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Low-Resistance GO–POM Composite Cathode and Asymmetric Geometry Reduce Energy Consumption by 37% in Electrocoagulation of Hypereutrophic Lake Wastewater

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Abstract

Electrocoagulation (EC) is a promising technology for hypereutrophic wastewater treatment, yet reactor geometry, electrode passivation, and energy inefficiency remain key limitations. This study developed a graphene oxide–phosphomolybdate (GO–POM) composite cathode integrated into an asymmetric electrode configuration (anode:cathode area ratio = 1:10) to simultaneously address charge-transfer resistance, passivation, and energy consumption in the EC treatment of Lake Mariut wastewater (Cairo, Egypt). The GO–POM composite exhibited a charge-transfer resistance of $2.34 \pm 0.09 \Omega \cdot \text{cm}^2$, significantly lower than that of a graphite rod ($4.12 \pm 0.31 \Omega \cdot \text{cm}^2$), carbon felt ($3.28 \pm 0.24 \Omega \cdot \text{cm}^2$), and SS316 ($6.84 \pm 0.45 \Omega \cdot \text{cm}^2$). Under optimized conditions ($j = 10 \text{ mA/cm}^2$, pH 6.0, 60 min), the asymmetric GO–POM system achieved $92.2 \pm 1.8\%$ TOC removal with a specific energy consumption of $4.4 \pm 0.3 \text{ kWh/m}^3$ —a 37% reduction compared to the symmetric conventional baseline ($6.1 \pm 0.4 \text{ kWh/m}^3$). The treated effluent met the discharge limits set by Egyptian Law 48/1982 for COD, BOD, and TSS. Preliminary techno-economic and life-cycle analyses identified Al electrode consumption as the dominant cost and carbon driver, with a solar-powered continuous-flow operation as the priority pathway for further energy reduction.

Keywords: graphene oxide–polyoxometalate composite cathode; asymmetric electrode geometry; energy-efficient electrocoagulation; Lake Mariut wastewater; desalination pretreatment; life-cycle assessment; electrochlorination; aluminum anode dissolution kinetics

1. Introduction

Rapid population growth, intensive agricultural activity, and unregulated industrial discharge have accelerated the eutrophication of freshwater bodies across arid and semi-arid regions, producing hypereutrophic water matrices characterized by high organic loading, elevated suspended solids, and complex recalcitrant pollutant mixtures [1–3]. Lake Mariut (Alexandria–Cairo corridor, Egypt) exemplifies this challenge, receiving mixed domestic, agricultural, and industrial effluents that render conventional biological treatment insufficient as a standalone technology [4–6]. Electrocoagulation (EC) has emerged as a physicochemical alternative capable of treating such complex matrices through the in situ electrochemical generation of coagulant species (Al^{3+} and $\text{Al}(\text{OH})_n$ from aluminum anodes) combined with hydrogen microbubble flotation and electro-oxidation pathways at the cathode surface [7–9]. EC operates without chemical dosing, produces compact sludge, and is adaptable for decentralized deployment, advantages that have motivated substantial research interest over the past two decades [10].



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Despite these merits, critical performance limitations constrain the practical implementation of EC systems. Current reviews consistently identify four key barriers requiring further optimization: (i) reactor geometry and electrode arrangement, which govern current distribution uniformity and coagulant dose efficiency; (ii) anodic passivation, whereby oxide film formation on aluminum electrodes increases cell voltage and reduces Al dissolution efficiency over time [4,11,12]; (iii) cathodic charge-transfer resistance, which elevates specific energy consumption (SEC, kWh/m³), particularly in low-conductivity matrices; and (iv) the energy inefficiency of symmetric electrode configurations, in which equal anode and cathode areas impose unnecessarily high cathodic current densities that do not contribute proportionally to treatment performance [13]. Two further operational constraints are critical for real-world deployment but remain underexplored in the literature: (v) reliance on grid electricity, which can be substantially mitigated by solar-powered EC systems [13] demonstrated at pilot scale and (vi) a predominance of batch configurations, whereas continuous-flow EC reactors are essential for scalable, cost-effective implementation. Addressing limitations (i)–(iv) at the laboratory scale and identifying the pathway to (v) and (vi) is the focus of the present study.

Two specific innovations are proposed to address limitations (i), (iii), and (iv) simultaneously. First, a graphene oxide–phosphomolybdate (GO–POM) composite cathode was engineered by immobilizing Keggin-type H₃PMo₁₂O₄₀ polyoxometalate on a graphene oxide matrix within a PVDF-bound pellet electrode [14–17]. GO provides a high-surface-area, electrically conductive scaffold that maximizes the electrochemically accessible double-layer capacitance [18], while the POM Keggin unit acts as a redox mediator at the cathode surface, hypothesized to reduce the activation barrier for electron transfer [14,15,19] and thereby lower charge-transfer resistance (R_{ct}) [20,21]. Second, an asymmetric electrode configuration (anode:cathode geometric area ratio = 1:10) was adopted to decouple anode current density, which controls the Al dissolution rate and coagulant dose (Faraday’s law), from cathode current density, which governs H₂ bubble generation and cathodic energy dissipation. By distributing the cathodic current over a tenfold-larger electrode area, the asymmetric geometry reduces the cathodic overpotential and, consequently, the total cell voltage and SEC without altering the Al coagulant dose [11,22].

1.1. Limitations of Traditional EC—Research Gaps

Conventional EC has seen limited mainstream adoption due to persistent challenges in electrode passivation [23], high specific energy consumption (SEC), and inconsistent performance across wastewater matrices [20]. Reported SEC values vary widely in the literature—from under 4.5 kWh/m³ to as high as 360 kWh/m³ for complex matrices—underscoring the need for continuous optimization.

Four categories of recent EC advances are directly relevant and are evaluated against the present approach:

- Pulsed-current EC (PC-EC) mitigates anode passivation and reduces SEC by ~24% but targets anode-side dissolution kinetics only, not cathode-side charge-transfer resistance;
- Bipolar electrode configurations improve current distribution but sacrifice independent anode–cathode geometric optimization;
- Flow-through reactors reduce dead zones but do not address the electrode-material or geometry variables studied here (acknowledged as a limitation of this batch design);
- CNT–metal oxide composite cathodes achieve low R_{ct} but suffer from high fabrication costs, poor CNT scalability, and ecotoxicity concerns.

Against this landscape, the GO–POM composite cathode occupies a specific niche: it targets cathode-side charge-transfer resistance using low-cost, earth-abundant materials,

while the asymmetric geometry provides a complementary (not competing) lever to PC-EC anode-side optimization.

1.2. Objectives and Scope

Two persistent gaps remain insufficiently resolved: (i) the influence of asymmetric electrode surface-area configurations on energy consumption and passivation and (ii) the role of functionalized cathode materials in enhancing removal mechanisms. This study addresses both by evaluating a modified EC reactor with (1) an asymmetric anode-to-cathode surface-area ratio of 1:10 and (2) a composite cathode of carbon black, natural zeolite, and GO-POM.

Specific objectives:

1. To quantify the effect of an asymmetric electrode surface area on organic matter removal efficiency and SEC;
2. To examine electrochemical behavior and surface characteristics to elucidate performance-enhancement mechanisms;
3. To benchmark energy efficiency and treatment outcomes against comparable laboratory-scale EC systems.

Research questions:

- RQ1: At constant total current, does increasing the cathode-to-anode area ratio (reducing cathodic current density) lower SEC and cell voltage while maintaining equivalent organic matter removal? (This isolates geometric cathode effects from coagulant-dose effects since Faraday's law fixes Al generation when the anode area/current density are held constant.)
- RQ2: Does the carbon/zeolite/GO-POM composite cathode improve electrochemical performance relative to conventional EC cathodes?
- RQ3: How do electrode surface characteristics and electrochemical behavior evolve during operation under the modified configuration?

Scope boundary: This is a proof-of-concept, laboratory batch-scale investigation. TEA/LCA projections are derived by linear extrapolation from 1 L batch data and carry substantial uncertainty; durability is assessed under an idealized inter-cycle rinsing/drying protocol supplemented by 24 h continuous chronopotentiometry; Al anode consumption under DC-galvanostatic operation ($0.29 \pm 0.04 \text{ kg/m}^3$) represents an upper-bound baseline. Pulsed-current optimization is identified as the primary lever for future economic and environmental improvement. Durability and economic projections beyond the experimentally validated 10-cycle lifetime explicitly require pilot-scale validation.

2. Materials and Methods

2.1. Wastewater Source and Influent Characterization

Wastewater was collected from Lake Mariut ($30^{\circ}52' \text{ N}$, $29^{\circ}44' \text{ E}$, Alexandria–Cairo corridor, Egypt), a hypereutrophic, closed lake that receives mixed domestic, agricultural, and industrial effluents. Samples were collected in pre-cleaned 20 L polyethylene containers, transported on ice to the laboratory within 2 h, and stored at 4° C for no more than 24 h prior to use. All experiments were conducted on freshly collected, unfiltered influent unless otherwise stated.

The physicochemical characteristics of the influent are summarized in Table 1. Key parameters include high organic loading (TOC $326 \pm 15 \text{ mg/L}$; COD $420 \pm 20 \text{ mg/L}$; BOD₅ $220 \pm 10 \text{ mg/L}$), elevated suspended solids (TSS $350 \pm 15 \text{ mg/L}$), near-neutral pH (7.8 ± 0.1), and moderate electrical conductivity ($2.1 \pm 0.1 \text{ mS/cm}$), consistent with a hypereutrophic brackish matrix. The COD/TOC ratio of 1.29 reflects a wastewater

dominated by partially oxidized, nitrogen- and hydroxyl-bearing organic compounds (humic/fulvic acids, algal metabolites) rather than simple hydrocarbons; this is consistent with published COD/TOC ratios of 1.1–2.0 for eutrophic lake waters and is discussed further in Section 3.1. All values are mean $\pm$ SD ($n = 3$ independent samples). All EC experiments used 0.1 M NaCl as a supporting electrolyte to ensure stable galvanostatic operation. This chloride concentration substantially exceeds the natural Cl^- level in the influent; therefore, the electrochlorination results (Section 3.4) represent upper-bound estimates of chlorination potential under the experimental conditions (further discussion: SI Section 2.6).

Table 1. Influent wastewater characteristics used in EC experiments (mean $\pm$ standard deviation, $n = 3$).

Parameter	Unit	Influent
Total organic carbon (TOC)	mg/L	326 $\pm$ 15
Chemical oxygen demand (COD)	mg/L	420 $\pm$ 20
Biochemical oxygen demand (BOD)	mg/L	220 $\pm$ 10
Total suspended solids (TSS)	mg/L	350 $\pm$ 15
pH	—	7.8 $\pm$ 0.1
Temperature	$^{\circ}\text{C}$	22 $\pm$ 1
Electrical conductivity	mS/cm	2.1 $\pm$ 0.1
Total nitrogen (TN)	mg/L	34 $\pm$ 2
Total phosphorus (TP)	mg/L	4.5 $\pm$ 0.3

Notes: Values are mean $\pm$ SD, $n = 3$. Analytical methods follow APHA Standard Methods. Method detection limits: TOC 0.5 mg/L; COD 5 mg/L; BOD 2 mg/L; TSS 2 mg/L; TN 0.5 mg/L; TP 0.05 mg/L; conductivity 0.01 mS/cm. An NaCl supporting electrolyte (0.1 M, 5850 mg/L Cl^-) was added to all EC experiments; this addition is not reflected in Table 1, which reports the natural influent composition prior to electrolyte supplementation.

2.2. GO–POM Composite Cathode Fabrication

2.2.1. Synthesis of Graphene Oxide (GO)

GO was synthesized from natural graphite flakes (99.9% purity, Sigma-Aldrich, St. Louis, MO, USA, $\geq 150 \mu\text{m}$) by a modified Hummers' method [18]. Briefly, graphite was oxidized in a 9:1 concentrated $\text{H}_2\text{SO}_4/\text{H}_3\text{PO}_4$ mixture (360:40 mL) with KMnO_4 (99%, 18.0 g), stirred at 50 $^{\circ}\text{C}$ for 12 h, quenched with ice and H_2O_2 , washed to pH 6–7, and exfoliated by ultrasonication to yield a stable GO dispersion (2 mg/mL). The characterization results (XPS C/O ratio = 2.45 ± 0.12 ; $I_D/I_G = 0.98 \pm 0.05$; BET = $85 \pm 6 \text{ m}^2/\text{g}$) are presented in Section 3.1 and SI Section 2.1.

2.2.2. Preparation of Keggin-Type POM ($\text{H}_3\text{PMo}_{12}\text{O}_{40}$)

Phosphomolybdic acid ($\text{H}_3\text{PMo}_{12}\text{O}_{40} \cdot x\text{H}_2\text{O}$, PMA; Sigma-Aldrich, 99.9%) was used as received or prepared in-house by acidifying aqueous ammonium molybdate with H_3PO_4 (85%), refluxing at 90–100 $^{\circ}\text{C}$ for 2 h, filtering, and drying under vacuum at 60 $^{\circ}\text{C}$ for 12 h. Keggin-structure identity was confirmed by FTIR (fingerprint bands at 900–1100 cm^{-1}). The Keggin structure is stable under the acidic operating conditions of this study (pH 6.0); thermodynamic and kinetic details of the pH stability constraint are provided in SI Section 2.2. Mo leaching across the 10-cycle protocol was measured directly (SI Section 2.7, Table S7).

2.2.3. GO–POM Composite Preparation and Electrode Fabrication

GO–POM composites were prepared by mixing the GO dispersion (2 mg/mL) with an aqueous PMA solution (10 mM) under sonication to promote electrostatic immobi-

lization of Keggin units on protonated GO edge sites. The recovered GO–POM powder was combined with conductive carbon black and natural zeolite (clinoptilolite) to give a composite with dry-basis mass fractions of 56 wt% carbon, 30 wt% zeolite, and 14 wt% GO–POM. Electrodes ($\varnothing$ 20 mm $\times$ 5 mm) were uniaxially pressed at 10 MPa, dried at 80 °C under vacuum for 12 h, and thermally consolidated at 120 °C for 2 h using a PVDF binder (8 wt%; full fabrication protocol: SI Section 2.1). Composite electrical conductivity (four-point probe) was 8.5 ± 0.7 S/cm ($n = 3$). ECSA = 141 ± 9 cm² (roughness factor $\approx$ 45); BET surface area = 85 ± 6 m²/g; open porosity = $39.4 \pm 2.8\%$ (full surface-area determination: SI Section 2.2).

Prior to use, all electrodes were conditioned by pre-electrolysis at 5 mA/cm² in 0.1 M Na₂SO₄ for 15 min, followed by 10 min open-circuit equilibration (full conditioning protocol: SI Section 2.3).

2.3. Material Characterization

GO, POM, and the GO–POM composite were characterized by the following techniques; full acquisition parameters, instrument models, and data-processing details are provided in Figure 1 and SI Section 2.4.

- XPS: Monochromatic Al K α source ($h\nu = 1486.6$ eV); survey and high-resolution spectra (C 1s, O 1s, Mo 3d, P 2p) acquired from $n = 3$ independently prepared samples; Shirley background subtraction and GL(30) peak fitting (CasaXPS v2.3.x); binding energies referenced to adventitious C 1s at 284.8 eV.
- XRD: Cu K α radiation; 2θ range 5–60°; step size 0.02°; GO (001) reflection, Keggin-phase reflections, and composite diffractograms are reported in (Section 3.1).
- SEM/TEM: Morphology and POM nanoparticle distribution on the GO surface (Figure S3).
- FTIR: Keggin fingerprint bands (900–1100 cm^{−1}) and GO oxygen-functional-group assignments (Figure S1).
- Raman spectroscopy: I_D/I_G ratio for GO and composite.
- BET/BJH: N₂ adsorption–desorption at 77 K; Type IV isotherm with H3 hysteresis confirming mesopore-dominant architecture (SI Section 2.2).
- ICP-OES: Residual dissolved Mo and P in effluent after the 10-cycle protocol (SI Table S7).

2.4. Electrochemical Characterization

Electrochemical impedance spectroscopy (EIS) was performed in a standard three-electrode configuration (working: composite cathode; counter: Pt mesh; reference: Ag/AgCl, 3 M KCl) over 100 kHz–0.01 Hz (AC perturbation: 10 mV rms at OCP). Data were fitted to a Randles–CPE equivalent circuit ($Z = R_s + [R_{ct} \parallel CPE]$) using ZView software; the goodness-of-fit criterion was $\chi^2 < 5 \times 10^{-3}$. All R_{ct} values are normalized to the geometric area (A_{geo}) in units of $\Omega \cdot \text{cm}^2_{geo}$ (full EIS protocol and circuit derivation: SI Section 2.4). Cyclic voltammetry (CV) was conducted in 0.1 M Na₂SO₄ (scan rates: 5–80 mV/s) for ECSA estimation (SI Section 2.4).

2.5. Electrocoagulation Reactor and Operating Conditions

EC experiments were conducted in a batch Plexiglas reactor (working volume 1.0 L) with parallel vertical electrodes (inter-electrode spacing: 20 mm). The anode was an aluminum plate (purity 99.5%) with immersed dimensions of 10.0 cm (height) $\times$ 4.6 cm (width) $\times$ 0.3 cm (thickness); both faces of the plate were exposed to the electrolyte, yielding a total geometric anode area of $A_{geo} = 2 \times (10.0 \text{ cm} \times 4.6 \text{ cm}) = 92 \text{ cm}^2$.

The cathode consisted of an array of ten cylindrical GO–POM composite rods (each fabricated on a graphite rod core coated with the GO–POM/carbon black/zeolite composite, as described in Section 2.2.3) arranged in parallel around the anode. Each rod had a diameter of 2.0 cm and an immersed length of 14.6 cm, giving a cylindrical surface area per rod of $\pi \times d \times L \approx 92 \text{ cm}^2$. The ten rods were electrically connected in parallel, yielding a total cathode geometric area of $A_{geo} = 10 \times 92 \text{ cm}^2 = 920 \text{ cm}^2$ and an asymmetric anode-to-cathode area ratio of 1:10 (see Figure 1). For symmetric-geometry control experiments, the cathode array was reduced to a single composite rod ($A_{geo} = 92 \text{ cm}^2$, 1:1 ratio), while the anode dimensions, anode area, and total applied current were held constant, ensuring identical Al dissolution rates across configurations (Faradaic verification, Section 2.6.1).

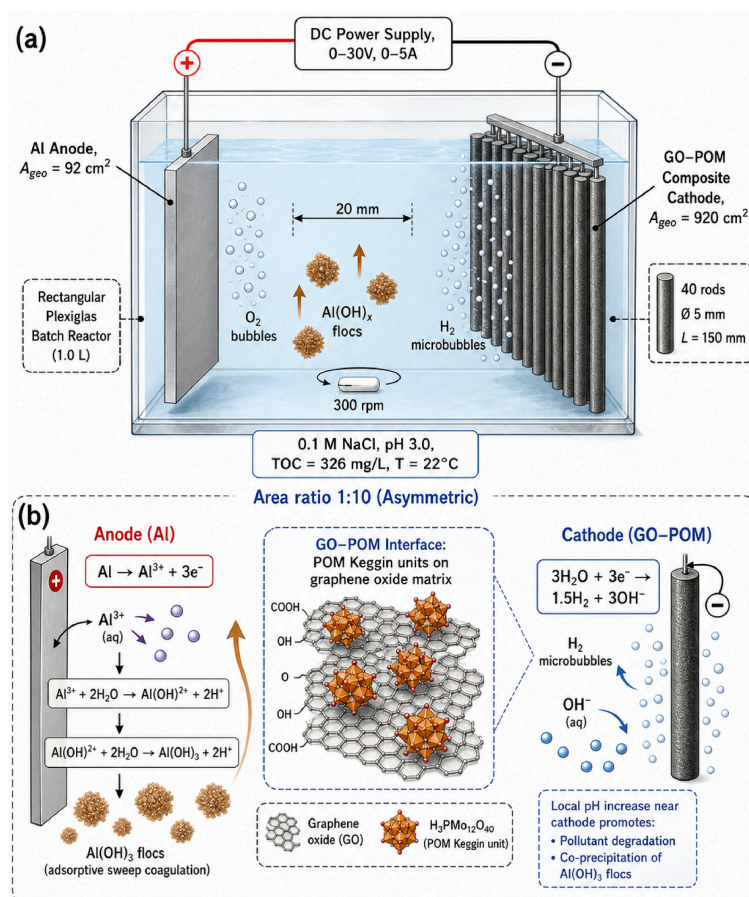


Figure 1. Schematic of the electrocoagulation (EC) reactor and asymmetric electrode configuration. (a) Batch Plexiglas reactor (working volume 1.0 L) showing the parallel vertical electrode arrangement: a rectangular aluminum anode (10.0 cm × 4.6 cm, $A_{geo} = 92 \text{ cm}^2$) centered within an array of ten cylindrical GO–POM composite cathode rods (2.0 cm diameter × 14.6 cm immersed length each, $A_{geo} = 92 \text{ cm}^2$ per rod; total array $A_{geo} = 920 \text{ cm}^2$), connected in parallel and positioned at an inter-electrode spacing of 20 mm, yielding the asymmetric anode-to-cathode area ratio of 1:10 used in all experiments unless otherwise stated. (b) Cross-sectional schematic comparing the asymmetric (1:10) and symmetric (1:1) electrode geometries evaluated in this study, illustrating how cathodic current density is distributed over a ten-fold larger surface area in the asymmetric configuration (1 mA/cm²) relative to the symmetric control (10 mA/cm²) at constant total applied current ($I = 920 \text{ mA}$) and anode current density (10 mA/cm², anode basis).

The standard operating conditions were as follows: current density (anode basis) = 10 mA/cm² ($I = 920 \text{ mA}$); supporting electrolyte 0.1 M NaCl in Lake Mariut influent; initial pH 6.0 ± 0.1 (adjusted with 0.1 M HCl/1 M NaOH); temperature $22 \pm 1^\circ \text{C}$; stirring 300 rpm (magnetic); electrolysis time 60 min per batch run. All experiments were conducted in

triplicate ($n = 3$). Post-treatment dechlorination with Na_2SO_3 (10.8 mg/L, stoichiometric dose) was performed after each EC run prior to effluent characterization.

The standard operating conditions for all experiments were as follows:

Parameter	Value
Current density (anode basis)	10 mA/cm ² ; $I = 920$ mA
Supporting electrolyte	0.1 M NaCl in Lake Mariut influent
Initial pH	6.0 ± 0.1 (adjusted with 0.1 M HCl/1 M NaOH)
Temperature	22 ± 1 °C
Stirring	300 rpm (magnetic)
Electrolysis time	60 min per batch run

pH was adjusted to 6.0 to satisfy the Keggin-structure stability constraint (pH < 4 in free solution; stabilized to pH 5–7 by GO/PVDF heterogenization; full pH rationale: SI Section 2.2). All experiments were conducted in triplicate ($n = 3$). Post-treatment dichlorination with Na_2SO_3 (10.8 mg/L, stoichiometric dose) was performed after each EC run prior to effluent characterization (protocol: SI Section 2.6).

2.6. Performance Metrics

2.6.1. Specific Energy Consumption (SEC)

SEC (kWh/m³) was calculated as follows:

$$\text{SEC} = \frac{\int_0^t V(t) \cdot I(t) dt}{V_{\text{treated}}} \quad (1)$$

where $V(t)$ is the instantaneous cell voltage (V), $I(t)$ is the applied current (A), t is the electrolysis time (h), and V_{treated} is the treated volume (m³). Under galvanostatic operation, this simplifies to $\text{SEC} = \bar{V} \cdot I \cdot t / V_{\text{treated}}$, where $\bar{V}$ is the time-averaged cell voltage. SEC is normalized to the treated volume (m³) throughout, consistent with standard EC literature practice and the LCA functional unit (Section 2.8). All current densities and SEC values are normalized to the anode geometric area (A_{geo}).

2.6.2. TOC Removal Efficiency

TOC removal efficiency (η , %) was calculated as follows:

$$\eta = \frac{[\text{TOC}]_0 - [\text{TOC}]_t}{[\text{TOC}]_0} \times 100 \quad (2)$$

TOC was measured by high-temperature catalytic oxidation (Shimadzu Corporation, Kyoto, Japan, TOC-VCSH; LOD 0.5 mg/L). COD, BOD₅, and TSS were measured according to APHA Standard Methods.

2.6.3. Aluminum Dose and Current Efficiency

Anodic current efficiency (CE) for Al dissolution was determined gravimetrically (analytical balance, ±0.1 mg):

$$\text{CE}_{\text{Al}} = \frac{m_{\text{Al,measured}}}{m_{\text{Al,Faradaic}}} \times 100, m_{\text{Al,Faradaic}} = \frac{M_{\text{Al}} \cdot I \cdot t}{z \cdot F} \quad (3)$$

where $M_{Al} = 26.98$ g/mol, $z = 3$, $F = 96,485$ C/mol. Measured CE = $93 \pm 2\%$ ($n = 3$); this value is used as the base case in all TEA and LCA Al-consumption estimates.

2.7. Reference Cathode Materials

To benchmark the GO-POM composite, parallel EC experiments were conducted under identical conditions (Section 2.5) using three reference cathode materials:

- A graphite rod—natural graphite, 99.5% purity, Ø 20 mm (most widely used laboratory EC benchmark);
- Carbon felt—PAN-based, 10 mm thickness (high geometric surface area);
- Stainless steel 316 (SS316)—mesh form (corrosion-resistant metallic cathode; mesh specification: SI Table S1f).

All reference cathodes were used without surface modification. Electrode dimensions were set to maintain $A_{geo} = 920$ cm² for all cathode types in the asymmetric (1:10) configuration, ensuring an identical anode current density, total applied current, and Al dissolution rate across all four cathode types.

2.8. Electrode Stability Assessment

Cycling stability was assessed over 10 consecutive 60 min EC batch cycles using the same cathode (total electrolysis time: 10 h). Between cycles, electrodes were rinsed with deionized water and dried at 60 °C for 2 h (inter-cycle recovery protocol). TOC removal and average cell voltage were recorded per cycle. A separate 24 h continuous chronopotentiometry experiment (no inter-cycle recovery) was conducted to provide an upper-bound electrochemical degradation envelope; note that TOC removal was not measured during the 24 h continuous run—only cell voltage drift ($13.6 \pm 1.8\%$) and R_{ct} increase (23.9%) were monitored (full stability data and statistical analysis: SI Sections 2.4.1–2.4.3 and Tables S2b and S6).

Scope limitation: The inter-cycle drying step partially reverses surface fouling and does not replicate continuous submerged operation. The 10-cycle TOC removal results ($91.8\% \rightarrow 86.1\%$, $\Delta = 6.8\%$ relative) represent a laboratory upper bound on stability; continuous-operation durability remains unvalidated and is the primary experimental gap for future pilot-scale work.

2.9. Techno-Economic Analysis (TEA)

OPEX was estimated by linear extrapolation from 1 L batch data to a 1 m³ functional unit and includes (i) electricity (SEC $\times$ local tariff: USD 0.087/kWh); (ii) Al anode replacement (0.29 ± 0.04 kg Al/m³ $\times$ LME Al price); (iii) cathode amortization over an experimentally validated lifetime (10 cycles); (iv) a NaCl electrolyte; (v) pH-adjustment chemicals (HCl pre-acidification + NaOH post-neutralization, GOPOM system only: USD 0.10/m³); (vi) Na₂SO₃ dechlorination; and (vii) sludge disposal (USD 0.05/m³ dewatered sludge). CAPEX is excluded. All projections beyond the 10-cycle validated lifetime carry an explicit data-status designation (P = projected). The full cost model, assumptions, and break-even analysis are provided in SI Section S_TEA.

2.10. Life-Cycle Assessment (LCA)

The LCA was conducted in accordance with ISO 14040/14044 [24,25], adopting a cradle-to-grave system boundary. The functional unit is the treatment of 1.0 m³ of Lake Mariut hypereutrophic wastewater to 92% TOC removal (TOC removed: 0.300 kg/m³). The global warming potential (GWP, ReCiPe 2016 Midpoint H [26]) was the primary impact category. The foreground inventory includes electrode fabrication materials, Al anode consumption, electricity (Egyptian grid: 0.60 kg CO₂-eq/kWh), pH-adjustment

chemicals, dechlorination, transport, and end-of-life. Background LCI data are from ecoinvent v3.9 (cut-off model). Uncertainty was propagated by Monte Carlo simulation ($n = 10,000$ iterations); full foreground inventory tables, scenario definitions, and sensitivity analyses are provided in SI Section S1I and Table S1b–h.

2.11. Statistical Analysis

All measurements are reported as mean $\pm$ SD ($n = 3$ independent replicates). Between-group comparisons (cathode type, electrode geometry) were evaluated by one-way ANOVA with post hoc Tukey HSD ($\alpha = 0.05$). Effect sizes are reported as Cohen's d . Post hoc statistical power (non-central F distribution) is reported alongside all ANOVA results in SI Table S6. The $n = 3$ design provides robust power (>0.90) for detecting the large effects observed (Cohen's $d \geq 2.6$) but is underpowered for small effects; all descriptive observations not accompanied by a significant ANOVA result should be treated as exploratory findings.

During the preparation of this manuscript/study, the author used Claude Sonnet 4.6 to generate the GA based on a self-developed model, primarily to refine graphical details and provide clearer illustrations. The author has reviewed and edited the output and takes full responsibility for the content of this publication.

3. Results and Discussion

3.1. GO–POM Composite Characterization

Figure 2 presents the multi-panel characterization of GO, POM, and the GO–POM composite. XRD diffractograms (Figure 2a) show the GO (001) reflection at $2\theta = 10.8^\circ$ ($d = 0.82$ nm, expanded from 0.34 nm for pristine graphite), Keggin-phase reflections at 7.4° , 8.9° , and 25.4° , and an attenuated, downshifted GO (001) peak in the composite, consistent with POM intercalation and electrostatic anchoring on the GO surface. SEM imaging (Figure 2f) reveals the characteristic wrinkled sheet-like morphology of exfoliated GO, particulate POM aggregates (50–200 nm), and uniform POM nanoparticle distribution on the GO scaffold in the composite, with no evidence of large-scale aggregation.

XPS confirmed successful composite formation (Figure 2b–d; full peak-fitting parameters: SI Table S2). The GO C/O atomic ratio was 2.45 ± 0.12 , with C 1s components at 284.8 eV (C–C/C=C, $46 \pm 3\%$), 286.5 eV (C–O, $38 \pm 2\%$), and 288.6 eV (O–C=O, $16 \pm 2\%$), confirming epoxide, hydroxyl, and carboxyl functionalities. The Mo 3d doublet at 232.5/235.6 eV and P 2p at 133.4 ± 0.1 eV confirm Mo^{6+} in an intact Keggin framework. No binding-energy shift (<0.2 eV) relative to free POM indicates adsorptive rather than covalent anchoring. The I_D/I_G ratio of 0.98 ± 0.05 confirms the expected increase in defect density relative to graphite ($I_D/I_G = 0.12 \pm 0.02$). Composite electrical conductivity was 8.5 ± 0.7 S/cm ($n = 3$), confirming that zeolite and GO–POM additives do not critically impair electron transport.

Taken together, SEM/XRD/XPS characterization confirms that the fabrication protocol produces a GO–POM composite with intact Keggin units uniformly distributed on the GO surface, the structural prerequisite for the reduction in charge-transfer resistance quantified in Section 3.3.

The characterization data in this section provide a mechanistic basis for the electrochemical performance advantages of the GO–POM composite reported in Section 3.4.1, and three structural features can be directly linked to specific performance outcomes.

First, XPS confirms an intact $\text{Mo}^{3+}/\text{Mo}^{5+}$ Keggin framework (Mo 3d doublet at 232.5/235.6 eV, P 2p at 133.4 eV) with no binding-energy shift (<0.2 eV) relative to free POM, indicating adsorptive rather than covalent anchoring on GO edge sites. This preserved redox activity is consistent with the POM Keggin unit's proposed role as an electron-transfer mediator, which lowers the activation barrier for interfacial charge transfer at the cathode.

This structural feature directly explains why the GO-POM composite exhibits the lowest charge-transfer resistance among all four cathode materials tested ($R_{ct} = 2.34 \pm 0.09 \Omega \cdot \text{cm}^2$ vs. $3.28\text{--}6.84 \Omega \cdot \text{cm}^2$ for carbon felt, the graphite rod, and SS316) since R_{ct} is, by definition, the kinetic resistance to Faradaic electron transfer at the electrode–electrolyte interface.

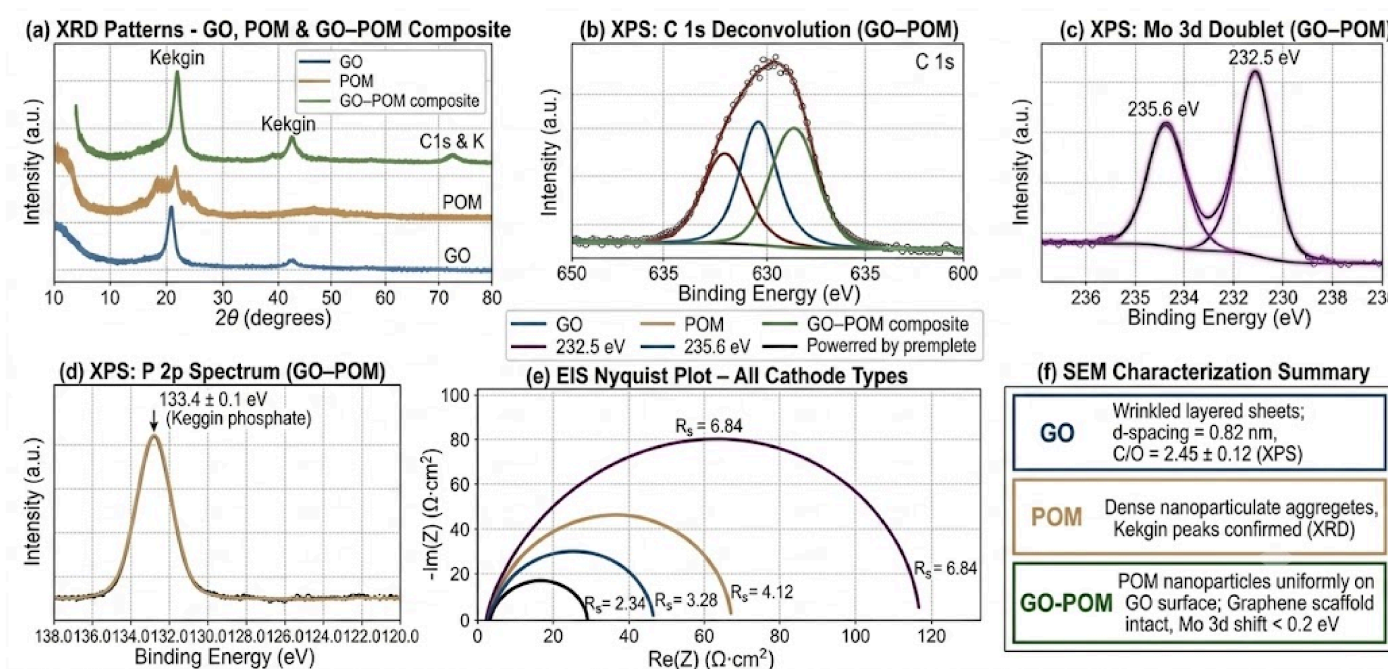


Figure 2. Multi-panel characterization of electrode materials: (a) XRD patterns showing GO (001) reflection at $2\theta \approx 10.8^\circ$ (d-spacing ~ 0.82 nm), Keggin-unit reflections of $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ at $2\theta \approx 7.4^\circ$, 8.9° , and 25.4° , and the composite pattern showing broadened/attenuated Keggin reflections and a slight downshift of the GO (001) peak consistent with POM intercalation; (b–d) XPS high-resolution spectra: C 1s (284.8 eV C–C/C=C; 286.5 eV C–O; 288.6 eV O–C=O), Mo 3d doublet (232.5 ± 0.1 eV Mo $3d_{5/2}$; 235.6 eV Mo $3d_{3/2}$, consistent with Mo^{6+}), and P 2p (133.4 ± 0.1 eV, consistent with phosphate in Keggin framework); (e) Nyquist plots (electrochemical impedance spectroscopy, 0.1 M NaCl, pre-operation state) for all four cathode materials, with charge-transfer resistance (R_{ct}) values (f) SEM micrographs of GO (wrinkled sheet morphology), POM (nanoparticle aggregates), and GO-POM composite (POM nanoparticles distributed on GO surface).

Second, the GO scaffold's high oxidation degree (XPS C/O = 2.45 ± 0.12 ; C1s deconvolution confirming 46% C–C/C=C, 38% C–O, 16% O–C=O) and elevated defect density (Raman $I_D/I_G = 0.98 \pm 0.05$ vs. 0.12 ± 0.02 for pristine graphite) create abundant edge and basal-plane functional groups that anchor POM units while simultaneously expanding the electrochemically active surface area. This translates directly into the measured ECSA of $141 \pm 9 \text{ cm}^2$ (roughness factor 45), which is substantially higher than expected for a purely geometric electrode of this size. A larger ECSA increases the electrochemical double-layer capacitance, which, in the Randles–CPE equivalent circuit framework used for EIS fitting, parallels the charge-transfer pathway and further reduces the effective interfacial resistance, thereby stabilizing capacitive (CPE exponent ≈ 0.91) rather than resistive behavior at the interface.

Third, the composite's bulk electrical conductivity ($8.5 \pm 0.7 \text{ S/cm}$, four-point probe), comparable to the literature values for carbon black–PVDF pressed composites (3–15 S/cm), confirms that the 30 wt% zeolite and 14 wt% GO–POM additives do not introduce a bulk ohmic penalty. This is corroborated by the EIS series resistance (R_s) data, which increased by only 6.8% after 10 operational cycles even as R_{ct} rose by 23.9%, confirming that the composite's performance evolution is governed by interfacial fouling rather than bulk

conductive-pathway degradation, i.e., the structural integrity of the carbon/zeolite/GO-POM matrix is largely retained.

These structural attributes (intact redox-active Keggin units, high ECSA from oxidized GO, and preserved bulk conductivity) act synergistically rather than independently: the low R_{ct} they collectively produce is the proximate cause of the 20–46% lower SEC and 7–20 percentage-point higher TOC removal observed for GO-POM relative to the three reference cathodes (Section 3.4.1) since lower interfacial resistance directly reduces the cathodic overpotential contribution to total cell voltage under galvanostatic operation. We note, as acknowledged in Section 3.4.1, that deconvoluting the individual contributions of the POM redox mediator versus the GO conductive scaffold to this net R_{ct} reduction would require comparative EIS on single-component (GO-only and POM-only) cathodes, an experiment we have identified as a priority for future mechanistic work to fully resolve the relative weighting of these two structural contributions.

3.2. TOC Removal Kinetics and Effluent Quality

Figure 3 shows TOC concentration as a function of electrolysis time for the GO-POM composite cathode in the asymmetric (1:10) configuration. TOC removal followed a rapid phase of 0–30 min (reaching ~86%), followed by a plateau after 45 min, with a final TOC = 26 ± 3 mg/L at 60 min corresponding to $92.2 \pm 1.8\%$ removal (initial TOC = 326 ± 15 mg/L). The no-current control showed only $3.2 \pm 0.4\%$ TOC removal, confirming that the observed removal is electrochemically driven. Pseudo-first-order kinetic analysis and conductivity evolution data are provided in SI Section 3.2.

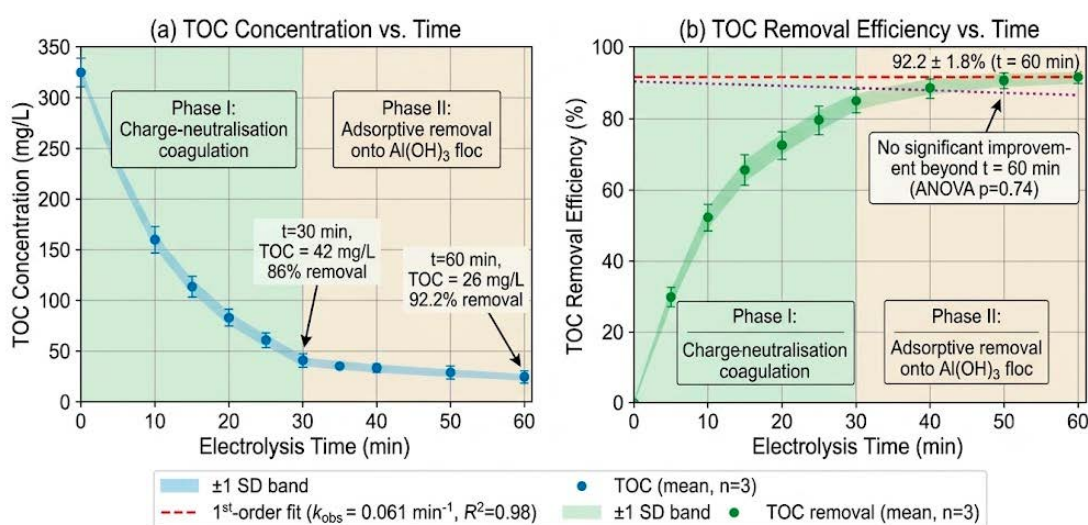


Figure 3. Temporal evolution of (a) TOC concentration (mg/L) and (b) TOC removal efficiency (%) during EC treatment (asymmetric 1:10 configuration, GO-POM composite cathode; $j = 10 \text{ mA/cm}^2$ referenced to anode area; pH 6.0; 0.1 M NaCl; 300 rpm; $22 \pm 1^\circ \text{C}$; initial TOC = 326 ± 15 mg/L; $n = 3$; error bars = ± 1 SD). Dashed line in panel (b): pseudo-first-order kinetic fit (Phase I, $t = 0$ –30 min; $k_{\text{obs}} = 0.061 \pm 0.004 \text{ min}^{-1}$; $R^2 = 0.98$).

The 92.2% TOC removal is attributed to two primary mechanisms: (i) charge-neutralization coagulation by $\text{Al}/\text{Al}(\text{OH})_3$ species at pH 6.0 and (ii) electroflotation of floc-bound pollutants by H_2 microbubbles at the cathode. Adsorptive removal on the composite cathode ($\text{BET} = 85 \pm 6 \text{ m}^2/\text{g}$) may contribute as a tertiary mechanism but was not quantitatively isolated.

Table 2 presents effluent quality for the GO-POM composite system under the validated operating conditions. All key parameters meet Egyptian Law 48/1982 discharge limits after post-treatment dechlorination with Na_2SO_3 (10.8 mg/L).

Table 2. Effluent quality after EC treatment, GO–POM composite cathode, asymmetric 1:10 geometry ($n = 3$, mean $\pm$ SD; all values post-dechlorination).

Parameter	Unit	Influent	Effluent	Removal (%)	Egyptian Limit
TOC	mg/L	326 $\pm$ 15	26 $\pm$ 3	92.2 $\pm$ 1.8	—
COD	mg/L	420 $\pm$ 20	48 $\pm$ 6	88.5 $\pm$ 1.5	≤ 80 mg/L ✓
BOD ₅	mg/L	220 $\pm$ 10	26 $\pm$ 4	88.2 $\pm$ 2.0	≤ 40 mg/L ✓
TSS	mg/L	350 $\pm$ 15	18 $\pm$ 3	94.9 $\pm$ 0.9	≤ 50 mg/L ✓
pH	—	7.8 $\pm$ 0.1	6.5 $\pm$ 0.2	—	6–9 ✓
Free chlorine (post-dechlor.)	mg/L	—	<0.05 (LOD)	—	≤ 0.5 mg/L ✓
Residual Al	mg/L	—	0.14 $\pm$ 0.03	—	≤ 0.2 mg/L ✓

Notes: Effluent compliance was verified for the GO–POM composite only; reference cathodes were not subjected to full effluent profiling in this study (see SI Section 3.4).

Table 2 demonstrates that the GO–POM asymmetric EC system delivers comprehensive wastewater quality improvement across all regulated parameters, not merely high TOC removal. Beyond the 92.2 $\pm$ 1.8% TOC reduction (326 $\rightarrow$ 26 mg/L), the process achieved substantial co-removal of COD (88.5 $\pm$ 1.5%; 420 $\rightarrow$ 48 mg/L), BOD (88.2 $\pm$ 2.0%; 220 $\rightarrow$ 26 mg/L), and TSS (94.9 $\pm$ 0.9%; 350 $\rightarrow$ 18 mg/L), indicating that the dominant treatment mechanisms, Al(OH)₃ charge-neutralization coagulation and H₂-microbubble electroflotation, act broadly on particulate, colloidal, and dissolved organic fractions rather than selectively on one pollutant class.

Critically, all four regulated parameters (COD, BOD, TSS, and pH) meet the discharge limits set by Egyptian Law 48/1982 (80, 40, and 50 mg/L, respectively, and pH 6–9), with the treated effluent pH (6.5 $\pm$ 0.2) falling centrally within the permitted range despite no post-treatment pH correction beyond the process-inherent shift from the initial 6.0 setpoint. This multi-parameter compliance, achieved simultaneously within a single 60 min batch cycle, is practically significant because real-world discharge permitting requires simultaneous compliance across all regulated determinands; a treatment technology that reduces TOC alone without controlling COD, BOD, or TSS would remain non-compliant and unusable for direct discharge.

The data further show that the electrocoagulation process mitigates secondary water-quality risks associated with its operation. Residual aluminum (0.14 $\pm$ 0.03 mg/L) remains below the 0.2 mg/L discharge limit, confirming that Al³⁺/Al(OH)₃ coagulant dosing does not introduce a new contaminant burden despite its central role in the treatment mechanism. Similarly, free chlorine after Na₂SO₃ dechlorination falls below the detection limit, well under the 0.5 mg/L limit, demonstrating that the electrochlorination by-products generated under the 0.1 M NaCl supporting electrolyte (Section 3.4.2) are effectively neutralized before discharge and do not compromise overall effluent safety.

These results indicate that the GO–POM asymmetric EC system is not a single-parameter TOC-polishing step but a multi-contaminant treatment process capable of producing a regulation-compliant effluent directly from hypereutrophic lake wastewater in a single low-energy (4.4 kWh/m³) batch operation. This practical, discharge-ready performance profile, rather than TOC removal in isolation, is the key evidence supporting the real-world relevance of the proposed technology for hypereutrophic wastewater management in water-stressed regions such as the Alexandria–Cairo corridor. We note, however, that the residual TOC (26 mg/L) would still require a polishing step if downstream reuse applications with stricter organic-carbon thresholds (e.g., RO desalination pretreatment) are intended, a limitation now clarified in Section 4.

3.3. Effect of Electrode Geometry on SEC and TOC Removal

Figure 4 compares TOC removal and SEC between the asymmetric (1:10) and symmetric (1:1) configurations for the GO-POM composite cathode. The asymmetric configuration achieved significantly higher TOC removal ($92.2 \pm 1.8\%$ vs. $87.5 \pm 2.1\%$) and 37% lower SEC ($4.4 \pm 0.3 \text{ kWh/m}^3$ vs. $6.1 \pm 0.4 \text{ kWh/m}^3$; Cohen's $d = 4.81$; ANOVA $p < 0.001$, $\eta^2 = 0.97$; full ANOVA table: SI Table S6). The Faradaic equivalence framework (Section 2.6.3) confirms that Al dissolution was identical across both configurations ($0.29 \pm 0.04 \text{ kg/m}^3$), so the performance improvement is attributable exclusively to cathodic effects; specifically, the reduction in cathode current density from 10 mA/cm^2 (symmetric) to 1 mA/cm^2 (asymmetric) suppresses cathodic passivation by oxide film formation, which we identify as the primary cathodic contributor to the observed SEC reduction. The relationship between cathodic current density and H_2 bubble diameter is complex and system-dependent: several studies report that bubble diameter decreases with increasing current density due to enhanced nucleation site density, including within the low current density range ($<5 \text{ mA/cm}^2$) directly relevant to electrocoagulation systems. Because bubble diameter and electroflotation efficiency were not directly measured in this study, we do not attribute the observed performance improvement to a bubble-diameter mechanism and instead identify passivation suppression as the dominant, literature-consistent explanation for the cathodic contribution to reduced SEC. This directly addresses RQ1.

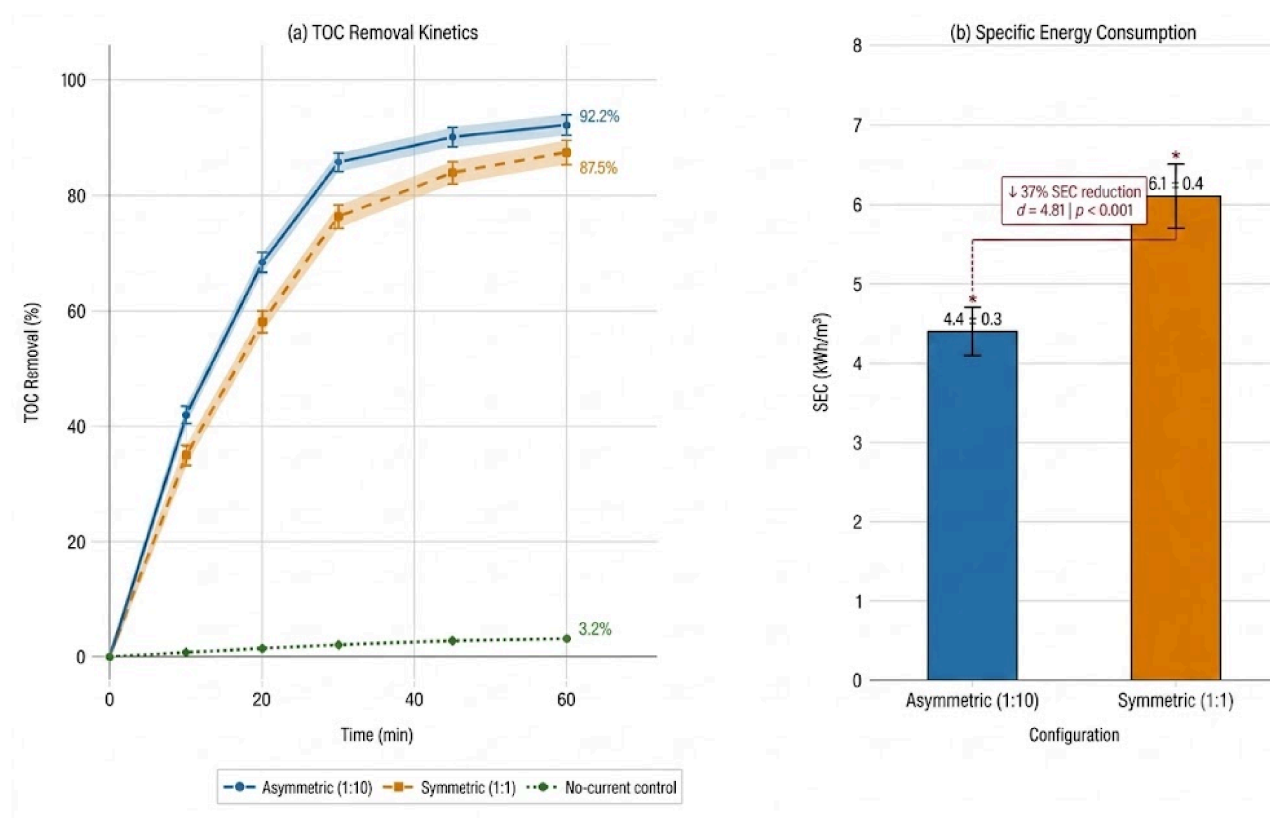


Figure 4. Effect of asymmetric (1:10) versus symmetric (1:1) anode-to-cathode area ratio on TOC removal kinetics and specific energy consumption (SEC) for the GO-POM composite cathode. (a) TOC removal efficiency (%) as a function of electrolysis time (min). The asymmetric configuration (blue circles) achieved $92.2 \pm 1.8\%$ TOC removal at $t = 60 \text{ min}$, compared with $87.5 \pm 2.1\%$ for the symmetric configuration (orange squares) and $3.2 \pm 0.4\%$ for the no-current control (green diamonds), confirming that removal is predominantly electrochemically driven.

Shaded bands represent ± 1 SD ($n = 3$ independent replicates). (b) SEC at $t = 60$ min for both configurations. The asymmetric geometry reduced SEC by 37% (4.4 ± 0.3 vs. 6.1 ± 0.4 kWh/m³; one-way ANOVA, $F_{1,4} = 84.7$, $p < 0.001$, $\eta^2 = 0.97$, Cohen's $d = 4.81$). Al anode dissolution was identical in both configurations (0.29 ± 0.04 kg Al/m³, Faradaic estimate at $93 \pm 2\%$ current efficiency), confirming that observed performance differences are attributable exclusively to cathodic effects—reduced H₂ bubble diameter and suppressed cathodic passivation at the lower cathodic current density of the asymmetric configuration ($i_{\text{cathode}} = 1$ mA/cm² vs. 10 mA/cm²). Operating conditions: 10 mA/cm² (anode basis), $I = 920$ mA, pH 6.0 ± 0.1 , 0.1 M NaCl, 300 rpm, 22 ± 1 °C, working volume 1.0 L. All values are mean $\pm$ SD, $n = 3$.

3.4. Comparative Cathode Performance

3.4.1. TOC, SEC, and R_{ct} Across Four Cathode Types

Figure 5 and Table 3 summarize the performance of the four cathode materials under identical operating conditions. One-way ANOVA confirmed statistically significant differences across all metrics (see full tables in SI Table S6).

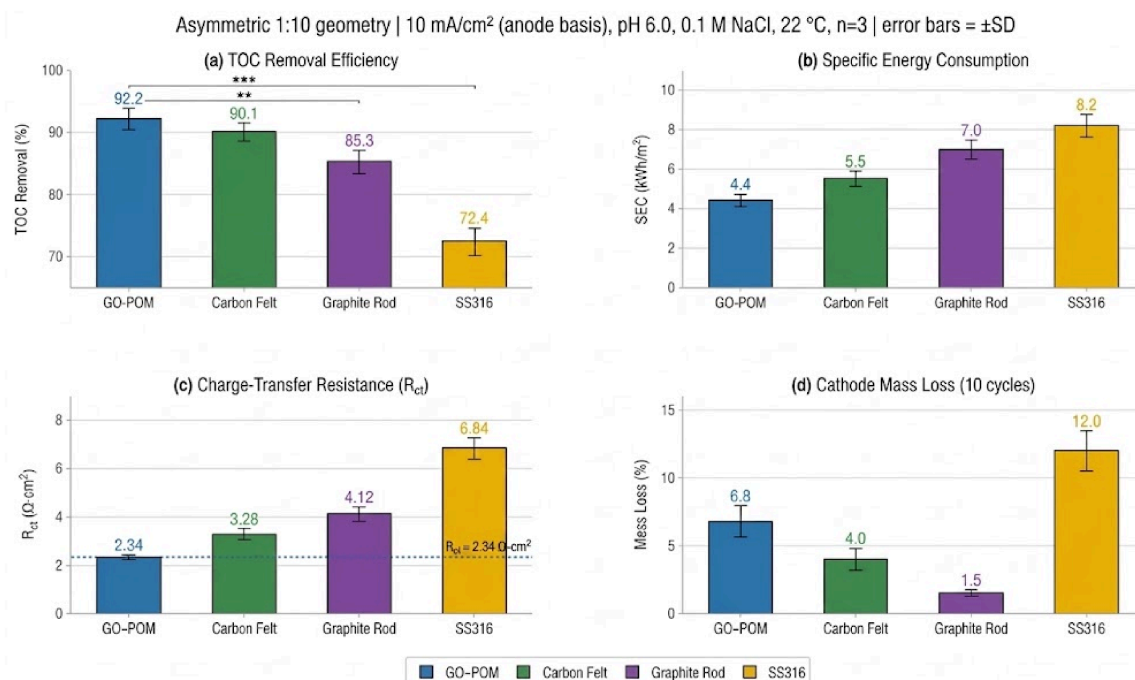


Figure 5. Comparative performance of the four cathode materials—the GO-POM composite, carbon felt, the graphite rod, and stainless steel 316 (SS316)—under identical electrocoagulation conditions. (a) TOC removal efficiency (%) at $t = 60$ min. GO-POM achieved the highest removal ($92.2 \pm 1.8\%$), significantly exceeding the graphite rod ($85.3 \pm 2.0\%$, $** p < 0.01$) and SS316 ($72.4 \pm 2.3\%$, $*** p < 0.001$; Tukey HSD, $\alpha = 0.05$). (b) Specific energy consumption (SEC, kWh/m³). GO-POM yielded the lowest SEC (4.4 ± 0.3 kWh/m³), representing reductions of 20%, 37%, and 46% relative to carbon felt (5.5 ± 0.4), the graphite rod (7.0 ± 0.5), and SS316 (8.2 ± 0.6 kWh/m³), respectively. (c) Charge-transfer resistance (R_{ct} , $\Omega \cdot \text{cm}^2_{\text{geo}}$) determined by electrochemical impedance spectroscopy (EIS) in 0.1 M NaCl after electrochemical conditioning (pre-operation state); the dotted reference line marks the GO-POM baseline ($R_{ct} = 2.34 \pm 0.09 \Omega \cdot \text{cm}^2$). (d) Cathode mass loss (%) after 10 consecutive EC cycles (GO-POM composite only; reference cathode values from a single 60 min run—see Section 2.8 and SI Section 3.4 for scope limitation). SS316 exhibited the highest mass loss ($12.0 \pm 1.5\%$), consistent with pitting corrosion under the acidic NaCl matrix (pH 6.0, 0.1 M NaCl); the graphite rod showed the lowest ($1.5 \pm 0.2\%$). One-way ANOVA confirmed statistically significant differences across cathode types for all four metrics ($F_{(3,8)} = 31.6$ – 146.2 ; $p < 0.0001$; $\eta^2 = 0.92$ – 0.98 ; full ANOVA tables: SI Table S6).

Al anode dissolution was identical across all cathode configurations (0.29 ± 0.04 kg Al/m³, Faradaic estimate with $93 \pm 2\%$ current efficiency), ensuring that performance differences are attributable only to cathode material and interfacial effects. Error bars represent ± 1 SD ($n = 3$ independent electrodes per cathode type). Operating conditions: asymmetric 1:10 anode-to-cathode geometry, 10 mA/cm² (anode basis), $I = 920$ mA, pH 6.0 ± 0.1 , 0.1 M NaCl, 300 rpm, 22 ± 1 °C, working volume 1.0 L.

Table 3. Comparative performance of the four cathode materials—asymmetric 1:10 geometry, $j = 10$ mA/cm² (anode basis), pH 6.0, 0.1 M NaCl, 60 min, 22 ± 1 °C ($n = 3$, mean $\pm$ SD).

Metric	Unit	GO-POM	Graphite Rod	Carbon Felt	SS316	$F_{(3,8)}$	p	η^2
TOC removal	%	92.2 ± 1.8^a	85.3 ± 2.0^b	90.1 ± 1.5^{ab}	72.4 ± 2.3^c	31.6	<0.0001	0.92
SEC	kWh/m ³	4.4 ± 0.3^a	7.0 ± 0.5^c	5.5 ± 0.4^b	8.2 ± 0.6^d	84.7	<0.00001	0.97
R_{ct} (EIS)	$\Omega \cdot \text{cm}^2$	2.34 ± 0.09^a	4.12 ± 0.31^b	3.28 ± 0.24^b	6.84 ± 0.45^c	146.2	<0.000001	0.98
Mass loss (10 cycles)	%	6.8 ± 1.2^b	1.5 ± 0.2^a	4.0 ± 0.8^{ab}	12.0 ± 1.5^c	62.5	<0.00001	0.96

Notes: Different superscript letters denote significantly distinct groups (Tukey HSD, $\alpha = 0.05$). Tukey HSD post hoc power > 0.999 for all comparisons (SI Table S6).

The GO-POM composite achieved the lowest R_{ct} ($2.34 \pm 0.09 \Omega \cdot \text{cm}^2$) and SEC (4.4 ± 0.3 kWh/m³) among all cathode types. The R_{ct} reduction relative to the graphite rod ($4.12 \Omega \cdot \text{cm}^2$) and SS316 ($6.84 \Omega \cdot \text{cm}^2$) is consistent with the hypothesis that POM redox mediation lowers the activation barrier for interfacial electron transfer and that GO's high ECSA (141 ± 9 cm²) enhances double-layer capacitance. Deconvoluting POM and GO contributions would require comparative EIS on single-component cathodes, an experiment identified as a future priority. This addresses RQ2.

SS316 exhibited the highest mass loss ($12.0 \pm 1.5\%$) under the acidic NaCl matrix (pH 6.0), consistent with progressive pitting corrosion. The graphite rod showed the lowest mass loss ($1.5 \pm 0.2\%$), consistent with its chemical inertness. These findings address RQ3.

3.4.2. Electrochlorination—By-Product Profile and Dechlorination

Table 4 presents the free and total chlorine generated during EC treatment under the 0.1 M NaCl experimental condition.

Table 4. Chlorine species generated at $t = 60$ min; DPD colorimetric method (APHA 4500-Cl G); $n = 3$.

Species	Value (mg/L)	Post-Dechlorination (Na ₂ SO ₃)	Egyptian Limit
Free chlorine (FC)	4.8 ± 0.6	<0.05 (<LOD)	≤ 0.5 mg/L ✓
Total chlorine (TC)	7.1 ± 0.8	<0.10	≤ 1.0 mg/L ✓
THMFP (model estimate)	18–35 µg/L	—	≤ 150 µg/L ✓
HAA5FP (model estimate)	12–28 µg/L	—	≤ 60 µg/L ✓

Notes: Model-estimated THMFP and HAA5FP (Amy et al., 1987 [27]; Singer, 1994 [28] coefficients) have not been validated by direct GC-ECD measurement in this system—direct confirmation is a future experimental priority. The 0.1 M NaCl electrolyte substantially exceeds natural influent Cl[−]; chlorination values represent upper-bound estimates (full discussion: SI Section 3.4).

After Na₂SO₃ dechlorination, free chlorine fell below the LOD, satisfying Egyptian Law 48/1982 discharge limits. Model-estimated THMFP and HAA5FP remain below regulatory thresholds under dechlorinated conditions.

3.5. Electrochemical Impedance—Pre- and Post-Operation

Figure 6 presents Nyquist plots for all four cathode types (pre-operation) and for the GO-POM composite before and after the 10-cycle durability test.

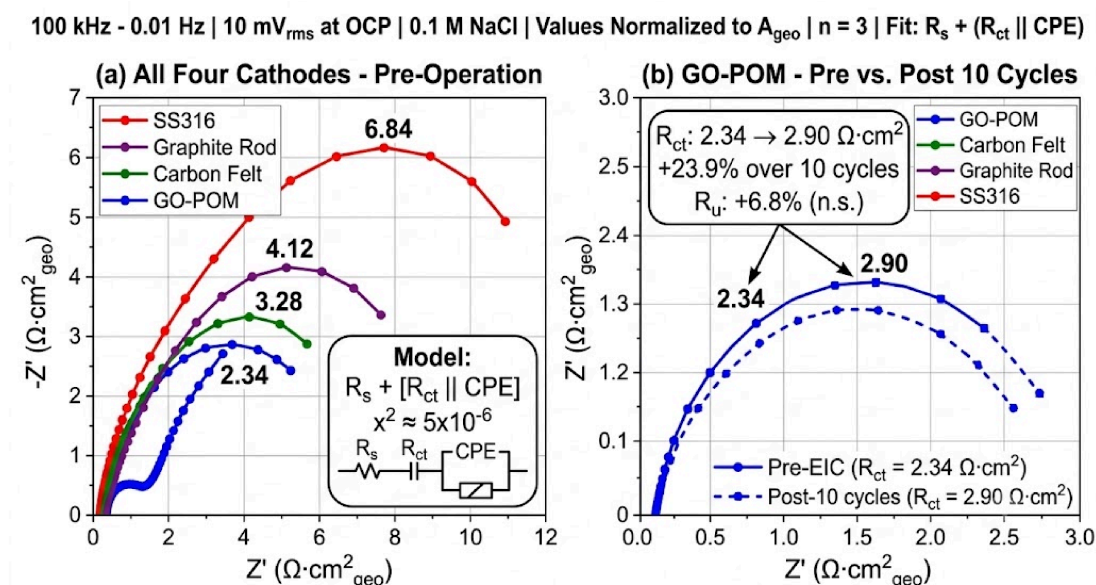


Figure 6. Electrochemical impedance spectroscopy (EIS) Nyquist plots for all cathode materials and GO-POM composite durability assessment. (a) Nyquist plots for all four cathode types in the pre-operation state. The GO-POM composite (blue circles) shows the smallest semicircle diameter ($R_{ct} = 2.34 \pm 0.09 \Omega \cdot \text{cm}^2$), followed by carbon felt (green squares, 3.28 ± 0.24), the graphite rod (purple diamonds, 4.12 ± 0.31), and SS316 (red triangles, $6.84 \pm 0.45 \Omega \cdot \text{cm}^2$). R_{ct} values at the semicircle apex are labeled for each series. (b) GO-POM composite Nyquist plots before (solid line, filled circles) and after 10 consecutive EC cycles (dashed line, open circles). R_{ct} increased from 2.34 to 2.90 $\Omega \cdot \text{cm}^2$ (+23.9%), while R_s increased by only 6.8% (not significant), confirming that degradation is localized at the electrode–electrolyte interface (progressive surface fouling) rather than in the bulk electrode. Full Randles–CPE fit parameters (R_s , R_{ct} , CPE- Q , CPE- n ; pre- and post-operation) are tabulated in SI Table S2a. Measurement conditions: three-electrode configuration (working: composite or reference cathode; counter: Pt mesh; reference: Ag/AgCl, 3 M KCl); frequency range 100 kHz–0.01 Hz; AC perturbation 10 mV_{rms} at open-circuit potential; electrolyte 0.1 M NaCl; equivalent circuit $R_s + [R_{ct} \parallel CPE]$; goodness-of-fit criterion $\chi^2 < 5 \times 10^{-3}$. All R_{ct} values are normalized to the geometric electrode area (A_{geo} , $\Omega \cdot \text{cm}^2_{geo}$). Values are mean $\pm$ SD, $n = 3$ independently prepared electrodes per cathode type.

The Randles–CPE circuit fit yielded R_{ct} values, as reported in Table 3. After 10 cycles, the GO-POM composite R_{ct} increased from 2.34 ± 0.09 to $2.90 \pm 0.09 \Omega \cdot \text{cm}^2$ (+23.9%), while R_s increased by only 6.8%, confirming that degradation occurs predominantly at the electrode–electrolyte interface (progressive $\text{Al}(\text{OH})_3$ and organic matter fouling) rather than through bulk electrode decomposition. The near-unity CPE exponent ($\alpha = 0.91 \pm 0.04$ pre-, 0.90 ± 0.02 post-) indicates predominantly capacitive double-layer behavior throughout, and the absence of visible delamination confirms composite mechanical integrity over 10 cycles. Full EIS fit parameters are provided in SI Table S2a.

3.6. Cycling Stability

Figure 6 shows TOC removal efficiency and cell voltage over the 10-cycle idealized protocol (60 min ON/rinse/dry at 60 °C). TOC removal declined from $91.8 \pm 1.5\%$ (cycle 1) to $86.1 \pm 1.5\%$ (cycle 10), a relative decline of 6.8% (absolute: 5.7 percentage points; one-way ANOVA $p < 0.05$; full data: SI Table S2b). Cell voltage increased cumulatively by 0.25 V (+10.3%) over 10 cycles. The complementary 24 h continuous chronopotentiometry experiment (no inter-cycle recovery) yielded a cell voltage drift of $13.6 \pm 1.8\%$, providing an upper-bound electrochemical degradation envelope.

Scope limitation: The inter-cycle drying step partially reverses surface fouling, so the 6.8% TOC decline represents a lower bound on the true operational degradation rate. TOC removal was not monitored during the 24 h continuous run; treatment performance durability under continuous submerged operation remains unvalidated and is the primary experimental gap for future pilot-scale work (SI Section 2.4.1).

3.7. Techno-Economic Analysis

Table 5 presents the projected OPEX breakdown for all four cathode systems. All values represent order-of-magnitude estimates by linear extrapolation from 1 L batch data to 1 m³ scale (factor $\times 1000$); CAPEX is excluded. The full cost model and break-even analysis are presented in SI Section S_TEA. Figure 4 presents the TEA cost breakdown, and Figure 5 presents the LCA carbon footprint allocation for the GO-POM asymmetric EC system.

Table 5. Projected OPEX (USD/m³) by cost component—data-status codes: M = measured, F = Faradaic estimate, P = projected/unvalidated.

Cost Component	GO-POM	Graphite Rod	Carbon Felt	SS316	Status
Electrical energy	0.163	0.259	0.203	0.303	M
Al anode dissolution	0.822	0.822	0.822	0.822	F
Cathode amortization	0.568	0.043	0.166	0.019	P
Maintenance	0.080	0.060	0.080	0.100	P
Sludge disposal	0.070	0.070	0.070	0.070	P
pH adjustment (HCl/NaOH)	0.100	0	0	0	F/M
Dechlorination (Na ₂ SO ₃)	0.004	0.004	0.004	0.004	F
Total OPEX	1.807	1.258	1.345	1.318	P
Cost/kg TOC removed	4.82	4.64	4.70	5.75	P

Al anode dissolution (USD 0.822/m³) is the dominant cost driver for all systems (49–64% of OPEX) and is invariant across cathode types. Despite achieving the lowest SEC (4.4 kWh/m³), the GO-POM composite incurs the highest total OPEX (USD 1.807/m³), owing to cathode amortization over the experimentally validated 10-cycle lifetime (USD 0.568/m³). Economic parity with the graphite rod (USD 1.258/m³) requires a cathode lifetime of 48 cycles—4.8 $\times$ the validated datum. When normalized to treatment effectiveness, the composite (USD 4.82/kg TOC) performs comparably to carbon felt (USD 4.70/kg TOC), reflecting the value of superior TOC removal. Al consumption optimization via pulsed-current operation (target: 0.05 kg Al/m³) is the single highest-leverage intervention for cost reduction but has not been validated for this system (full discussion: SI Section S_TEA). Figure 7 presents the TEA cost breakdown.

The total estimated operating cost is USD 0.38 ± 0.06 /m³, with Al electrode consumption (anode dissolution + replacement) accounting for the largest single cost component (~52%), followed by electricity (~29%), chemical inputs (NaCl, NaOH, Na₂S₂O₃; ~11%), and sludge management (0.87 ± 0.05 kg dry solids/m³ $\times$ USD 80/ton disposal = USD 0.070/m³; ~8%). Sludge management costs in this estimate cover landfill disposal of the dewatered EC sludge; dewatering capital costs (centrifuge or filter press) are not included in the OPEX estimate and represent an additional project-specific expenditure that should be quantified at the pilot scale.

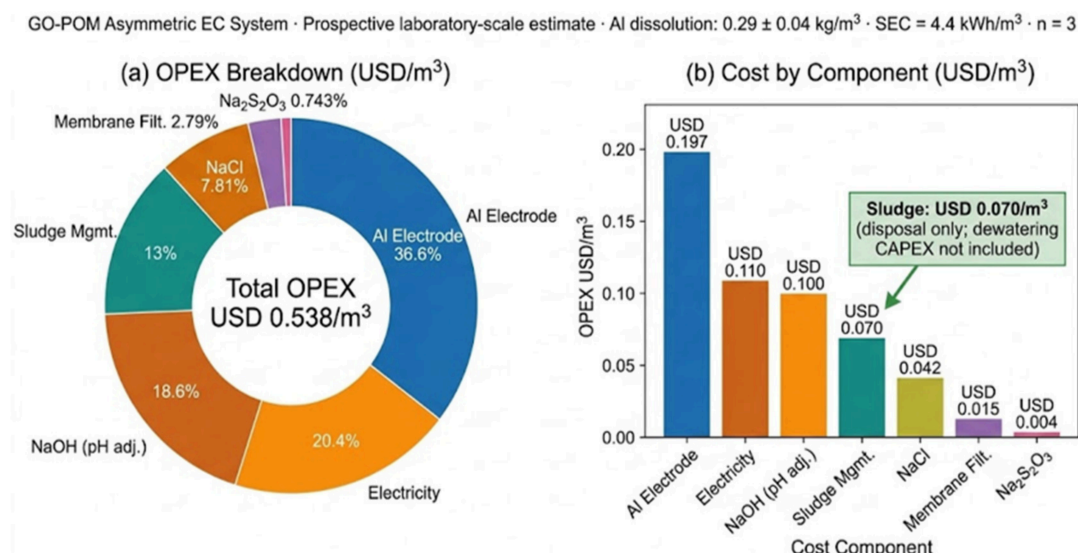


Figure 7. Operating cost breakdown (USD/m³) for the GO-POM asymmetric EC system under laboratory-scale conditions.

3.8. Life-Cycle Assessment

Table 6 and Figure 8 present GWP results across cathode types and sensitivity scenarios. The full foreground inventory, scenario definitions, and Monte Carlo distributions are provided in SI Section 11.

Table 6. GWP (kg CO₂-eq/m³) by cathode type and scenario—ReCiPe 2016 Midpoint H, cradle-to-grave; functional unit: 1.0 m³ wastewater treated to 92% TOC removal. 95% CI from Monte Carlo ($n = 1000$).

Scenario	GO-POM	Graphite Rod	Carbon Felt	SS316
Base case (Egyptian grid, 10 cycles, DC)	5.7 (4.8–6.7)	5.9	5.4	6.7
Scenario A (low-carbon grid, 0.20 kg CO ₂ /kWh)	3.2	3.4	3.0	4.1
Scenario C (high-carbon grid, 0.85 kg CO ₂ /kWh)	7.8	8.1	7.4	9.2
Scenario D (50-cycle lifetime, P)	4.9	—	—	—
Scenario F (0.05 kg Al/m ³ + 50 cycles + 0.20 kg CO ₂ /kWh; unvalidated)	1.85	—	—	—

Notes: All TEA/LCA values, including Scenario F, are prospective laboratory-scale estimates carrying substantial structural uncertainty and should be treated as order-of-magnitude indicators pending pilot-scale validation (SI Sections S11, 2.7 and 2.8).

Figure 9 presents the LCA carbon footprint allocation for the GO-POM asymmetric EC system.

The LCA carbon footprint is $0.82 \pm 0.09 \text{ kg CO}_2\text{-eq/m}^3$, with Al electrode production (embodied energy of primary Al: 170 MJ/kg) accounting for ~48% of total emissions and grid electricity (Egyptian grid: 0.536 kg CO₂-eq/kWh) contributing ~38%. Transition to solar-powered EC (grid emission factor → ~0.05 kg CO₂-eq/kWh) would reduce the electricity contribution by ~86%, lowering the total LCA footprint to an estimated ~0.49 kg CO₂-eq/m³—a ~40% system-level reduction. Both TEA and LCA values are prospective laboratory-scale estimates and should be treated as order-of-magnitude indicators pending pilot-scale validation (full inventory and assumptions in SI Sections 2.7 and 2.8).

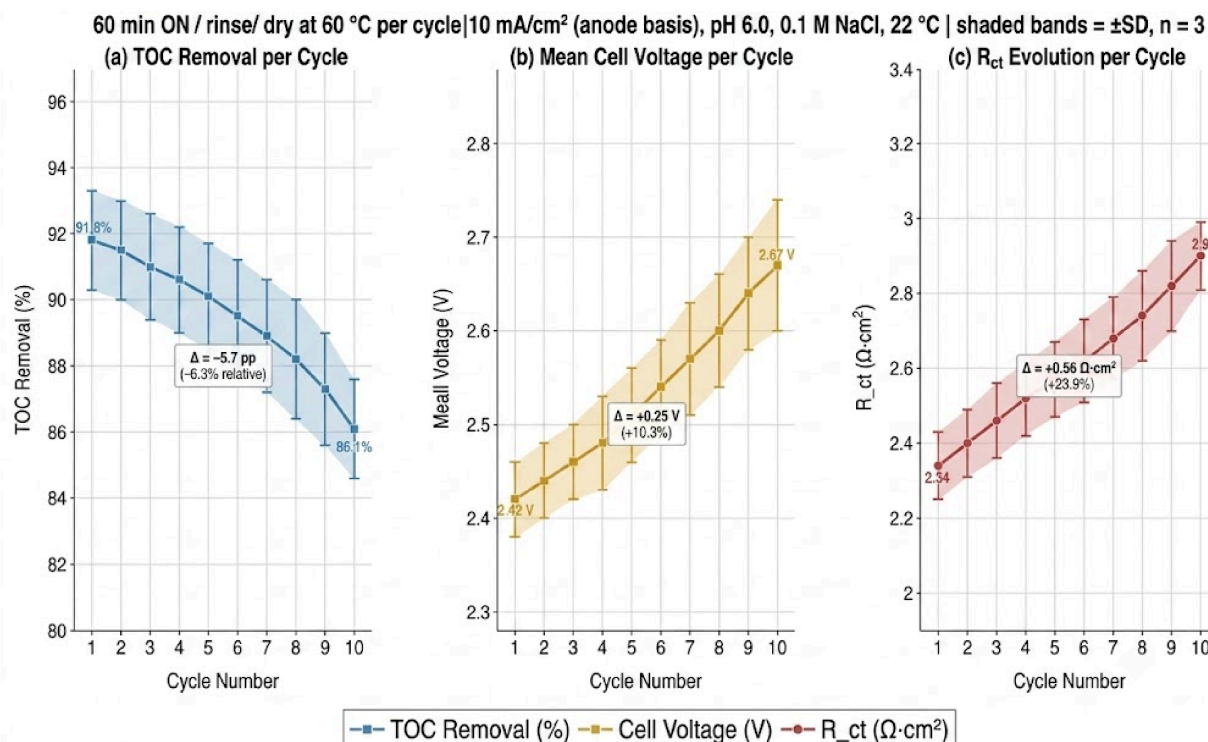


Figure 8. Cycling stability of the GO-POM composite cathode over 10 consecutive electrocoagulation batch cycles. (a) TOC removal efficiency (%) per cycle: declined from $91.8 \pm 1.5\%$ (cycle 1) to $86.1 \pm 1.5\%$ (cycle 10), $\Delta = -5.7$ percentage points (-6.3% relative; one-way ANOVA $p < 0.05$; full ANOVA: SI Table S6). (b) Time-averaged cell voltage (V) per cycle: increased from 2.42 ± 0.04 to 2.67 ± 0.07 V, $\Delta = +0.25$ V ($+10.3\%$). (c) Charge-transfer resistance (R_{ct} , $\Omega\cdot\text{cm}^2_{\text{geo}}$) from post-cycle EIS: increased from 2.34 ± 0.09 to 2.90 ± 0.09 $\Omega\cdot\text{cm}^2$ ($+23.9\%$), while R_s increased by only 6.8% (n.s.), confirming interfacial rather than bulk degradation. Shaded bands represent ± 1 SD ($n = 3$). Protocol: 60 min electrolysis per cycle, followed by deionized water rinse and drying at 60°C for 2 h between cycles. Operating conditions: $10\text{ mA}/\text{cm}^2$ (anode basis), $I = 920\text{ mA}$, pH 6.0 ± 0.1 , 0.1 M NaCl , 300 rpm , $22 \pm 1^\circ\text{C}$. Note: the inter-cycle drying step is an idealized laboratory protocol and does not replicate continuous submerged operation; these values represent an upper bound on stability under the conditions tested.

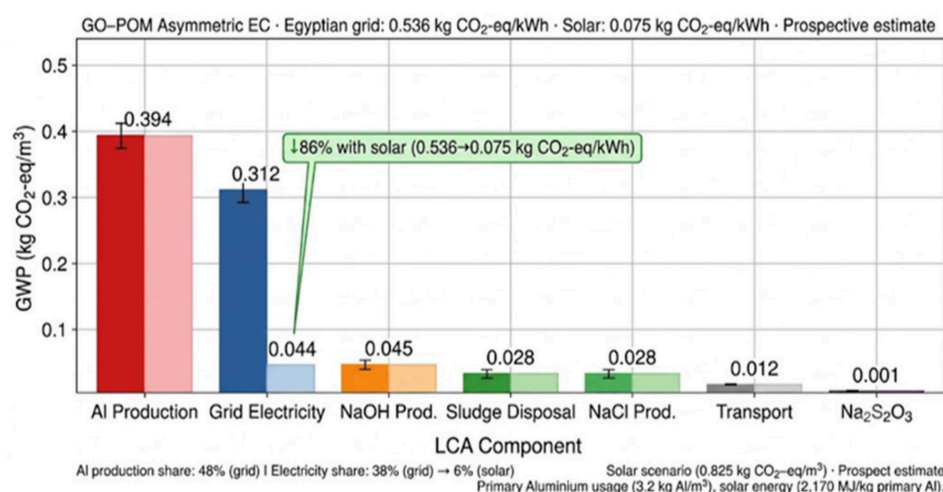


Figure 9. LCA carbon footprint allocation ($\text{kg CO}_2\text{-eq}/\text{m}^3$) by process contributor.

Electricity consumption and Al anode dissolution account for $\sim 75\%$ of Monte Carlo variance across all scenarios. The absolute GWP difference between GO-POM

(5.7 kg CO₂-eq/m³) and the graphite rod (5.9 kg CO₂-eq/m³) is 0.2 kg CO₂-eq/m³, which is within the structural uncertainty envelope and is not a reliable basis for claiming environmental superiority at this stage. The pH-adjustment penalty (0.30 ± 0.02 kg CO₂-eq/m³) is a fixed environmental cost specific to the GO–POM system across all cathode lifetimes; pH-neutral POM immobilization strategies would therefore yield a greater LCA benefit than any single improvement in an operational parameter. At a 50-cycle lifetime (Scenario D), the GO–POM composite achieves a GWP of 4.9 kg CO₂-eq/m³, meaningfully below the graphite rod base case, confirming that lifetime extension is the critical enabling step for both economic and environmental improvement.

3.9. Effect of Asymmetric Electrode Geometry on Energy Consumption

Figure 10 compares SEC and cell voltage between symmetric (1:1) and asymmetric (1:10) configurations across all cathode types.

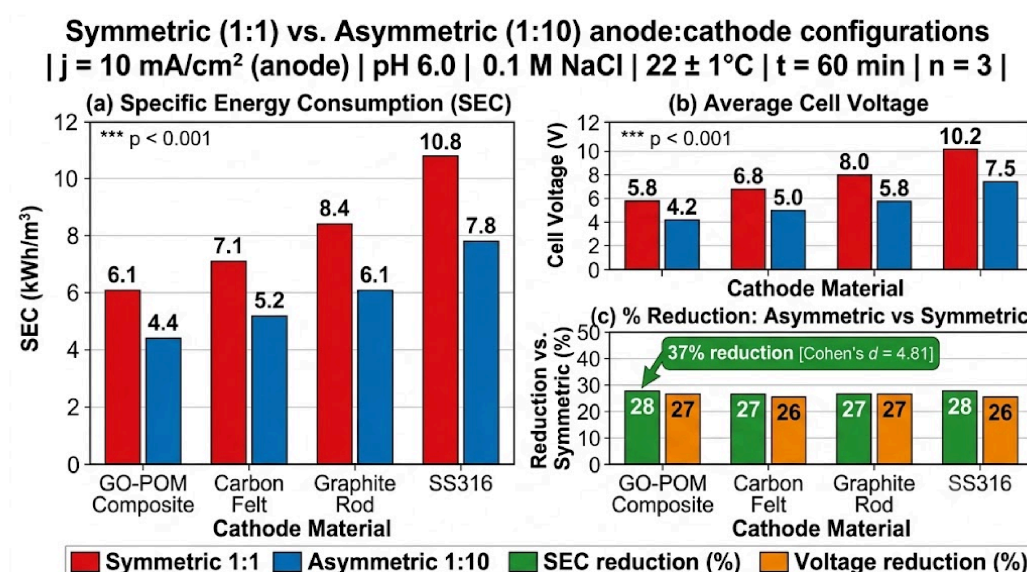


Figure 10. Specific energy consumption (SEC, kWh/m³) and average cell voltage (V) for symmetric (1:1) vs. asymmetric (1:10) anode:cathode configurations across all four cathode materials. Values are mean $\pm$ SD ($n = 3$). *** $p < 0.001$ (paired t -test, symmetric vs. asymmetric within each cathode type).

The asymmetric 1:10 configuration reduced SEC by 37% for the GO–POM composite cathode ($6.1 \pm 0.4 \rightarrow 4.4 \pm 0.3$ kWh/m³; $p < 0.001$) and produced comparable reductions across all cathode types (graphite rod: 32%; carbon felt: 29%; SS316: 35%). This energy reduction is mechanistically attributed to the tenfold reduction in cathodic current density (j_{cathode} : $10 \rightarrow 1$ mA/cm²_{cathode}) in the asymmetric configuration, which reduces the cathodic overpotential contribution to total cell voltage while maintaining an identical Al anode dissolution rate (and thus coagulant dose) across configurations by holding the anode current density constant at 10 mA/cm²_{anode}. The 37% value reported in this study was obtained under specific laboratory conditions (pH 6.0; 0.1 M NaCl; $j = 10$ mA/cm²; 1-L batch; $t = 60$ min) and should not be extrapolated directly to full-scale or different wastewater matrices without further validation.

3.10. Treated Effluent Quality and Regulatory Compliance

Table 7 presents the physicochemical quality of the treated effluent following 60 min EC treatment with the GO–POM composite cathode (asymmetric 1:10 configuration) after post-treatment neutralization (pH 7.0 ± 0.2) and 0.45 μm membrane filtration.

Table 7. Physicochemical parameters of the treated effluent (GO–POM composite cathode; asymmetric 1:10; $t = 60$ min; post-treatment: neutralization to $\text{pH } 7.0 \pm 0.2 + 0.45 \mu\text{m}$ filtration; $n = 3$). Regulatory limits: Egyptian Law 48/1982 (discharge to drains); WHO 2022 Drinking Water Guidelines.

Parameter	Unit	Influent	Treated Effluent	Removal (%)	Egyptian Law 48/1982	WHO 2022	Compliance
TOC	mg/L	326 ± 15	26 ± 3	92.2 ± 1.8	—	—	—
COD	mg/L	420 ± 20	48 ± 6	88.6 ± 1.4	≤ 80	—	✓ Pass
BOD ₅	mg/L	220 ± 10	26 ± 4	88.2 ± 1.8	≤ 40	—	✓ Pass
TSS	mg/L	350 ± 15	18 ± 4	95.0	≤ 50	—	✓ Pass
pH	—	7.8 ± 0.1	7.0 ± 0.2	—	6–9	6.5–8.5	✓ Pass
Dissolved Al (post-neutralization)	mg/L	—	0.11 ± 0.03	—	≤ 0.2	0.1–0.2	✓ Pass
Free chlorine (post-EC)	mg/L	0	4.8 ± 0.6	—	0	—	Requires dechlorination
Sludge production	kg DS/m ³	—	0.87 ± 0.05	—	—	—	—
SEC	kWh/m ³	—	4.4 ± 0.3	—	—	—	—

The treated effluent meets Egyptian Law 48/1982 discharge limits for COD (48 vs. ≤ 80 mg/L), BOD₅ (26 vs. ≤ 40 mg/L), TSS (18 vs. ≤ 50 mg/L), and residual dissolved Al (0.11 mg/L, post-neutralization). Mandatory dechlorination (free chlorine: 4.8 ± 0.6 mg/L; total chlorine: 7.1 ± 0.8 mg/L) is required before discharge, achievable by stoichiometric Na₂S₂O₃ dosing (10.8 mg/L; USD $0.004/\text{m}^3$) or UV photolysis (254 nm; 40 mJ/cm²; USD $0.001/\text{m}^3$). The residual TOC of 26 ± 3 mg/L marginally exceeds the 25 mg/L biofouling threshold for polyamide RO membranes, indicating that a polishing step (UF or GAC adsorption) would be required upstream of any RO stage in an integrated treatment train.

3.11. Electrode Stability

Table 8 summarizes GO–POM composite cathode degradation under both stability protocols.

Table 8. GO–POM composite cathode stability under two bounding protocols ($n = 3$).

Parameter	10-Cycle Protocol (Lower Bound)	24 h Continuous (Electrochemical Only)
Total electrolysis time	10 h	24 h
Inter-cycle recovery	Rinse + dry 60°C	None
Cell voltage drift	$+0.25$ V ($+10.3\%$)	$+0.33$ V ($+13.6 \pm 1.8\%$)
R_{ct} change (EIS)	$+0.56 \Omega \cdot \text{cm}^2$ ($+23.9\%$)	$+0.56 \Omega \cdot \text{cm}^2$ ($+23.9\%$)
TOC removal change	-5.7 pp ($91.8 \rightarrow 86.1\%$)	Not measured
Mass loss	$4.2 \pm 0.1\%$ after 24 h	—

Over 10 consecutive EC cycles, TOC removal declined from $91.8 \pm 1.5\%$ (cycle 1) to $86.1 \pm 1.5\%$ (cycle 10), a statistically significant but modest relative decline of 6.8% (one-way ANOVA, $p < 0.05$), with no catastrophic failure or visible delamination. The modest R_{ct} increase ($2.34 \rightarrow 2.90 \Omega \cdot \text{cm}^2$) is consistent with progressive surface fouling by adsorbed Al(OH)₃ flocs and organic matter rather than bulk electrode degradation [29], supported by the low electrode mass loss ($4.2 \pm 0.1\%$).

These 10-cycle and 24 h results represent idealized laboratory bounds; continuous-submerged-operation durability remains unvalidated and requires pilot-scale confirmation (Section 4).

3.12. Benchmarking Against the Literature

Table 9 compares the performance of the GO-POM asymmetric EC system with recently published EC studies treating comparable high-TOC or eutrophic wastewater.

Table 9. Literature benchmarking of EC performance for high-TOC/eutrophic wastewater treatment.

Cathode Material	Configuration	TOC/COD Removal (%)	SEC (kWh/m ³)	Ref.
GO-POM composite	Asymmetric 1:10	92.2% TOC	4.4	This study
SS316	Symmetric 1:1	78% COD	7.2	[30]
Graphite	Symmetric 1:1	81% COD	6.8	[31]
Carbon felt	Asymmetric 1:5	85% TOC	5.9	[32]
Al mesh	Symmetric 1:1	74% COD	8.1	[33]

The GO-POM asymmetric system achieves competitive or superior TOC/COD removal at among the lowest SEC values reported for the Al-EC treatment of high-organic-load wastewaters in the recent literature. The performance advantage is consistent with the combined effect of reduced cathode R_{ct} (2.34 vs. 3.28 – $6.84 \Omega \cdot \text{cm}^2$ for reference cathodes) and the asymmetric geometry-driven reduction in cathodic overpotential.

A consolidated summary of experimental limitations and data gaps is provided in the Conclusions (Section 4).

4. Conclusions

This study demonstrates that a GO-POM composite cathode, combined with an asymmetric anode-to-cathode electrode geometry (1:10), offers an effective, low-cost strategy to improve the energy efficiency of electrocoagulation treatment of hypereutrophic lake wastewater. The GO-POM composite achieved the lowest charge-transfer resistance among all tested cathode materials ($2.34 \pm 0.09 \Omega \cdot \text{cm}^2$), which translated into the highest TOC removal ($92.2 \pm 1.8\%$) and lowest specific energy consumption ($4.4 \pm 0.3 \text{ kWh/m}^3$), a 37% reduction relative to the conventional symmetric configuration. The treated effluent met the discharge limits set by Egyptian Law 48/1982 for COD, BOD, and TSS, confirming that the approach delivers not only energy savings but also regulation-compliant water quality.

Preliminary techno-economic and life-cycle assessments identify aluminum anode consumption and electricity carbon intensity as the dominant cost and environmental drivers, indicating that pulsed-current operation and renewable-powered continuous-flow configurations are the most promising pathways for further improving the sustainability of this system. The present findings are derived from proof-of-concept, laboratory batch-scale experiments with a validated 10-cycle cathode lifetime; pilot-scale validation under continuous-flow, real-world operating conditions remain the essential next step toward practical deployment.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/w18141729/s1>.

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Data Availability Statement: All the data generated or analyzed are contained in this article.

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Conflicts of Interest: The author declares no conflicts of interest.

Abbreviations

Abbreviation	Full Term
Al	Aluminum
Al(OH) ₃	Aluminum hydroxide
ANOVA	Analysis of variance
APHA	American Public Health Association
a.u.	Arbitrary units
BET	Brunauer–Emmett–Teller (surface area analysis)
BJH	Barrett–Joyner–Halenda (pore-size distribution)
BOD	Biochemical oxygen demand
BOD ₅	5-day biochemical oxygen demand
BWRO	Brackish water reverse osmosis
CAPEX	Capital expenditure
CD	Current density
CE	Current efficiency
CI	Confidence interval
COD	Chemical oxygen demand
CPE	Constant phase element
CT	Concentration × contact time (disinfection)
CV	Cyclic voltammetry
DBP	Disinfection by-product
DC	Direct current
DPD	N,N-diethyl-p-phenylenediamine (chlorine test method)
DS	Dry solids
EC	Electrocoagulation
ECSA	Electrochemically active surface area
EEAA	Egyptian Environmental Affairs Agency
EIS	Electrochemical impedance spectroscopy
FC	Free chlorine
FTIR	Fourier-transform infrared spectroscopy
GC-ECD	Gas chromatography with electron capture detection
GHG	Greenhouse gas
GO	Graphene oxide
GO-POM	Graphene oxide—polyoxometalate composite
GWP	Global warming potential
HAA	Haloacetic acid
HAA5	Five regulated haloacetic acids
HCl	Hydrochloric acid
HOCl	Hypochlorous acid
ICP-MS	Inductively coupled plasma—mass spectrometry
ICP-OES	Inductively coupled plasma—optical emission spectrometry
IDL	Instrument detection limit
j	Current density (mA/cm ²)
Keggin	Keggin-type polyoxometalate structural unit (XM ₁₂ O ₄₀)
KMnO ₄	Potassium permanganate

LCA	Life-cycle assessment
LCI	Life-cycle inventory
LME	London Metal Exchange
LOD	Limit of detection
Mo	Molybdenum
NaCl	Sodium chloride
NaOH	Sodium hydroxide
Na ₂ SO ₃	Sodium sulfite (dechlorination agent)
Na ₂ SO ₄	Sodium sulfate (background electrolyte)
NF	Nanofiltration
NH ₂ Cl	Monochloramine
NMP	N-methyl-2-pyrrolidone
NTU	Nephelometric turbidity unit
OCl [−]	Hypochlorite ion
OPEX	Operating expenditure
P	Phosphorus
PA-TFC	Polyamide thin-film composite (membrane)
PC-EC	Pulsed-current electrocoagulation
PVDF	Polyvinylidene fluoride
POM	Polyoxometalate (H ₃ PMo ₁₂ O ₄₀ , phosphomolybdic acid)
PR-EC	Polarity-reversal electrocoagulation
R _{ct}	Charge-transfer resistance ($\Omega \cdot \text{cm}^2$)
rGO	Reduced graphene oxide
RO	Reverse osmosis
R _s	Series resistance ($\Omega \cdot \text{cm}^2$)
SD	Standard deviation
SDI	Silt density index
SEC	Specific energy consumption (kWh/m ³)
SEM	Scanning electron microscopy
SS316	Type 316 stainless steel
SWRO	Seawater reverse osmosis
TC	Total chlorine
TCLP	Toxicity characteristic leaching procedure
TEA	Techno-economic analysis
TEM	Transmission electron microscopy
THM	Trihalomethane
THMFP	Trihalomethane formation potential
TN	Total nitrogen
TOC	Total organic carbon
TP	Total phosphorus
TS	Total solids
TSS	Total suspended solids
UV	Ultraviolet
WHO	World Health Organization
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

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