



RESEARCH PAPERS

Cerium Loading Redistributes Tetracycline Removal between Dark Adsorption and the UV Stage on Hydrothermal Niobium Oxide

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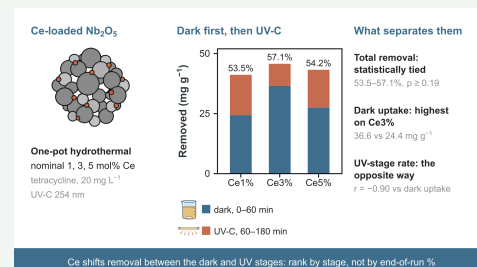
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ABSTRACT

Photocatalysts that also adsorb are usually ranked by how much pollutant disappears at the end of a run, a number that says nothing about which of the two processes did the work. We separate the two on a single material family by running a dark stage and an illuminated stage in sequence and following the solution in-line. Niobium oxide with nominal Ce loadings of 1–5% was prepared in one hydrothermal step from ammonium niobate oxalate and cerium nitrate, and tested against 20 mg L⁻¹ tetracycline under 254 nm irradiation. Total removal after 180 min did not differ significantly across the three compositions, at (53.5 ± 0.7) %, (57.1 ± 1.9) % and (54.2 ± 3.2) % for Ce1%-Nb₂O₅, Ce3%-Nb₂O₅ and Ce5%-Nb₂O₅ respectively (Welch $p \geq 0.19$). The dark stage did separate them: uptake before illumination was (24.4 ± 0.7) mg g⁻¹, (36.6 ± 0.8) mg g⁻¹ and (27.5 ± 0.4) mg g⁻¹ ($p \leq 0.047$ for every pair, $n = 2$), and the apparent first-order constant of the UV stage fell as dark uptake rose ($r = -0.90$, $p = 0.014$, $n = 6$). Direct photolysis cannot produce that spread, because it would be fastest where the solution is most dilute, and dark uptake was close to its plateau by lamp-on, so the spread is attributable to the solids. Ce loading therefore redistributes removal between dark uptake and the UV stage rather than changing the total, so a fixed-time removal percentage cannot rank these materials, whereas dark-stage uptake separates them.



KEYWORDS niobium pentoxide; cerium loading; tetracycline; sequential dark and UV stages; stage-resolved removal

1. INTRODUCTION

Tetracyclines are among the most heavily used antibiotic classes worldwide, and between 40 % and 90 % of an administered dose leaves the body still biologically active [1]. Wastewater treatment plants pass a measurable fraction to receiving waters: a synthesis of more than 170 Chinese field studies puts median antibiotic concentrations at 15 ng L⁻¹ in treatment-plant effluent and 8.1 ng L⁻¹ in surface water, with tetracycline among the seven compounds detected in nearly every environmental compartment [2]. Composting and conventional biological treatment remove part of the load but leave transformation products and resistance genes whose fate is poorly resolved [1], and the advanced options (oxidation, membranes and adsorption) have been reviewed for tetracycline specifically [3]. Photocatalysis is attractive here because it attacks the molecule rather than relocating it [4], and it has been demonstrated on tetracycline and on other pharmaceuticals over Nb₂O₅-based systems, including levofloxacin, norfloxacin and acetaminophen [5–9].

Nb₂O₅ is a useful host for this chemistry because it both binds and degrades, and because it survives repeated use [10]. Hydrothermal synthesis from an ammonium niobate complex yields a deformed orthorhombic phase that adsorbs cationic dyes by cation exchange and then mineralises them under UV [11, 12]. The same route, run for 72 h, gives a high-surface-area *ortho*-Nb₂O₅ that takes up 32 mg g⁻¹ tetracycline at equilibrium and degrades it under UV [13], and the pairing of uptake with photolytic regeneration has since been reproduced on Nb₂O₅ nanofibre membranes [14] and on Nb₂O₅-modified clays [15]. The 3.28 eV band gap of *ortho*-Nb₂O₅ [13] restricts activation to the UV, which is why doping has become the standard lever. Metal (Zn, Mo, Bi) and non-metal (N) dopants have each been shown to shift the optical response or the defect population of Nb₂O₅ [16–21], alongside the heterojunction routes

that dominate the recent literature [22]. Cerium is a natural candidate because the Ce³⁺/Ce⁴⁺ couple supplies oxygen vacancies that act as charge traps [23, 24], and the reciprocal composition, Nb-doped CeO₂, degrades tetracycline about twice as fast as undoped CeO₂ [25]. Ceria is itself a common doping host [26, 27]; Ce added to Nb₂O₅ is the arrangement examined here.

What these studies share is the metric. Performance is reported as the fraction of pollutant lost from solution after a fixed irradiation time, occasionally with an apparent rate constant attached [5, 17, 28]. On a material that adsorbs strongly the metric is ambiguous by construction, because two mechanisms with different consequences, transfer to a surface and transformation under illumination, are summed into one number [14]. The ambiguity matters for doping series in particular: a dopant that raises surface affinity and a dopant that raises photochemical activity both move the same percentage upward, and ranking a series by that percentage cannot tell which happened. Studies that do separate the stages usually do so to subtract adsorption as a nuisance term before fitting kinetics. To our knowledge, no doped or metal-loaded Nb₂O₅ series has reported dark-stage uptake and UV-stage removal as a paired property of each composition, and that is the gap this work addresses.

Herein we resolve removal into its dark and UV stages for a Ce-loaded niobium oxide series by following the solution in-line through a dark stage and an illuminated stage, and by renormalising every trace to its own measured initial concentration. UV-C is used deliberately: the question is how removal divides between the stages, not how far the response extends into the visible. The three compositions turn out not to differ significantly in total removal but to differ clearly in how that total is split, with dark uptake and the apparent UV-stage rate constant moving in opposite directions across the series.

2. MATERIALS AND METHODS

Fig. 1 summarises the synthesis and the reactor.

2.1 Synthesis

Ammonium niobate(V) oxalate hydrate (2.244 g) was dissolved in 70 mL deionised water under magnetic stirring at 200 rpm and room temperature. Cerium(III) nitrate hexahydrate was then added at nominal Ce loadings of 1, 3 and 5 mol % relative to Nb, and the solution was stirred to homogeneity. Each solution was transferred to a 100 mL polytetrafluoroethylene (PTFE)-lined autoclave and held at 175 °C for 72 h. The solid was recovered by vacuum filtration, dried at 80 °C for 4 h, ground in an agate mortar and stored in sealed glass vials. The three products are denoted Ce1%–Nb₂O₅, Ce3%–Nb₂O₅ and Ce5%–Nb₂O₅. The Ce content of the products was not determined, so the loadings are nominal throughout. The niobium precursor, temperature and duration reproduce the route reported for undoped *ortho*-Nb₂O₅ [12, 13], so that, apart from the added cerium nitrate, the synthesis matches that benchmark; no undoped material was prepared in this campaign.

2.2 Characterisation

X-ray diffraction (XRD) patterns were collected over 5–80° 2θ. Infrared spectra were recorded over 400–4000 cm^{−1} in transmittance mode. Surface morphology was imaged by scanning electron microscopy (SEM) at 1000–10 000×. All three measurements were made on a commercial contract basis before the removal experiments.

2.3 Removal Experiments

A 500 mg L^{−1} tetracycline hydrochloride stock was prepared in deionised water and diluted to working concentrations with no buffer or background electrolyte, so the ionic strength is set by the salt alone, about 4 × 10^{−5} mol L^{−1} at 20 mg L^{−1}. Solution pH was neither adjusted nor recorded. Tetracycline has pK_a values near 3–4, 7–8 and 9–10 [29]; without a measured pH its speciation is not known, and we do not use it in the interpretation. Absorbance was read at the tetracycline maximum, 273 nm, located by a 230–400 nm scan of a standard, with a Shimadzu UV-1280 spectrophotometer calibrated over 5–25 mg L^{−1}.

Runs were carried out in the closed photoreactor described in [25, 26], fitted here with a single 10 W Philips TUV UV-C lamp (λ = 254 nm) about 5 cm above the solution surface. The suspension was held in a 325 mL Pyrex dish of 10 cm internal diameter, giving an irradiated area of about 79 cm² and a liquid depth of about 2.5 cm at 200 mL, and stirred magnetically at 300 rpm. Lamp output and photon flux were not measured. Runs started at room temperature, and the temperature of the closed reactor, which also houses the lamp, was neither controlled nor recorded. A peristaltic pump drew the solution through an inert PTFE membrane filter, which kept the solid in the dish, and circulated the filtrate through the flow cell of the spectrophotometer, which recorded absorbance automatically every 5 min; the same in-line arrangement has been used to follow dye removal over doped Nb₂O₅ [16]. The contact-time analysis uses the readings at 0, 5, 15, 30, 45 and 60 min in the dark and every 15 min under illumination, the times retained in the original workbook; the initial-concentration analysis uses every 5 min reading.

Two experiments were run, both with 0.05 g solid in 200 mL solution. Each condition was run in duplicate as two independent runs, each with fresh solid and freshly diluted solution; values are the mean of the two runs. The ± values in the text and tables, and the error bars on single-value plots, are the standard deviation of the two runs; the error bars on time traces span the two runs. In the *contact-time* experiment, all three compositions were exposed to 20 mg L^{−1} tetracycline for 60 min in the dark, after which the lamp was switched on and the run continued to 180 min, the illuminated stage. In the *initial-concentration* experiment, Ce3%–Nb₂O₅ was exposed to 10, 15, 20 and 25 mg L^{−1} tetracycline under continuous illumination for 60 min, with no dark stage.

2.4 Data Treatment

Absorbances were converted to concentrations through the calibration line $A = 0.03264 C - 0.00480$ ($R^2 = 0.9997$; limit of quantification 1.7 mg L^{−1}), refitted here from the raw standards. Removal was referred to the *measured* initial concentration of each run,

$$\eta(t) = \frac{C_0 - C(t)}{C_0} \times 100\%, \quad (1)$$

rather than to the nominal make-up concentration. The distinction is not cosmetic: measured C_0 ranged over 19.0–20.3 mg L^{−1} across nominally identical 20 mg L^{−1} runs, and normalising to the nominal value transfers that solution-preparation scatter into the reported efficiency.

Removal was decomposed into the two stages of the contact-time run,

$$q_{\text{dark}} = \frac{(C_0 - C_{60})V}{m}, \quad q_{\text{UV}} = \frac{(C_{60} - C_{180})V}{m}, \quad (2)$$

with $V = 0.200$ L and $m = 0.050$ g. Apparent first-order constants were fitted to the illuminated window alone, $\ln(C_{60}/C_t) = k_{\text{app}}(t - 60 \text{ min})$, so that the dark stage does not enter the fit. Candidate kinetic models were fitted to each run separately: pseudo-first order [30], pseudo-second order [31] and Elovich [32] to the dark stage, and first-, zero- and second-order decay to the illuminated stage. Candidates were ranked by the small-sample corrected Akaike information criterion (AICc) [33], which the six points per dark-stage fit make the appropriate form, with both parameters free in every candidate so the comparison is like-for-like. The first-order constants from those two-parameter fits agree with the anchored k_{app} of Table 1 to within the replicate spread. Composition means were compared by Welch's t -test [34] on the duplicates; with $n = 2$ these p -values are indicative rather than confirmatory and are reported alongside the replicate spread. The analysis scripts and the intermediate tables are archived with the raw workbook.

3. RESULTS AND DISCUSSION

3.1 Structure and Surface Groups

All three patterns (Fig. 2a) carry the same reflection set: a sharp line at 22.6°, weaker lines at 46.2° and 54.8°, a broad hump centred near 26.5°, and a pair of narrow lines at 16.15° and 16.67°. The 22.6° and 46.2° reflections are those of the deformed orthorhombic phase obtained by this route [12, 35]. The low-angle pair is not part of that pattern and is markedly narrower than it (full widths of 0.31–0.36° against 0.62–0.66° at 22.6°), so it belongs to a second, better-ordered phase, plausibly an ammonium-bearing layered niobate carried through from the precursor [11]. We do not assign it further here; a database search-match on the full pattern, including an undoped reference, is needed for that. Apparent crystallite sizes from the 22.6° width [36], uncorrected for instrumental broadening, are 12.3 nm, 12.5 nm and 13.1 nm for Ce1%–Nb₂O₅, Ce3%–Nb₂O₅ and Ce5%–Nb₂O₅.

What changes across the series is the balance between the two phases, and it changes only at the top loading. Ce1%–Nb₂O₅ and Ce3%–Nb₂O₅ are indistinguishable: the intensity of the 16.15° line relative to the 22.6° niobate reflection is 0.63 and 0.61, and the ratio within the low-angle pair is 0.88 and 0.87. In Ce5%–Nb₂O₅ the same ratios are 1.71 and 1.32, a 2.7-fold growth of the low-angle phase against the niobate, while the integrated intensity over 10–60° falls by 44 % relative to Ce1%–Nb₂O₅. At 5 % nominal loading, Ce is therefore not accommodated without changing the product: it shifts the product towards the secondary phase and costs long-range order, consistent with the disorder reported for heavily doped Nb₂O₅ and for the reciprocal Nb–CeO₂ system [25, 37]. No reflection appears at 28.5°, 33.1° or 47.5°, so segregated fluorite CeO₂ is below the detection limit in all three samples.

Infrared spectra (Fig. 2b) show the Nb–O and Nb–O–Nb envelope at 480 cm^{−1} and 560 cm^{−1}, a band at 1415–1420 cm^{−1} assigned to the ammonium deformation, the water bending mode near 1640 cm^{−1}, and a broad hydroxyl stretch centred between 3235 cm^{−1} and 3363 cm^{−1}; the sharp feature at 2350 cm^{−1} is atmospheric CO₂. Retained ammonium appears in the undoped reference spectrum as well, which supports the cation-exchange picture established for this material [12] but does not by itself discriminate between the three Ce loadings.

Electron microscopy (Fig. 3) shows the same texture in all three samples: irregular agglomerates built from sub-micrometre primary particles, with no faceted crystals resolved at 10 000×. The 1000× fields differ in apparent packing density, Ce5%–Nb₂O₅ looking the finest-grained of the three, but these are single fields recorded without particle-size statistics and we treat the difference as qualitative. None of the three techniques varies monotonically with the uptake ranking of Section 3.2: Ce1%–Nb₂O₅ and Ce3%–Nb₂O₅, of which Ce3%–Nb₂O₅ takes up 50 % more in the dark, are indistinguishable by diffraction, by infrared spectroscopy and by microscopy alike. We return to this in Section 3.5.

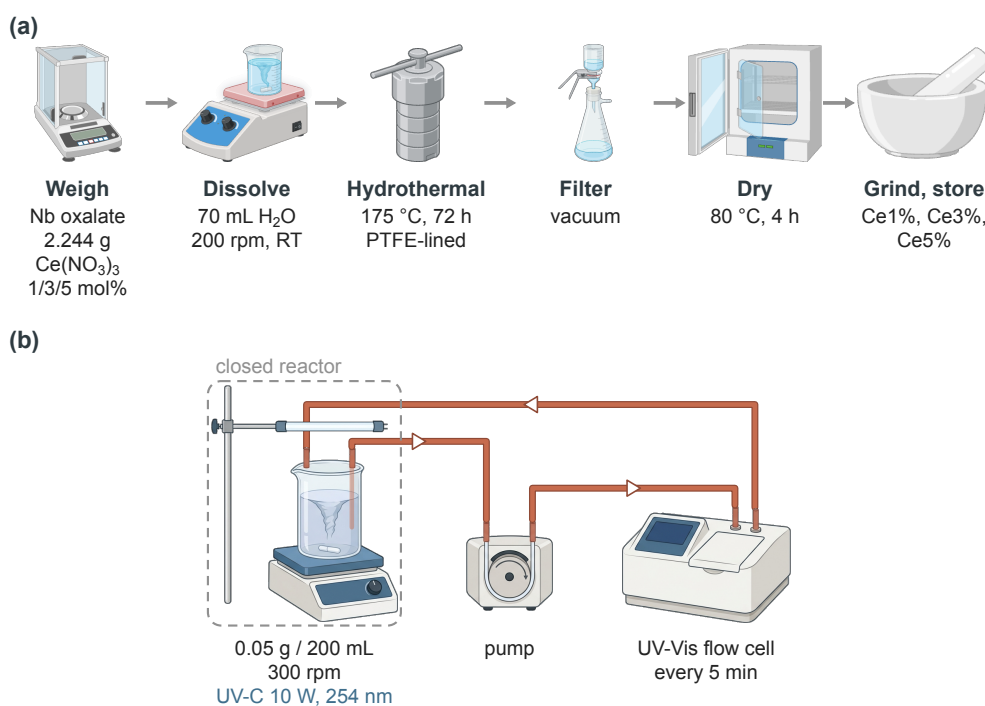


Figure 1. (a) One-pot hydrothermal synthesis of the Ce-loaded Nb₂O₅ series. (b) Closed reactor with in-line UV-Vis readout; arrowheads, flow direction; dashed outline, closed reactor housing the lamp.

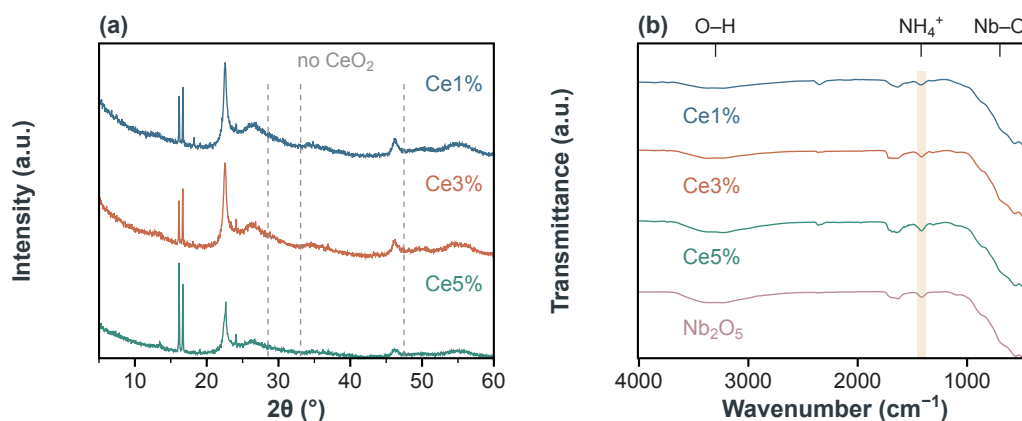


Figure 2. (a) XRD patterns; dashed lines, fluorite CeO₂ reflection positions. (b) Infrared transmittance spectra, including undoped Nb₂O₅; shaded band, ammonium deformation region. Both panels are offset vertically.

3.2 Removal Traces and the Two Stages

Fig. 4a shows the full 180 min traces. Removal rises steeply for the first 15 min in the dark, flattens towards 60 min, and then climbs again under illumination to the end of the run. The first illuminated reading, at 75 min, lies below the 60 min value in four of the six runs, by 0.2–2.1 pp and most clearly on Ce3%–Nb₂O₅ (1.4 pp and 2.1 pp), and above it by 0.2–0.6 pp in the other two. We read this dip as a change in the measured absorbance rather than as a pause in removal: tetracycline photolysis intermediates absorb in the same region as the parent compound, so a single-wavelength reading cannot separate parent loss from the appearance of absorbing products (Section 3.5).

The totals do not separate the materials. End-of-run removal spans 53.5–57.1 % (Table 1), a range of 3.7 pp against a replicate standard deviation of up to 3.2 pp (Ce5%–Nb₂O₅, whose two runs differ by 4.5 pp); no pairwise comparison approaches significance (Welch $p = 0.19, 0.79$ and 0.41 for the pairs Ce1/Ce3, Ce1/Ce5 and Ce3/Ce5). Ranking this series by end-of-run efficiency would therefore rank replicate scatter.

Dark uptake does separate them, and the UV stage moves the opposite way (Fig. 5). Dark uptake is $(24.4 \pm 0.7) \text{ mg g}^{-1}$ for Ce1%–

Nb₂O₅, $(36.6 \pm 0.8) \text{ mg g}^{-1}$ for Ce3%–Nb₂O₅ and $(27.5 \pm 0.4) \text{ mg g}^{-1}$ for Ce5%–Nb₂O₅; every pair is separated ($p = 0.004, 0.047$ and 0.012 , same pair order), and the Ce3%–Nb₂O₅ advantage over Ce1%–Nb₂O₅ is 12.2 mg g^{-1} against replicate standard deviations of at most 0.8 mg g^{-1} . What is captured in the dark is then no longer in solution for the UV stage to remove: q_{UV} runs the other way, at 16.8 mg g^{-1} , 9.2 mg g^{-1} and 15.7 mg g^{-1} , and the apparent rate constant follows it down, from $3.52 \times 10^{-3} \text{ min}^{-1}$ on Ce1%–Nb₂O₅ to $2.30 \times 10^{-3} \text{ min}^{-1}$ on Ce3%–Nb₂O₅. Across the six individual runs, k_{app} correlates negatively with dark uptake ($r = -0.90, p = 0.014$).

The campaign has no photolysis blank (Section 3.5), so the UV-stage numbers include direct photolysis of tetracycline at 254 nm [38, 39]. That term cannot, however, produce the spread between compositions. All six runs used the same lamp, reactor geometry, volume and solid mass, so direct photolysis can differ between runs only through the absorbance of the solution and through any effect of the solids on the light or the solution. Its pseudo-first-order constant can only stay level or fall as the solution absorbs more of the light, which would make the runs with the lowest C_{60} the fastest. The opposite is observed: Ce3%–Nb₂O₅,

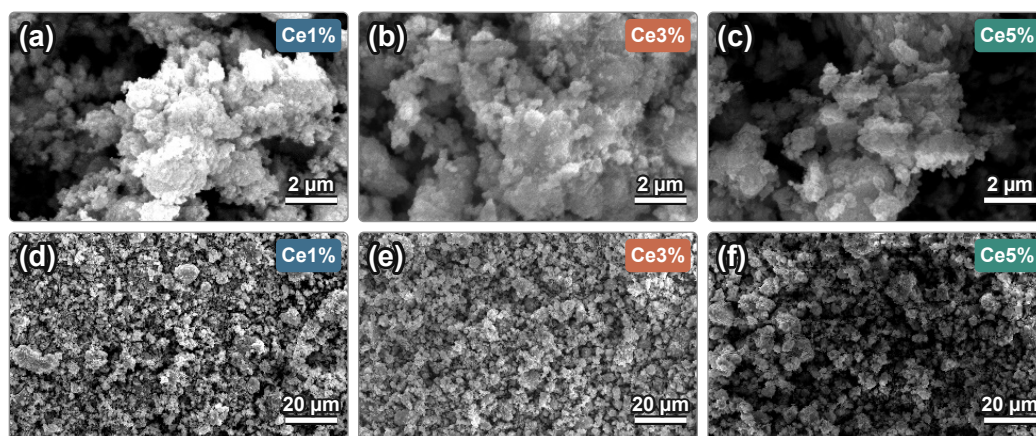


Figure 3. SEM micrographs (secondary electrons, 15 kV, working distance 11 mm) at 10 000× (a–c) and 1000× (d–f). Scale bars redrawn from the instrument calibration.

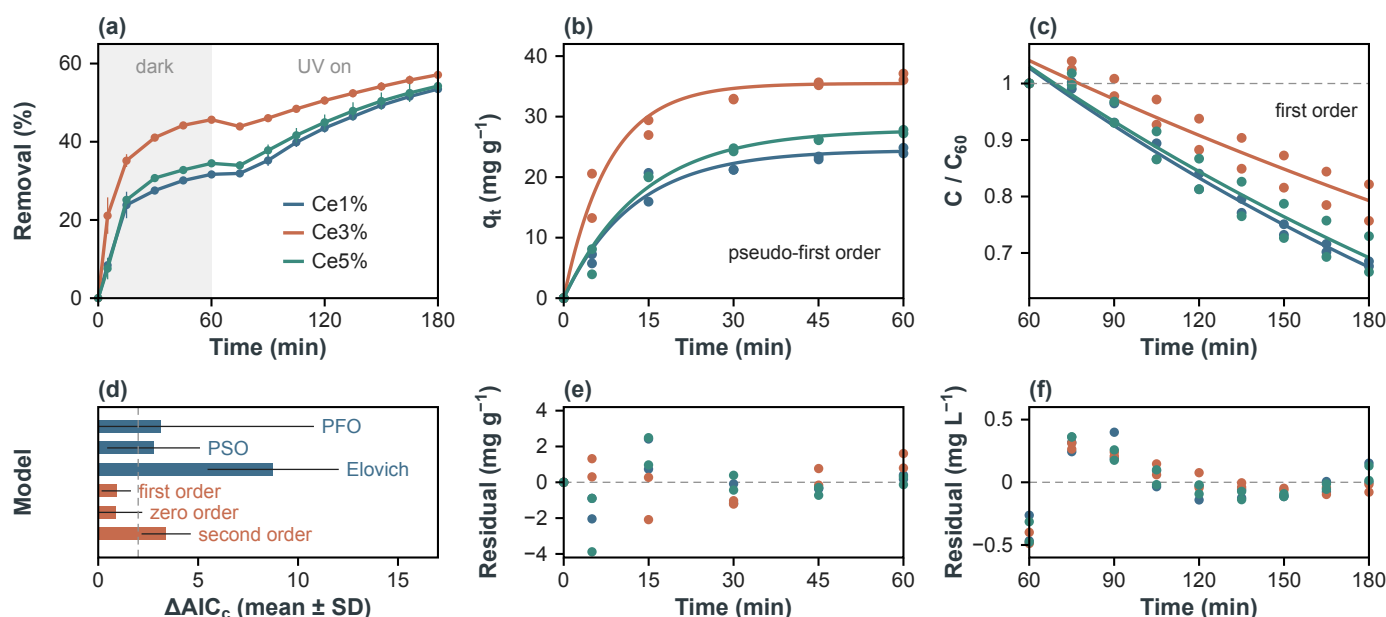


Figure 4. Tetracycline removal at 20 mg L⁻¹, all panels. (a) Traces; shaded, dark stage; points, duplicate means; bars, range of the two runs. (b) Dark-stage uptake with pseudo-first-order fits. (c) Illuminated stage normalised to C_{60} , with first-order fits; dashed line, $C/C_{60} = 1$. (d) Model comparison across the six runs; dashed line, $\Delta AIC_c = 2$. (e, f) Residuals of the fits in (b) and (c). Colours as in (a).

Table 1. Stage-resolved removal at 20 mg L⁻¹ tetracycline, 0.05 g solid in 0.200 L. Mean ± standard deviation of duplicate runs; k_{app} fitted over 60–180 min only.

Sample	q_{dark} (mg g ⁻¹)	q_{UV} (mg g ⁻¹)	η_{180} (%)	k_{app} (10 ⁻³ min ⁻¹)
Ce1%–Nb ₂ O ₅	24.4 ± 0.7	16.8 ± 0.5	53.5 ± 0.7	3.52 ± 0.04
Ce3%–Nb ₂ O ₅	36.6 ± 0.8	9.2 ± 2.1	57.1 ± 1.9	2.30 ± 0.47
Ce5%–Nb ₂ O ₅	27.5 ± 0.4	15.7 ± 1.8	54.2 ± 3.2	3.33 ± 0.53

with the lowest C_{60} (10.8–11.0 mg L⁻¹ against 12.8–13.4 mg L⁻¹ for the other two), has the smallest k_{app} . Photolysis therefore contributes no more than the smallest single-run constant, 1.97×10^{-3} min⁻¹, in any run, and if anything more on Ce3%–Nb₂O₅ than on Ce1%–Nb₂O₅, so the 1.22×10^{-3} min⁻¹ difference between them is a lower bound on the part that depends on the solids. Nor is it the dark-stage adsorption continuing: the pseudo-first-order plateau of the dark stage (Section 3.3) exceeds the measured 60 min uptake by at most 0.3 mg g⁻¹, against UV-stage removal of 9.2–16.8 mg g⁻¹. Whether the solid-dependent part is photocatalytic turnover, light-induced exchange at the surface, or a difference in how the solids attenuate the lamp light inside the reactor is not resolved by these data.

The normalisation of k_{app} does not explain the k_{app} – q_{dark} anticorrelation either: k_{app} is referred to the solution concentration at 60 min, which

is lowest on Ce3%–Nb₂O₅, so a fixed absolute removal rate would give Ce3%–Nb₂O₅ the largest constant, not the smallest; its absolute UV-stage removal is also the smallest of the three (Table 1). One reading consistent with this is that the adsorbed layer blocks the surface sites that generate or transfer reactive species, so that the composition holding most tetracycline removes the remainder slowest under UV; site blocking of this kind is the usual explanation when uptake and turnover trade off on a bifunctional oxide [14]. It cannot be confirmed without a measurement of what sits on the surface, which the present dataset does not contain.

For an external reference point, undoped *ortho*-Nb₂O₅ from the same synthesis route takes up 32 mg g⁻¹ tetracycline at equilibrium [13]. The Ce3%–Nb₂O₅ value of 36.6 mg g⁻¹ is reached in 60 min at a single initial concentration, so the two numbers are not measured on the same basis and the comparison is indicative only, and the undoped material is a

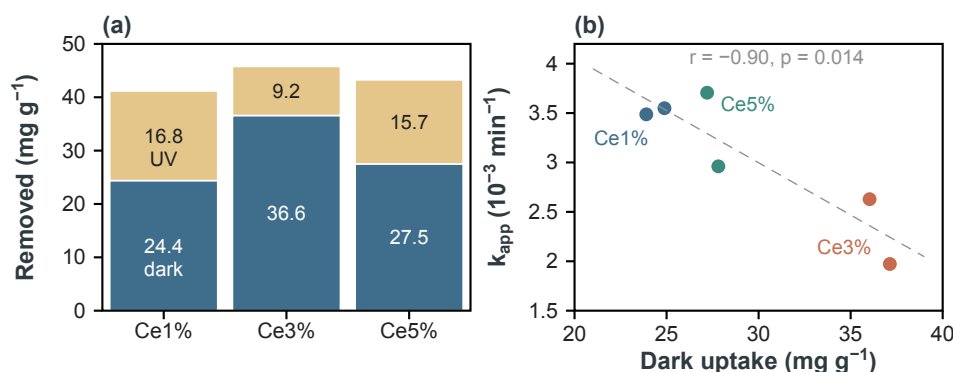


Figure 5. (a) Removal split by stage, mass basis; values in mg g⁻¹. (b) Apparent rate constant against dark uptake, one point per run; dashed line, least-squares fit.

benchmark, not a control. It does indicate that the Ce-loaded materials are in the same capacity band as the undoped host rather than an order apart.

3.3 Kinetic Models and Their Residuals

Both models that dominate the adsorption literature reproduce the dark stage (Fig. 4b): pseudo-first and pseudo-second order return R^2 of 0.988 and 0.984 and root-mean-square errors (RMSE) of 1.05 mg g⁻¹ and 1.10 mg g⁻¹. Pseudo-first order has the lowest AICc in five of the six runs, but AICc does not separate the two (Fig. 4d; mean ΔAICc 3.1 and 2.8, with replicate spreads of 7.7 and 2.3), while Elovich is worse by a margin that survives the spread (ΔAICc 8.8 ± 3.3). Reading a marginally better pseudo-second-order fit as evidence of chemisorption is routine, and it is a step taken across this literature on layered double hydroxides, desilicated zeolite, mixed metal oxides and printed monoliths [40–44], including on Nb₂O₅ itself [45]. On the present dataset it is not available: the two models are separated by less than the spread between replicates, so the ranking carries no mechanistic information, and we do not read one into it. The pseudo-first-order plateau, 24.5 mg g⁻¹, 35.5 mg g⁻¹ and 27.8 mg g⁻¹ for Ce1%–Nb₂O₅, Ce3%–Nb₂O₅ and Ce5%–Nb₂O₅, reproduces the measured 60 min uptake of Table 1 to within 1.1 mg g⁻¹, so the dark stage is close to its own plateau when the lamp comes on.

The illuminated stage behaves the same way (Fig. 4c). First and zero order are indistinguishable (ΔAICc 0.90 ± 0.73 and 0.85 ± 1.35) and only second order is rejected (3.40 ± 1.23). Over a window in which the concentration falls by a fifth to a third, an exponential and a straight line are barely different curves; that is a property of the window, not of the material.

The residuals are more informative than the ranking. In the dark stage (Fig. 4e) the pseudo-first-order residuals are largest at 5 min and 15 min, spanning -3.9 – 2.5 mg g⁻¹, and their sign there differs between runs: the misfit is concentrated in the steep first quarter of the stage. In the illuminated stage (Fig. 4f) they are not scattered but ordered in time: negative at 60 min, positive through 75–90 min, then decaying towards zero. Random error does not produce that shape. It is the absorbance excursion that follows lamp-on (Section 3.2), and it sets a floor on how far any rate constant taken from this window can be pushed.

3.4 Initial Concentration

Fractional removal after 60 min rises with initial concentration, from 21.7% at 10.1 mg L⁻¹ to 41.5% at 24.6 mg L⁻¹ (Table 2). Read as a performance statement, as removal efficiency against initial concentration routinely is [5, 6, 28], this is anomalous, since a system limited by a fixed flux of reactive species loses a smaller fraction of a larger load.

The time traces show that it is an artefact of the fixed window (Fig. 6a). At 10.1 mg L⁻¹ the trace is flat over the last quarter of the run, (0.06 ± 0.09) pp min⁻¹, so the system has stopped changing; at 24.6 mg L⁻¹ it is still rising at (0.21 ± 0.03) pp min⁻¹ when the run is stopped. A 60 min percentage therefore compares a system that has reached its plateau against one that has not, and the comparison rewards the latter. On a mass basis the picture is straightforward (Fig. 6b):

specific removal climbs from 8.8 mg g⁻¹ to 40.9 mg g⁻¹ over the same range with no sign of saturation, and the initial rate rises roughly in proportion to the load, from 0.123 mg L⁻¹ min⁻¹ to 0.419 mg L⁻¹ min⁻¹ ($r_0/C_0 = 0.012 - -0.017$ min⁻¹). Neither an equilibrium capacity nor a Langmuir–Hinshelwood saturation can be extracted from this window, and we do not fit one: with the lamp on from the start, what Ce3%–Nb₂O₅ removes is adsorption, any photocatalytic contribution and direct photolysis summed at a fixed time, and Fig. 6c shows that this sum is not even a stable baseline.

One condition was run twice, once in each experiment, and the two do not agree. At 20 mg L⁻¹ with Ce3%–Nb₂O₅, removal after 60 min was 45.7% when the stage was dark and 33.9% when the lamp was on from the start (Fig. 6c), on measured initial concentrations differing by 0.3 mg L⁻¹. The gap of 11.7 pp opens inside the first 5 min, where the two runs stand at 21.1% and 7.9%. Illumination did not add to removal over this window; it appeared to subtract from it. Three readings survive the data: photolysis intermediates absorbing at the analytical wavelength, so that part of the parent loss is masked (Section 3.2); desorption as the lamp warms a closed reactor; and ordinary between-campaign variability, the two experiments having been run separately. Nothing here separates them, and the gap is large enough that results from the two experiments should not be pooled.

3.5 What These Data Cannot Establish

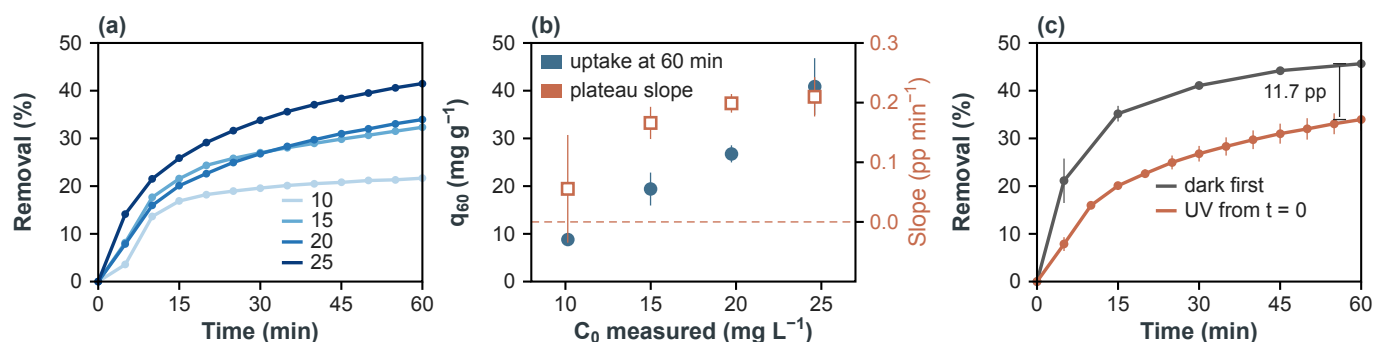
The campaign carries no undoped control, no photolysis blank and no dark control extended to 180 min. Without them the series cannot be used to claim that Ce loading improves on *ortho*-Nb₂O₅. Undoped material from the same route takes up 32 mg g⁻¹ tetracycline and has a Brunauer–Emmett–Teller (BET) area of 242 m² g⁻¹ [13], but it was tested under a separate protocol and serves as a benchmark, not a control. The UV-stage numbers include whatever direct 254 nm photolysis contributes; at that wavelength tetracyclines absorb and photolyse without any catalyst [38, 39], so in absolute terms the contribution is not negligible. Section 3.2 shows only that it cannot account for the differences between compositions. All comparisons above are therefore internal to the Ce series, where the missing controls would act alike on the three compositions and, for photolysis, cannot produce the differences between them.

The Ce loadings are nominal. Neither the Ce content of the products, nor its distribution, nor its oxidation state was measured, so the series is defined by what entered the autoclave rather than by what is in the solid. The growth of the low-angle phase at 5% Ce (Section 3.1) shows that Ce changes the product, and the absence of fluorite reflections excludes segregated CeO₂ above the diffraction detection limit; neither establishes substitution into the Nb₂O₅ lattice. For that reason we describe the materials as Ce-loaded rather than Ce-doped.

Detection is by absorbance at a single wavelength, 273 nm. That measures the disappearance of the parent chromophore, not degradation or mineralisation; under UV alone, oxytetracycline loses most of its parent signal while about 7% of its carbon is mineralised over the same hour [46]. The reading is also open to interference from transformation prod-

Table 2. Ce3%–Nb₂O₅ under continuous UV, no dark stage. Mean ± standard deviation of duplicates; r_0 fitted over the first 15 min, plateau slope over 45–60 min.

C_0 measured (mg L ⁻¹)	η_{60} (%)	q_{60} (mg g ⁻¹)	r_0 (mg L ⁻¹ min ⁻¹)	plateau slope (pp min ⁻¹)
10.1	21.7 ± 0.7	8.8 ± 0.4	0.123 ± 0.023	0.06 ± 0.09
15.0	32.3 ± 5.7	19.4 ± 3.4	0.222 ± 0.018	0.17 ± 0.03
19.7	33.9 ± 3.2	26.8 ± 1.8	0.270 ± 0.005	0.20 ± 0.02
24.6	41.5 ± 5.5	40.9 ± 5.9	0.419 ± 0.099	0.21 ± 0.03

**Figure 6.** Ce3%–Nb₂O₅ at 0.05 g in 0.200 L. (a) Removal traces under continuous UV; legend, C_0 in mg L⁻¹. (b) Uptake at 60 min (left axis) and removal slope over 45–60 min (right axis); dashed line, zero slope. (c) The same material and load at 20 mg L⁻¹ run two ways: dark stage first, or lamp on from $t = 0$. Bars: (b) standard deviation, (c) range of the two runs.

ucts that absorb near the parent maximum, which would make UV-stage removal appear smaller than it is, and from changes in absorbance on lamp-on, visible as the post-illumination dip of Fig. 4a. Scattering by the catalyst does not enter the reading, because the solution was filtered before the flow cell. For these reasons we report UV-stage removal of the parent signal and do not describe it as degradation. Total organic carbon or chromatographic detection would settle these points.

Solution conditions were controlled only loosely. pH and temperature were not recorded, the lamp shares the closed box with the reactor, and the solution carries no background electrolyte. The electrostatic state of the surface and of tetracycline during the runs is therefore unknown, and the uptake ranking cannot be linked to it.

The mechanism behind the uptake maximum at 3 % Ce remains open. Diffraction and infrared spectroscopy resolve a structural difference between Ce5%–Nb₂O₅ and the other two, but Ce1%–Nb₂O₅ and Ce3%–Nb₂O₅ are indistinguishable by both techniques although Ce3%–Nb₂O₅ takes up 50 % more in the dark. Surface area, pore volume, pore-size distribution, zeta potential, point of zero charge and Ce oxidation state were not measured, and one of those is the likely explanatory variable.

The two experiments are also not on a common baseline, as Fig. 6c shows at the single condition they share; only Section 3.2 and Section 3.3, which draw on one experiment, are internally consistent.

Finally, each condition was run in duplicate. The dark-uptake separation is large relative to that spread, but the p -values quoted throughout rest on two points per group and no correction for multiple comparisons; they indicate, they do not confirm.

4. CONCLUSIONS

Ce loading between 1 % and 5 % did not measurably change how much tetracycline a hydrothermal niobium oxide removes in 180 min; it changed which stage does the removing. Dark uptake peaks sharply at 3 % Ce, at (36.6 ± 0.8) mg g⁻¹ against (24.4 ± 0.7) mg g⁻¹ at 1 %, while the apparent UV-stage rate constant moves the opposite way and the two anticorrelate across individual runs. End-of-run removal spans only 3.7 pp across the series and separates nothing.

Two practical consequences follow. A loading or doping series of an adsorbent tested under illumination cannot be ranked by fixed-time removal, because the metric sums the two stages it should be distinguishing; recording q_{dark} and q_{UV} separately, at the cost of one dark stage, shows where the differences lie. And a rising removal percentage with rising initial concentration should be checked against the trace before it is interpreted: here it reflects one system at its plateau being compared with another still climbing. Establishing whether Ce loading helps at all,

whether the UV-stage removal is degradation, and why uptake peaks at 3 %, requires the undoped control, the photolysis blank, chromatographic or carbon analysis, Ce quantification and the surface characterisation identified in Section 3.5.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this work the authors used two generative AI tools. Claude (Anthropic) was used to write and run the scripts for the data re-analysis, kinetic fitting and figures, and to draft and edit the manuscript text. Gemini (Google) was used to generate the apparatus illustration in Fig. 1b, which was then edited; the tubing, flow arrows, labels and all text in that panel were drawn as vector graphics. Three decorative icons in the graphical abstract (a particle, a beaker and a UV lamp) were generated with an image-generation model (OpenAI, via Codex); all text, numbers and the bar chart in the graphical abstract were drawn by the authors from the data. No AI tool generated or altered the experimental data. After using these tools the authors reviewed and edited all output, checked every number against the raw data, and take full responsibility for the content of the article.

CRedit AUTHORSHIP CONTRIBUTION STATEMENT

Novia Safitri: Investigation, Data curation, Writing – original draft. **Kamilah Nada Maisa:** Formal analysis, Writing – review & editing. **Kholik Hidayatullah:** Formal analysis, Writing – review & editing. **Abdul Latief Salim Azpar:** Formal analysis, Writing – review & editing. **Alam Hannaan Muhammad:** Formal analysis, Writing – review & editing. **Sephia Amanda Muhtar:** Conceptualization, Methodology, Supervision, Project administration, Writing – review & editing.

DECLARATION OF COMPETING INTEREST

The authors declare no competing interests.

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DATA AVAILABILITY

Raw absorbance data, the analysis scripts and the intermediate tables are available from the corresponding author.

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