



Cynodon dactylon-Mediated Zinc Oxide Nanoparticles for Eriochrome Black T Removal Under Simulated Sunlight

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ABSTRACT

Background: This study evaluates *Cynodon dactylon* leaf extract as a synthesis aid for ZnO nanoparticles used to remove Eriochrome Black T (EBT) from water. **Methods:** The material was characterized by diffraction, spectroscopy and microscopy. EBT removal was tested after 30 min dark equilibration and 90 min exposure to simulated sunlight, with dark, light-only and dye-stability controls. **Results:** The ZnO pattern was consistent with wurtzite, the estimated optical band gap was 3.20 eV, and TEM particle diameter averaged 27.6 ± 6.8 nm (200 particles). At the best-performing conditions tested (10 mg L⁻¹ EBT, 2.0 g L⁻¹ ZnO, pH 10 and 35 °C), apparent EBT removal reached 92.4% (10 independent trials). The Langmuir fit gave a separate estimated capacity, $q_{\text{max}} \approx 92.4$ mg g⁻¹. Total organic carbon and chemical oxygen demand decreased by 78.6% and 82.4%, respectively; these measures do not establish complete mineralization. Apparent removal declined by 4.5 percentage points, from 92.4% initially to 87.9% after five reuse cycles. **Conclusion:** Plant-mediated ZnO gave substantial EBT removal under the tested conditions. The contributions of adsorption and photochemistry, and the identity of reactive species, remain unresolved.

Keywords: *Cynodon dactylon*; Zinc oxide; Eriochrome Black T removal; Photocatalysis; Zinc dissolution.

1. INTRODUCTION

Eriochrome Black T (EBT) is an azo dye whose decrease in visible absorbance can arise from adsorption, photolysis, chromophore transformation or other processes. A lower EBT signal therefore describes apparent dye removal or decolorization; it does not, by itself, demonstrate molecular degradation or mineralization. Total organic carbon (TOC) and chemical oxygen demand (COD) provide additional, but still incomplete, evidence about changes in the organic load. ZnO is a light-responsive semiconductor with a pH-dependent surface. Plant constituents may influence ZnO precipitation and particle growth (Mutukwa et al. 2024). Meenatchi et al. (2020) reported ZnO prepared with *C. dactylon* extract, but that work did not evaluate EBT removal. Related wastewater studies have also examined agricultural-waste-derived adsorbents for dye removal, demonstrating the value of comparing adsorption-based treatment with semiconductor photocatalysis (Rajesh et al. 2024). Green-synthesized plant-assisted metal-oxide nanocomposites have likewise been investigated for multifunctional environmental applications, providing broader context for plant-mediated nanomaterial preparation (Hema et al. 2025). Earlier ZnO–EBT studies

used different catalyst preparations, pH values and irradiation conditions (Sadollahkhani and Kazeminezhad, 2014; Vaiano et al. 2017; Lanjwani et al. 2023). These differences prevent a direct ranking of removal percentages. Solution pH affects both EBT speciation and ZnO surface charge. Below the measured point of zero charge (pHPZC $\approx$ 6.2), protonated ZnO surfaces may favor adsorption of anionic EBT; above it, deprotonated surfaces may reduce electrostatic attraction. This simple picture cannot alone predict removal, because photochemical behavior, dye speciation, and ZnO dissolution also change with pH. ZnO dissolution is especially relevant under acidic conditions (Jiang et al. 2015; Cardoso et al. 2021; Kürsteiner et al. 2022). The gap addressed here is the limited assessment of *C. dactylon*-mediated ZnO for EBT removal with separate dark, photolysis and illuminated-catalyst controls. We characterize the material, examine the tested effects of pH, catalyst dose, initial dye concentration, time, temperature and NaCl, and report EBT removal together with TOC, COD and dissolved zinc. We interpret the results as removal under specified conditions, while identifying the experiments still required to establish transformation pathways and catalyst stability.

2. MATERIALS AND METHODS

2.1 Materials and Preparation of Leaf Extract

Healthy leaves of *C. dactylon* were harvested from the Dammam area in the Eastern Province of Saudi Arabia. The plant was identified based on its typical morphology, but no voucher specimen was kept. The leaves were cleaned with tap water, rinsed with ultrapure water, shade-dried, and powdered. The leaf powder (20.0 g of the powder) was processed with 100 mL of ultrapure water at 60 °C for 40 minutes, after which the extract was filtered and cooled before use. All the chemicals, including zinc acetate dihydrate, sodium hydroxide, hydrochloric acid, EBT, ethanol, and ultrapure water, were of analytical grade.

2.2 Green Synthesis of ZnO

For a typical batch, 100 mL of 0.5 M zinc acetate solution was mixed with 50 mL of leaf extract at 60 °C (150 mL before NaOH addition). A 1 M NaOH solution was added dropwise to pH 10.0 ± 0.2 . The mixture was stirred for approximately 2 h and aged overnight. The precipitate was collected by centrifugation and washed with ultrapure water and ethanol until the wash liquid was neutral. The product was dried overnight at 80 °C and calcined in air at 450 °C for 2 h ($5\text{ }^{\circ}\text{C min}^{-1}$). The source record does not report stirring speed, NaOH volume, centrifugation settings, wash-cycle count or the experimental rationale for calcination; these details require confirmation from the laboratory record. No plant-extract-only or chemically synthesized ZnO control is documented.

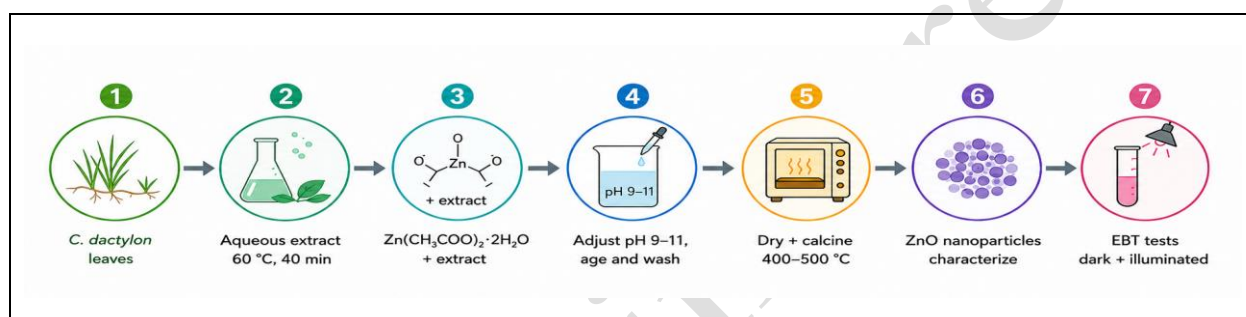


Fig. 1: Schematic of the plant-mediated ZnO synthesis and EBT-evaluation workflow used in this study

2.3 Characterization

Characterization was performed at the Core Research Facilities, King Fahd University of Petroleum and Minerals, Dhahran, Saudi Arabia. UV–visible diffuse reflectance spectra were recorded from 200 to 800 nm at 1 nm resolution with BaSO₄ as reference. Reflectance *R* was transformed using $F(R) = (1 - R)^2/(2R)$. Assuming a direct allowed transition, the optical gap was estimated by extrapolating the approximately linear portion of $[F(R)h\nu]^2$ versus $h\nu$ to zero ordinate; the selected fitting interval and uncertainty were not retained in the source record. ATR-FTIR spectra covered 4000–400 cm^{−1} at 4 cm^{−1} resolution with 32 scans. Powder XRD used Cu K α radiation ($\lambda = 1.5406\text{ \AA}$), 40 kV and 40 mA over $2\theta = 20\text{--}80^{\circ}$ in 0.02° steps; the stated scan rate was approximately $1^{\circ}\text{ min}^{-1}$. FESEM used 10 kV and an 8–10 mm working distance; EDX used 15 kV and 60 s live time. TEM, HRTEM and SAED images were used for the reported size and lattice observations. Manufacturer/model identifiers, TEM preparation and operating settings, FESEM sample preparation, and the selected XRD fitting details were not supplied in the source record and must be inserted from instrument logs before submission.

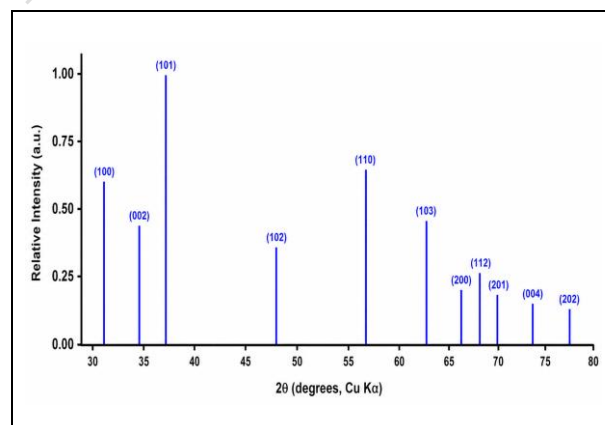


Fig. 2: Reference positions for wurtzite ZnO, this reference graphic is not the measured XRD pattern; measured data are shown in Fig. 6

2.4 EBT Adsorption and Illuminated-removal Experiments

At each pH, full EBT spectra were checked for spectral shifts before quantification near 530 nm in a 1 cm quartz cuvette. External standards covered 1–20 mg L^{−1}; acceptance criteria were calibration $R^2 \geq 0.995$ and quality-control results within $\pm 5\%$. Unless stated otherwise, 100 mL of 10 mg L^{−1} EBT and 2.0 g L^{−1} ZnO were stirred in a 250 mL temperature-controlled

borosilicate reactor at pH 10 and 35 °C. After 30 min in the dark, suspensions were exposed for 90 min to an AM1.5G solar simulator at a stated incident irradiance of 100 mW cm⁻². Samples were centrifuged and passed through 0.22 µm filters before UV–visible analysis. Table 1 distinguishes dark adsorption, direct photolysis, illuminated ZnO treatment and dye-stability controls. The solar simulator model, spectral range, lamp-to-reactor distance and illuminated area were not recorded in the source manuscript and require verification.

Variables tested were pH 2–12, ZnO dosage 0.1–2.0 g L⁻¹, initial EBT concentration 10–150 mg L⁻¹, irradiation time 0–150 min, temperature 15–45 °C and NaCl concentration 0–0.1 mol L⁻¹. The reported combination of 10 mg L⁻¹ EBT, 2.0 g L⁻¹ ZnO, pH 10, 35 °C, 30 min dark equilibration and 90 min irradiation was the best-performing combination tested; a formal

statistical optimization was not reported. Dissolved zinc was analyzed by ICP-OES at 213.857 nm after 0.22 µm filtration and acidification to 1% (v/v) HNO₃. Calibration standards covered 0.01–1.00 mg L⁻¹; the acceptance criteria were R² ≥ 0.995 and 90–110% matrix-spike recovery. The ICP-OES model, method detection limit and verification that the filtrate was particle-free were not supplied. Acidification was performed after filtration, because acidification before particle removal could dissolve residual ZnO.

Apparent EBT removal (%) = 100(C₀ – C_t)/C₀; q_t (mg g⁻¹) = (C₀ – C_t)V/m, where C₀ and C_t are measured dissolved EBT concentrations (mg L⁻¹), V is solution volume (L), and m is ZnO mass (g). These quantities represent loss from the measured liquid phase and do not identify the fate of EBT.

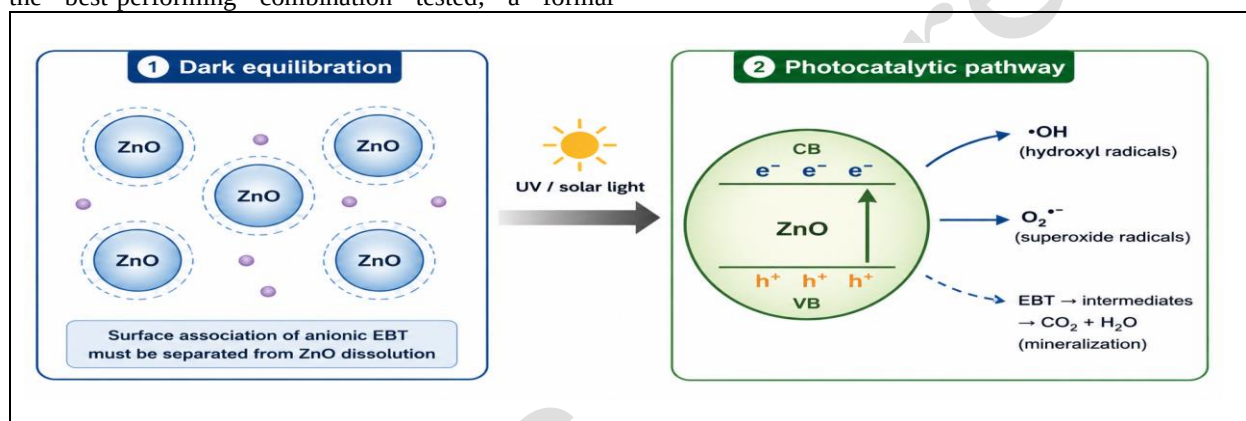


Fig. 3: Schematic of a possible light-associated pathway; reactive intermediates shown are hypothetical; they were not identified experimentally. The actual treatment controls are listed in Table 1

Table 1. Experimental controls used to distinguish apparent removal pathways and analytical blanks

Run	EBT	ZnO	Light	Purpose
A	Yes	Yes	No	Dark adsorption
B	Yes	Yes	Yes	Adsorption + photocatalysis
C	Yes	No	Yes	Direct photolysis
D	Yes	No	No	Dye stability
E	No	Yes	Yes	Catalyst/optical blank
F	No	Yes	No	Zn ²⁺ release without dye

2.5 Kinetic, Equilibrium and Stability Assessments

Dark adsorption data were assessed using pseudo-first-order (PFO), pseudo-second-order (PSO) and intraparticle-diffusion models. Equilibrium data were assessed using Langmuir and Freundlich isotherms. Illuminated-removal kinetics were measured after dark equilibration. TOC and COD were used to evaluate changes beyond the EBT chromophore signal, and dissolved zinc was monitored during five reuse cycles.

The source record does not specify the TOC/COD instruments and methods, the catalyst recovery, wash and drying steps, the mass carried into each cycle, or the reason for choosing five cycles; those details require author verification.

For model interpretation: PFO, $q_t = q_c[1 - \exp(-k_1t)]$; PSO, $t/q_t = 1/(k_2q_c^2) + t/q_c$; intraparticle diffusion, $q_t = k_3t^{1/2} + C$; Langmuir, $q_c = q_{\max}KLC_c/(1 + KLC_c)$; and Freundlich, $q_c = KFC_c^{1/n}$. Here q_c and C_c are equilibrium uptake and concentration. These empirical fits do not, by themselves, identify a reaction mechanism. The fitting method, residuals and parameter uncertainty should be reported from the original analysis files.

2.6 Statistical Analysis

ZnO was prepared in three independent batches. The manuscript reports 10 independent treatment trials per experimental condition, distributed across those batches, with at least duplicate analytical readings for each sample. Treatment trials are the experimental replicates; duplicate readings are technical replicates and should be averaged within a trial before statistical

comparison. TEM particle size was measured for 200 particles in separate image fields; the number of independent micrographs was not documented. Shapiro–Wilk and Levene tests were described for normality and variance homogeneity. One-way ANOVA with Tukey HSD, or Kruskal–Wallis when assumptions were not met, was specified at $p < 0.05$. The exact groups, test outputs and raw replicate values were not supplied, so no new claim of statistical significance is made here.

3. RESULTS AND DISCUSSION

The plant-mediated product showed a diffraction pattern consistent with wurtzite ZnO, an estimated 3.20 eV optical gap and a 27.6 ± 6.8 nm mean TEM particle diameter. Under the best-performing tested conditions, apparent EBT removal reached 92.4%, while TOC and COD decreased by 78.6% and 82.4%. The following sections examine what these measurements do and do not establish.

3.1 ZnO Structure and Morphology

Diffuse reflectance showed an absorption edge near 368 nm. A direct-transition Tauc extrapolation gave an approximate optical gap of 3.20 eV (Fig. 4). Because the fitting interval and uncertainty were unavailable, the small difference from a typical bulk-ZnO value should not be attributed to any specific defect or capping effect.

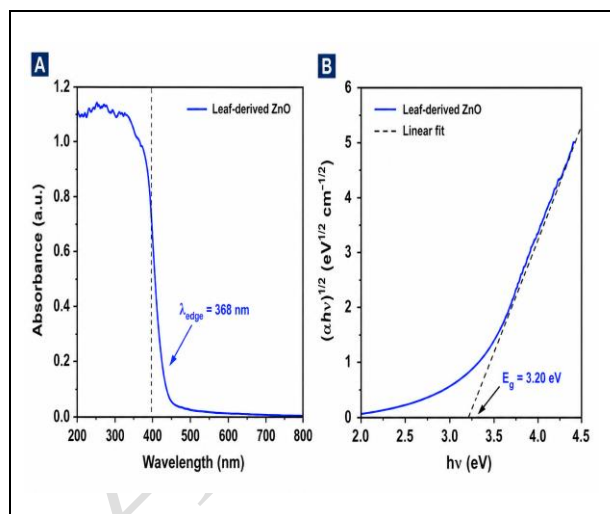


Fig. 4: Diffuse reflectance spectrum and Tauc plot for the prepared ZnO, the estimated edge is near 368 nm and the approximate gap is 3.20 eV

The leaf extract showed bands at 3410, 2924, 1635, 1384, 1043 and 612 cm^{-1} ; the uncalcined precursor showed bands at 3425, 1628, 1392 and 873 cm^{-1} . Calcined ZnO showed features at 3420, 1620 and 437 cm^{-1} (Fig. 5). The low-frequency feature is consistent with a Zn–O lattice vibration. The broad 3420 cm^{-1} feature indicates surface hydroxyl groups and/or adsorbed moisture; the 1620 cm^{-1} band may include a

water-bending contribution. FTIR alone does not establish EBT coordination or a reaction pathway.

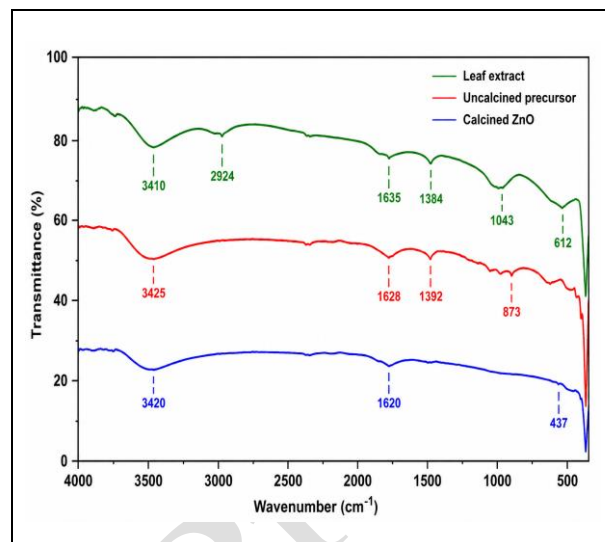


Fig. 5: FTIR spectra of leaf extract, precursor and ZnO after calcination

The measured powder XRD pattern (Fig. 6) was indexed to wurtzite ZnO with reflections assigned to (100), (002), (101), (102), (110), (103), (200) and (112). The strongest reported peak, near $2\theta = 36.25^\circ$, was assigned to (101). The source manuscript does not provide a complete numerical list of observed 2θ positions, a database card identifier or a crystallite-size calculation; those should be added from the original diffractogram if available. Fig. 2 shows reference peak positions only.

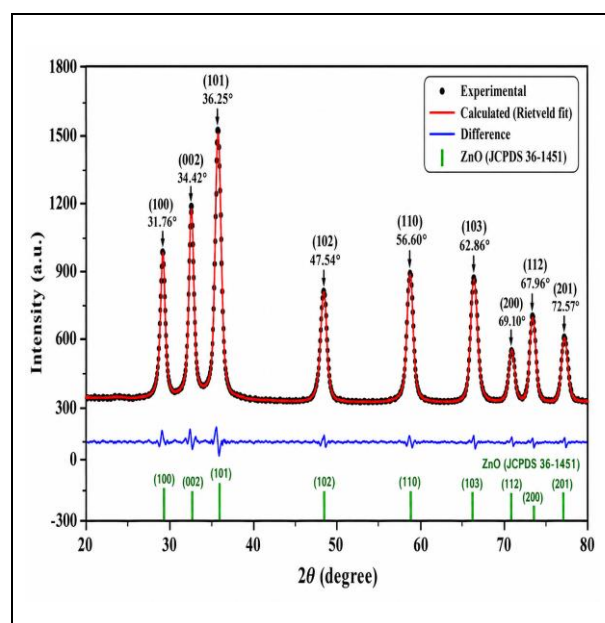


Fig. 6: Measured powder XRD pattern of the prepared material with wurtzite ZnO reference markers and fit residual

FESEM showed approximately spherical particles in agglomerates (Fig. 7). Local EDX analysis gave 77.99 wt% Zn and 22.01 wt% O (53.82 at% Zn and 46.18 at% O). These semiquantitative, local values do not establish bulk stoichiometry or zinc oxidation state.

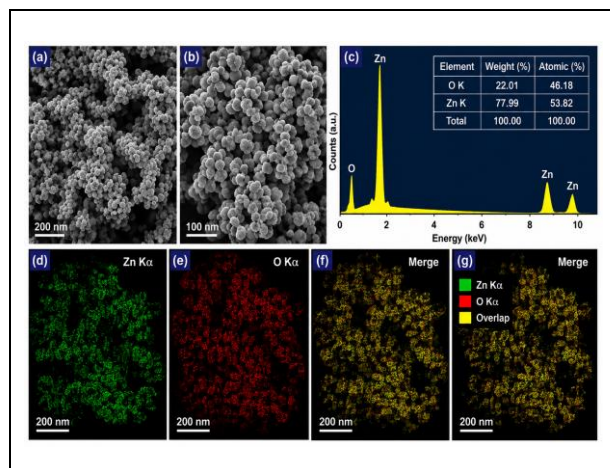


Fig. 7: FESEM images, EDX spectrum and composition, and Zn/O elemental maps

TEM analysis gave a mean primary-particle diameter of 27.6 ± 6.8 nm (200 particles; Fig. 8). This estimate is compatible with the larger agglomerates observed by FESEM. The reported HRTEM fringe spacing was approximately 0.28 nm. The plane annotation in the supplied figure is inconsistent with the tabulated d-spacings and requires checking against the original micrograph. The SAED pattern was described as polycrystalline.

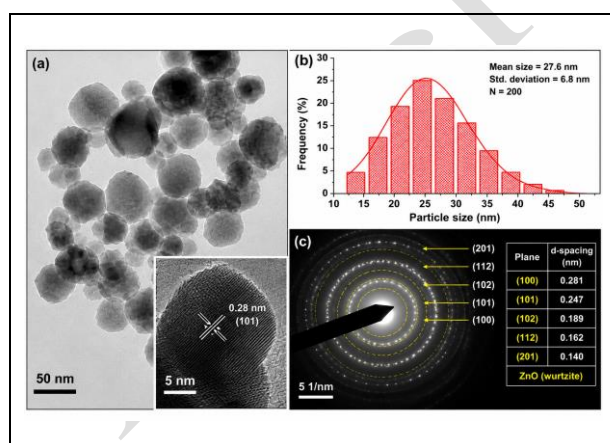


Fig. 8: TEM image, particle-size distribution (n = 200), HRTEM lattice-fringe inset and SAED pattern, the plane annotation on the HRTEM inset requires verification against the tabulated d-spacings

3.2 Adsorption, pH and ZnO Dissolution

The measured pHPZC was approximately 6.2 (Fig. 9). Below this value, protonation can favor electrostatic attraction of anionic EBT, while acidic ZnO

dissolution can complicate interpretation. Above pHPZC, deprotonation may weaken simple electrostatic attraction, yet the measured illuminated removal increased toward pH 10–11. EBT speciation, adsorption and light-driven processes may all contribute; the present controls do not quantify each contribution.

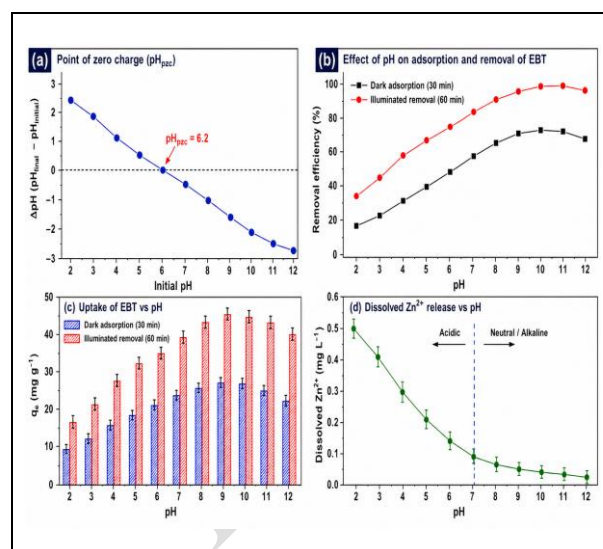


Fig. 9: Point of zero charge and effects of pH on dark adsorption, illuminated EBT removal, apparent uptake and dissolved zinc. Values are reported as mean $\pm$ SD

The plotted results indicate greater apparent removal near pH 10–11 and higher dissolved zinc under acidic conditions. This pattern argues against acid-promoted ZnO dissolution as the main explanation for the elevated alkaline removal, but the numerical zinc values by pH are needed to support a quantitative comparison. At alkaline pH, catalyst stability also needs confirmation beyond a single filtrate measurement.

3.3 Photochemical Interpretation and Limits

Light-excited ZnO may produce charge carriers capable of participating in redox reactions. The difference between illuminated ZnO treatment and the dark and direct-photolysis controls is consistent with a light-associated contribution. TOC and COD reductions indicate lower measured organic load, but adsorption and other pathways have not been fully separated. No scavenger, electron-paramagnetic-resonance or transformation-product analysis was reported; therefore, no specific reactive oxygen species or complete mineralization is claimed.

3.4 Kinetics, Equilibrium and Reuse

The reported PSO fit had a higher R^2 than the PFO fit, but this alone cannot establish chemisorption. Parameter plausibility, residuals and uncertainty must also be assessed. The source record lacks the raw series

required for a more discriminating error analysis; model comparisons are therefore descriptive.

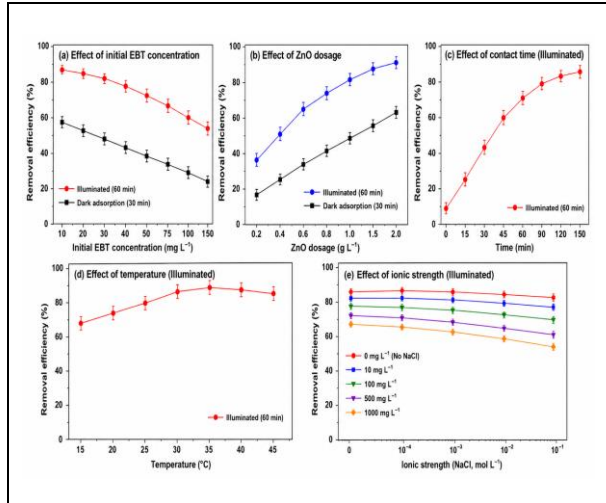


Fig. 10: Effects of initial EBT concentration, ZnO dosage, illuminated contact time, temperature, and ionic strength. Values are mean $\pm$ SD ($n = 10$ independent replicates)

Apparent removal decreased as initial EBT concentration increased and generally increased with ZnO dose over the tested range (Fig. 10). The response approached a plateau after approximately 90–120 min. The highest observed removal occurred at 30–35 °C, which may reflect competing temperature effects on adsorption and reaction rates; the data do not establish a unique thermal mechanism. Added NaCl reduced removal under the tested conditions, consistent with possible electrostatic screening and competitive effects, but those mechanisms were not isolated experimentally.

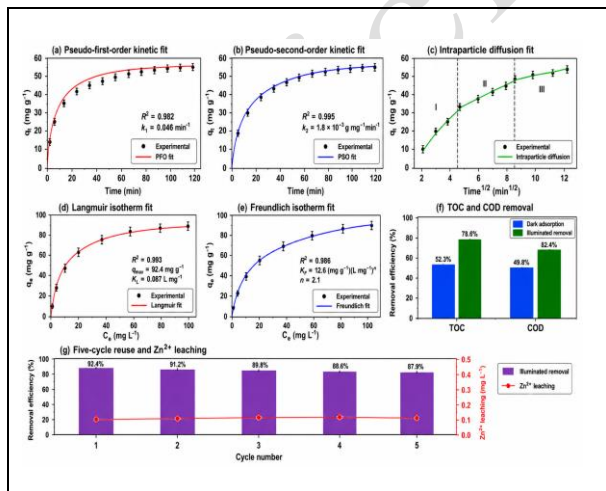


Fig. 11: Kinetic and isotherm fits and measured TOC and COD changes; model curves are descriptive; fit residuals and uncertainties should be added from the underlying data

At the best-performing tested conditions, apparent illuminated EBT removal averaged 92.4% over

10 independent trials. Reported PFO fitting gave $R^2 = 0.982$ and $k_1 = 0.046 \text{ min}^{-1}$; the PSO fit gave $R^2 = 0.995$. The Langmuir fit gave $R^2 = 0.993$ and $q_{\text{max}} \approx 92.4 \text{ mg g}^{-1}$, while the Freundlich fit gave $R^2 = 0.986$, $K_F \approx 12.6$ and $n \approx 2.1$ (Fig. 11). The 92.4% removal and 92.4 mg g^{-1} fitted capacity are different quantities: at 10 mg L^{-1} EBT and 2.0 g L^{-1} ZnO, the maximum mass-balance uptake is only 5 mg g^{-1} . The fitted q_{max} therefore cannot describe uptake in that single experiment and requires verification against the full equilibrium data. TOC and COD decreased by 78.6% and 82.4% during illuminated treatment, compared with 52.3% and 49.8% in dark trials. These bulk measures support a decrease in measured organic load but do not prove complete mineralization. Across five reported reuse cycles, apparent illuminated removal declined from 92.4% to 87.9%, a loss of 4.5 percentage points. The manuscript reports dissolved zinc near 0.1 mg L^{-1} during reuse, but catalyst recovery and zinc mass balance are insufficiently described to establish long-term stability.

3.5 Comparison with Published ZnO–EBT Systems

Table 2 summarizes selected ZnO–EBT studies. The synthesis route, dose, initial dye concentration, pH, illumination and treatment time differ. The table therefore provides context rather than a direct efficiency ranking.

Table 2. Selected ZnO systems evaluated for Eriochrome Black T removal under differing experimental conditions

System	Conditions	Reported Outcome	Relevance
Precipitated ZnO (~20 nm)	—	fastest decomposition at pH 11	pH benchmark
Sadollahkhani and Kazeminezhad (2014)	sunlight; pH 4, 8, 11	complete discoloration by 120 min; Pr/Ce mineralization by 180 min	doped comparator
Pr/Ce/Eu–ZnO — Vaiano <i>et al.</i> (2017)	UV irradiation	83% in 20 min	optimized UV benchmark
ZnO — Lanjwani <i>et al.</i> (2023)	0.3 g L^{-1} ; 25 mg L^{-1} EBT; pH 7.91; 96 W UV	complete degradation in 90 min	condition dependence
Thermal-oxalate ZnO — Kaur and Singhal (2015)	1 g L^{-1} ; optimized acidic condition	98.5% degradation	modified-catalyst comparator
5% Ag/ZnO — Shaikh <i>et al.</i> (2023)	UV; 90 min	up to ~92.4% removal	leaf-mediated catalyst
C. dactylon leaf ZnO — Present study	10 mg L^{-1} EBT; 2.0 g L^{-1} ZnO; pH 10; 35		

System	Conditions	Reported Outcome	Relevance
	°C; AM1.5G, 100 mW cm ⁻² ; 30 min dark + 90 min light; n = 10		

The specific contribution of this study is to evaluate *C. dactylon*-mediated ZnO with dark, direct-photolysis and illuminated-treatment controls while also reporting organic-load indicators, dissolved zinc and short-term reuse. Missing recovery and mechanism measurements limit the strength of the comparison.

4. CONCLUSIONS

C. dactylon leaf extract was used to prepare ZnO with a diffraction pattern consistent with wurtzite. Apparent EBT removal reached 92.4% under the best-performing tested conditions and fell to 87.9% after five reuse cycles, a 4.5 percentage-point decline. TOC and COD also decreased, although neither complete mineralization nor specific reactive species were established. Removal was greatest near pH 10–11, while dissolved zinc was highest under acidic conditions; full zinc-by-pH data and stability tests are needed to interpret this relationship quantitatively. The combined control design is useful for assessing a plant-mediated ZnO system, but validation in real wastewater must address matrix effects, catalyst recovery and zinc release.

Funding: No specific grant was reported from public, commercial or nonprofit funding agencies.

ETHICS AND PLANT-MATERIAL STATEMENT

This study did not involve human participants or animals. *C. dactylon* was collected in the Dammam region, Eastern Province, Saudi Arabia, from a site accessible to the authors. The species is a common grass, and collection was limited to the amount required for laboratory research. The authors are responsible for confirming compliance with any institutional or site-specific collection requirements before submission.

DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author on reasonable request. The published figures and tables contain the summarized data used in the present analysis.

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This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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