis, it was assumed that the reaction time corresponds to that in the experiments. The chemical requirements of the Fenton process were scaled linearly with the treated volume. A reaction time of 2 h was assumed. For electrochemical oxidation, the ratio of BDD surface area to wastewater volume was kept the same as in the tests.

Upscaling Model and Data:

The upscaling model and data can be found in the upscaling Excel sheet in Appendix A.

Figure 7 compares the LTCs of the three AOPs for treating 1 m³ of wastewater from LIB recycling in order to achieve the limit value for discharge into the sewer system. A distinction is made between investment costs, highlighted in different shades of blue, with the primary investment, re-investments over the operation time, land use with housing and operating costs, compared to the cost of needed chemicals, cost of electricity, discharging fees and waste disposal fees, which are highlighted in different shades of red.

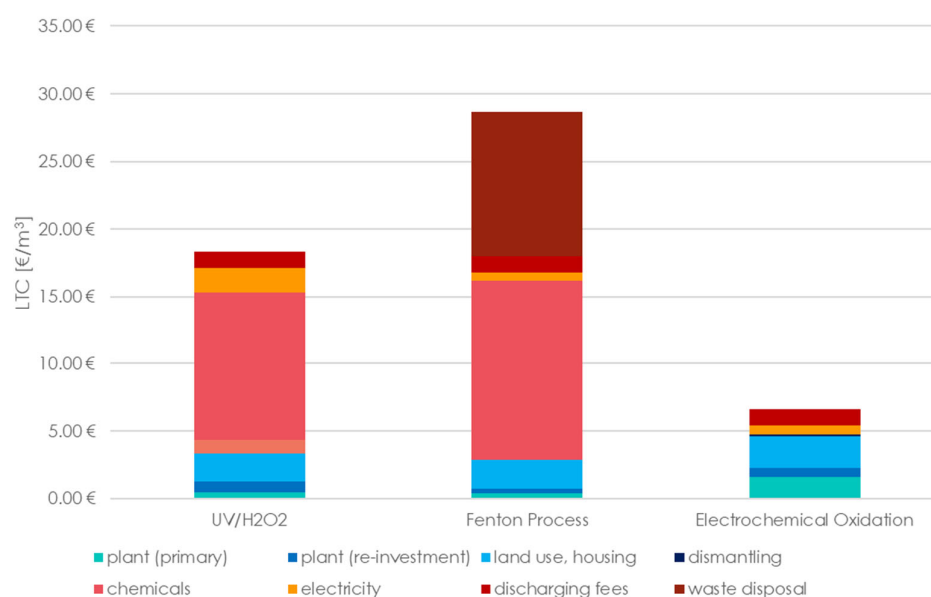


Figure 7. Levelized treatment costs of UV/H₂O₂ oxidation, the Fenton process and electrochemical oxidation.

In comparison, the Fenton process had the highest costs at EUR 28.66/m³, while electrochemical oxidation was the most cost-effective at EUR 6.63/m³. For UV/H₂O₂ oxidation (EUR 17.31/m³) and the Fenton process, chemical consumption was the primary cost driver. The Fenton process also incurs high disposal costs due to the inorganic sludge produced. Although electrochemical oxidation involved higher investment costs than the other two processes, this was of minor relevance over the plant's lifetime. In order to discharge the wastewater into the sewer system, an additional treatment step would have to be installed to ensure compliance with the fluoride limit and total phosphorus limit. Therefore, the economic analysis can only provide a comparison of the three AOPs and no absolute conclusions can be drawn. A further analysis including fluoride and total phosphorus removal would be required. Currently, the wastewater must be disposed of by a waste management company in a chemical–physical treatment plant at a cost of EUR 661/m³. A comparison of electrochemical oxidation for compliance with the limit values for discharge into the sewer system and direct discharge into the receiving water body can be found in Appendix A (Figure A4).

4. Conclusions

This study systematically evaluated three advanced oxidation processes, UV/H₂O₂ oxidation, the Fenton process and electrochemical oxidation, for the treatment of wastewater generated during lithium-ion battery recycling. Owing to the unfavorable BOD₅/COD ratio and the predominance of degradation products over intact organic carbonates, neither biological treatment nor solvent recovery represents a feasible treatment strategy for this type of wastewater.

Both UV/H₂O₂ oxidation and the Fenton process achieved compliance only with the sewer discharge limit and were characterized by high oxidant consumption and elevated energy or secondary treatment requirements compared to the data in the literature for other industrial wastewaters. In contrast, electrochemical oxidation was the only process capable of meeting both the sewer and direct discharge limits, while operating without chemical additives and at a lower energy demand for sewer discharge.

The economic evaluation confirmed these findings, identifying electrochemical oxidation as the most cost-effective treatment option. Taken together, the results demonstrate that electrochemical oxidation provides a technically robust, economically favorable and environmentally advantageous solution for the treatment of wastewater from LIB recycling.

5. Outlook

Further research should focus on validating the scalability of the proposed wastewater treatment concept by long-term stability tests prior to pilot-scale implementation. Process optimization, such as the integration of membrane processes to enable permeate recirculation and concentrate treatment, could substantially reduce the water footprint. While this study addresses the removal of the organic load, LIB recycling wastewater also contains high concentrations of inorganic contaminants. Therefore, future treatment concepts should simultaneously consider organic degradation, inorganic contaminant control and resource recovery, while ensuring compliance with regulatory discharge limits. Based on the present and previous findings, the introduction of TOC as a sum parameter and total fluorine as a monitoring parameter is recommended to improve environmental protection for wastewater from LIB recycling.

6. Patents

Patent pending Deutsche Patentanmeldung 10 2024 204 322.2

“Verfahren und Anlage zur Abreicherung von oxidierbaren organischen Substanzen und Anionen in Prozesswasser”.

Author Contributions: Conceptualization, R.W.-W.; methodology, R.W.-W. and R.P.; investigation, R.W.-W., F.F., R.P. and T.N.; resources, F.B.; writing—original draft preparation, R.W.-W. and F.F.; writing—review and editing, R.W.-W., F.F. and T.N.; visualization.; supervision, M.P., M.E., A.W. and E.I.; project administration, R.W.-W.; funding acquisition, A.W. and E.I. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

AOPs	Advanced Oxidation Processes
BDD	Boron-Doped Diamond
BOD ₅	Biological Oxygen Demand (5 days)
CAPEX	Capital Expenditures
CIC	Combustion Ion Chromatography
COD	Chemical Oxygen Demand
DEC	Diethyl Carbonate
DMC	Dimethyl Carbonate
EC	Ethylene Carbonate
EDX	Energy-Dispersive X-Ray Spectroscopy
EHF	Electrohydraulic Fragmentation
EMC	Ethyl Methyl Carbonate
EU	European Union
Fe ²⁺	Ferrous Ion
HF	Hydrofluoric Acid
HPLC	High-Performance Liquid Chromatography
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
ICSD	Inorganic Crystal Structure Database
IE	Ion Exchange
KPF ₆	Potassium Hexafluorophosphate
LIB	Lithium-Ion Battery
LTC	Levelized Treatment Cost
MBR	Membrane Bioreactor
NMC	Nickel Manganese Cobalt Oxide
NPOC	Non-Purgeable Organic Carbon
OPEX	Operational Expenditures
PC	Propylene Carbonate
PF ₆ [−]	Hexafluorophosphate
PXRD	Powder X-Ray Diffraction
SI	Supporting Information
SuSi	Skimming and Sieving

TOC Total Organic Carbon
 UV Ultraviolet

Appendix A

Table A1. Composition of the process water using cuvette test, HPIC and ICP-OES. The table has been adapted for this publication [7].

Parameters		Tab Water	Process Water
Volume [L]	[L]	-	30–42
pH value	[-]	6.8	7.1–7.4
Conductivity	[$\mu\text{S}/\text{cm}$]	400	1280–2280
Chemical oxygen demand (COD)	[mg/L]	-	916–1570
Biological oxygen demand (BOD ₅)	[mg/L]	-	235–340
COD/BOD ₅ ratio	[-]	-	3.9–4.6
Al	[mg/L]	0.03	0.31–0.56
Ca	[mg/L]	67.3	26.2–35
Co	[mg/L]	-	0.08–0.12
Cu	[mg/L]	0.17	0.12–0.59
Li	[mg/L]	0.19	127–157
Mg	[mg/L]	13.2	6.0–7.3
Mn	[mg/L]	-	0.08–0.20
Na	[mg/L]	1.76	2.8–40.5
Ni	[mg/L]	-	0.36–0.60
P	[mg/L]	0.94	61.2–84.6
S	[mg/L]	13.7	165–200
F _{total}	[mg/L]	-	123–468
F ⁻	[mg/L]	-	29.6–38.3
Cl ⁻	[mg/L]	40.7	46.4–51.2
SO ₄ ²⁻	[mg/L]	42.5	39.1–45.2
PO ₄ ³⁻	[mg/L]	-	-

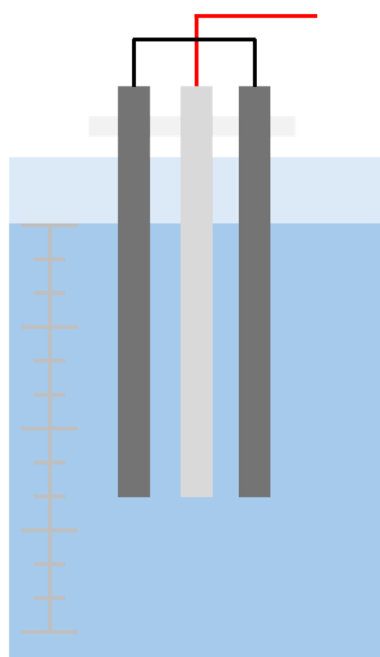


Figure A1. Schematic illustration of the experimental setup of the electrochemical oxidation (dark grey: steel electrode, light grey: boron-doped diamond electrode).

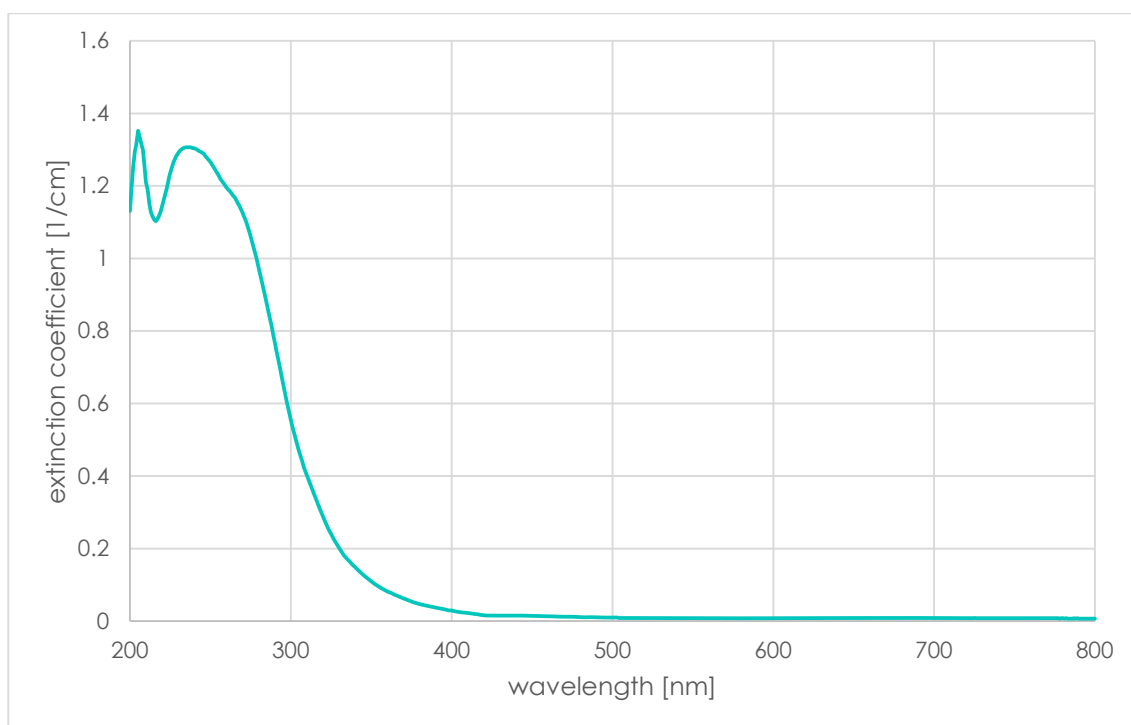
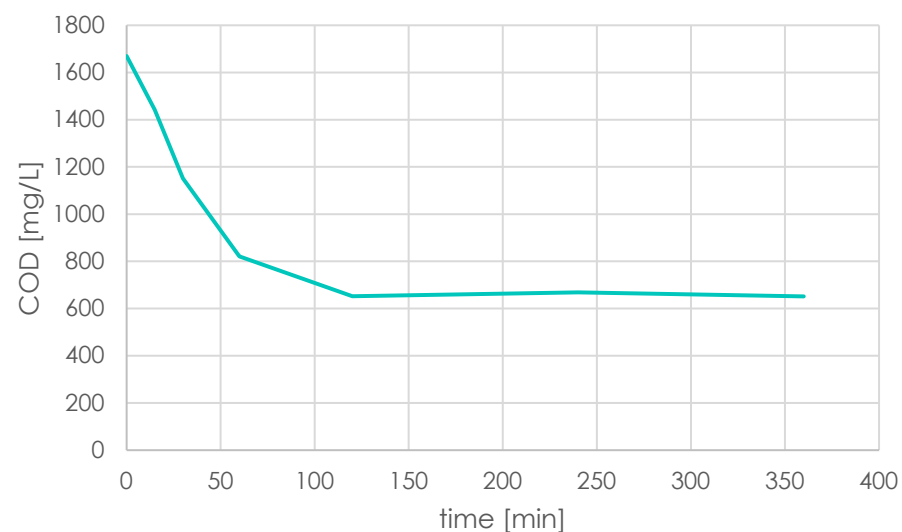


Figure A2. Extinction spectrum in the wavelength range between 200 and 800 nm of the process water.

Table A2. Fixed asset service life, primary investment demand, reinvestment demand and costs per item or per m² [45–48].

Fixed Assets	Service Life	Primary Investment Demand	Reinvestment Demand	Costs Per Item
Wastewater treatment plant	20 years	1	0	-
Housing	33 years	0.6	0	EUR 2827/m ² in Hanau, Germany
Pumps	7 years	3	5.6	EUR 6477 acid-resistant centrifugal pump EUR 3450 centrifugal pump EUR 6900 screw pump
Tank	20 years	2	0	EUR 3500
UV oxidation plant	7 Years	1	1.9	EUR 49,950
Anodes	6.3 years	1.9 m ²	4.1 m ²	EUR 19,993/m ²
Cathodes	10 years	1.9 m ²	3.8 m ²	EUR 994/m ²
Mixer	14 years	1	0.43	EUR 168

**Figure A3.** Fenton process COD removal over time.**Table A3.** Process parameters of advanced oxidation processes from the literature.

UV/H ₂ O ₂ Oxidation	COD [mg/L]	COD _{removed} [%]	H ₂ O ₂ /COD [kg/kg]	E/COD [kWh/kg]	Treated Volume [mL]	Source
Battery wastewater	1632	51	7.8	56.3	500	this work
Hospital laundry wastewater	6364	31.4	-	38.6	250	[9]
Textile wastewater	397	86.7	0.0025	-	300	[10]
Oil recovery	21,000	39	2	-	-	[11]

Table A3. Cont.

UV/H ₂ O ₂ Oxidation	COD [mg/L]	COD _{removed} [%]	H ₂ O ₂ /COD [kg/kg]	E/COD [kWh/kg]	Treated Volume [mL]	Source
Wastewater from the production of pharmaceuticals	1580	84.6	0.32	-	300	[12]
Petrochemical wastewater	950	52.6	0.6	131.1	250	[13]
Fenton process	COD [mg/L]	COD _{removed} [%]	H ₂ O ₂ /COD _{removed} [kg/kg]	H ₂ O ₂ /Fe ²⁺ [kg/kg]	Treated volume [mL]	Source
Battery wastewater	1632	51	8.06	2.52	50	this work
Pulp production	3756	32	1.19	0.32	250	[15]
Petro chemistry	436	55	0.96	2.22	500	[16]
	-	61	2.00	3.51	500	
	11,500	97.5	-	50	150	[17]
Carpet dyeing	2576	Up to 95		95–290	100	[18]
Olive oil wastewater	60,500	70	1.75	15	500	[19]
Livestock	5000–5700	88	1.05	5	100	[20]
Cork-cooking	5000	87.3		5	750	[21]
Anodic Oxidation	COD mg/L	COD _{removed} [%]	E/COD [kWh/kg]	Current density [mA/cm ²]	Treated volume [mL]	Source
Battery wastewater	1632	51	32.8	25	200 mL	this work
	1632	91.7	95.3	50	200 mL	
Textile wastewater	729	100	11.12	60	10,000	[23]
Landfill water	1050	83.1 (TOC removal)	22.1	-	100	[24]
Wastewater containing phenol	633	77.1	31	20	4501	[25]
Vinasse	-	43	17	6.6	150	[26]
Wastewater from wine production	3490	100	27.5	60	200	[27]
Wastewater containing phenol	540	75.9	84.8	-	4501	[25]
Hydrothermal carbonization process	28,050 (PW-AD)	95	20.2	66	-	[28]
Hydrothermal carbonization process Post-hydrothermal liquefaction	11,233 (MBR 1-1)	-	22.1	66 30	60	[29]
	11,233 (MBR 1-1)	-	46.6			
	675 (MBR 1-10)	41	241			
	675 (MBR 1-10) 36,000	99 99	534 28			
Aqueous phase of thermo-catalytic reforming of sewage sludge	-	95	39–58.3	50	30	[30]

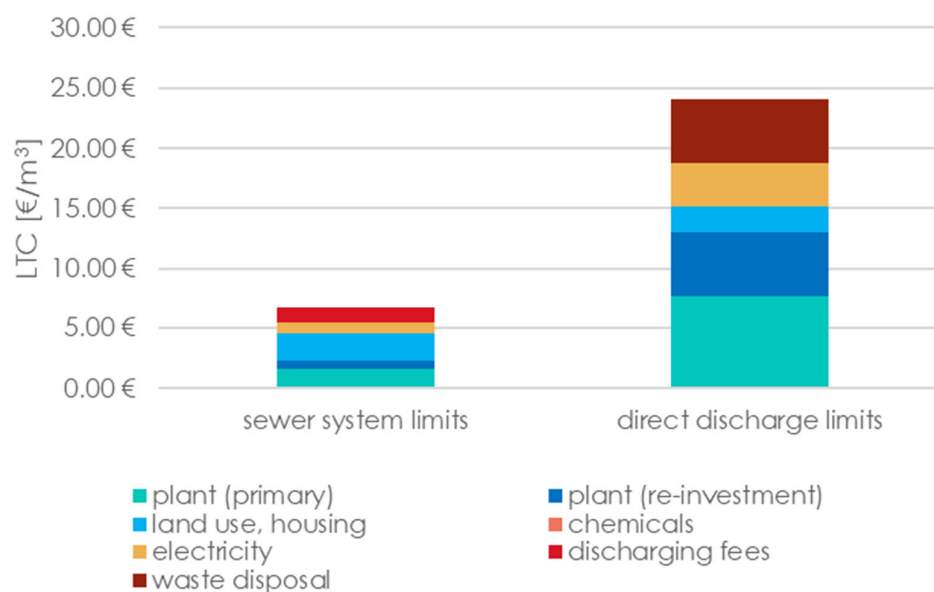


Figure A4. Comparison of the electrochemical wastewater treatments required to reach the limit values for discharge into the sewer system and direct discharge.

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