



Eco-friendly Fabrication of Nickel Oxide Nanoparticles and Their Application in Water Purification

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ABSTRACT

Mentha arvensis leaf extract was employed for the green synthesis of nickel oxide (NiO) nanoparticles and their applicability in water purification and photocatalysis was validated. Pure crystalline NiO nanoparticles were characterized through FTIR, XRD, SEM, TEM and thermal analysis. The NiO nanoparticles degraded methyl orange dye with almost 90% removal efficiency under visible-light irradiation, indicating excellent photocatalytic properties. The NiO nanoparticles also demonstrated potential antibacterial properties in wastewater samples collected from residential, agricultural and industrial sources. In addition, when the NiO-coated materials were exposed to sunlight, a significant decrease was observed in microbial contamination, electrical conductivity and total dissolved solids, with an average reduction of greater than 85% in bacterial count. Therefore, this study demonstrates that green-synthesized NiO nanoparticles exhibit photocatalytic and antibacterial properties and have potential as sustainable materials for wastewater-treatment applications.

Keywords: Green synthesis; NiO nanoparticles; *Mentha arvensis*; Photocatalysis; Water purification.

1. INTRODUCTION

Water is an essential resource for sustainable ecosystems and human health. However, untreated wastewater and the accumulation of biological and chemical contaminants continue to threaten potable and agricultural water resources (D'Odorico *et al.* 2020; Mehdi *et al.* 2021). Conventional wastewater-treatment methods rely on physical, chemical and biological operations, but many of these approaches require high chemical input, high energy demand or costly post-treatment handling (Crini and Lichtfouse, 2019; Qu *et al.* 2013).

Nanotechnology offers an attractive route for wastewater remediation because nanoparticles can provide high surface area, tunable morphology and strong adsorption or photocatalytic activity at relatively low dosage (Ealia and Saravanakumar, 2017; Qu *et al.* 2013). Nanoparticles are commonly defined as materials in the 1-100 nm range and can occur as spheres, rods, tubes, plates, dots, wires and other morphologies. Their high reactivity and large surface-area-to-volume ratio are useful for contaminant capture and degradation, although environmental fate and food-chain transfer remain important concerns (Maharramov *et al.* 2019; Mishra *et al.* 2017).

Metal oxide nanoparticles such as TiO₂, ZnO and NiO have been widely investigated as adsorbents, photocatalysts and antimicrobial materials for polluted

water (Mustapha *et al.* 2020; Siddique *et al.* 2021; Haruna *et al.* 2022). Green synthesis routes based on plant, algal or microbial extracts are especially attractive because biomolecules in the extract can act as reducing, stabilizing and capping agents while reducing reliance on harsh chemical reagents (Haidri *et al.* 2023; Mansour *et al.* 2022). Recent NiO and Ni/NiO studies also show promising antibacterial and environmental-remediation performance (Ali *et al.* 2022; Agalya *et al.* 2024).

In the present study, NiO nanoparticles were prepared through an eco-friendly route using *Mentha arvensis* leaf extract. The synthesized nanoparticles were characterized by thermal analysis, FTIR, XRD, SEM and TEM, and their applicability was evaluated through methyl orange photocatalytic degradation and water-purification tests.

2. MATERIALS AND METHODS

2.1 Experimental

The nickel(II) chloride hexahydrate used in this study (99.99% purity) was obtained from Merck/Sigma-Aldrich (Burlington, MA, USA). Distilled water and sodium hydroxide (98% by weight) were obtained from Fluka (Merck, Burlington, MA, USA). All chemicals were of analytical grade and were used without further purification. The following laboratory instruments were used: a Memmert GmbH (Büchenbach, Germany) electric oven model UF55; a Nabertherm GmbH

(Lilienthal, Germany) furnace model L 9/11/180; and a Daihan LabTech (Batam, Indonesia) hot-plate magnetic stirrer model LMS-1003.

2.2 Preparation of NiO NPs using *Mentha Arvensis* Extract

Mentha arvensis leaves were collected from gardens and parks located in Dehradun District, Uttarakhand, India. The leaves were dried for 24 h at 50 °C to remove residual moisture, ground into a fine powder and stored in sterilized glass jars. Approximately 2 g of the herbal powder was combined with 40 mL of distilled water in a vessel placed on a hot plate and maintained at 45 °C for 30 min. The resulting aqueous extract was filtered using Whatman No. 1 filter paper. A visible colour shift occurred when 1.6 g nickel(II) chloride hexahydrate salt was added gradually to 50 mL of herbal solution at 75 °C. The mixture was then stirred continuously for 24 h at 70 °C. After completion of the reaction, the colour changed from greenish to yellowish-brown, indicating the formation of NiO nanoparticles. The samples were centrifuged for 15 min at 5000 rpm and room temperature. The resulting powder was air dried and annealed for 2 h at 400 °C to produce pure NiO nanoparticles, which were stored at 4 °C until use.

2.3 Characterization

The synthesized NiO nanoparticles were characterized through thermal analysis, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), photocatalytic tests and antimicrobial activity tests against Gram-positive and Gram-negative bacteria.

FTIR measurements were performed using a Thermo Fisher Scientific Nicolet iS50 spectrometer with an attenuated total reflection accessory. Spectra were acquired from 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹ using 30 scans at room temperature. Each spectrum required approximately 45 s, and the data were processed using the Omnic software package.

XRD analysis was performed using a Malvern Panalytical Empyrean diffractometer with Ni-filtered Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). The diffractometer was equipped with a PIXcel3D detector, anti-scatter slits on both the incident and diffracted beam channels, and a fixed divergence slit. HighScore Plus software and the International Centre for Diffraction Data (ICDD) PDF-4+ database were used to process and analyze the patterns. Crystal structures were refined by the Rietveld method with pseudo-Voigt peak shapes and polynomial background functions (Rietveld, 1969).

The surface morphology and microstructure of NiO nanoparticles were examined using an FEI Quanta

Inspect F50 scanning electron microscope equipped with an energy-dispersive X-ray spectroscopy detector. Transmission electron microscopy was performed using an FEI Tecnai G2 F30 S-TWIN high-resolution transmission electron microscope with a Gatan selected-area electron diffraction module. TEM samples were prepared by drying and ultrafine ball milling, and bright-field TEM examinations were performed at an accelerating voltage of 300 kV.

Thermal studies were performed using a NETZSCH STA 449 F3 Jupiter thermal analyzer. To study photocatalytic activity under illumination, methyl orange (MO) was used as the target dye. A 20 mg quantity of MO was dissolved in 10 mL ultra-pure water, and 0.025 g NiO powder was added to the dye solution. The suspension was magnetically stirred in the dark for 30 min to establish adsorption-desorption equilibrium before visible-light irradiation using a 150 W fluorescent lamp placed approximately 20 cm from the reaction vessel. Aliquots were collected during illumination and analyzed by UV-Visible spectroscopy using a JASCO V-560 spectrophotometer.

3. RESULTS AND DISCUSSION

3.1 Analytical Results

Thermogravimetric and differential scanning calorimetric (TG/DSC) studies were used to assess the thermal stability, phase purity and calcination temperature of the fabricated NiO nanoparticles. Fig. 1 shows that the precursor retained hydroxyl groups, physically adsorbed water and nickel-precursor residues. The first two weight-loss stages were linked to the removal of hydroxyl species and surface water, with clear endothermic effects at both degradation stages. Similar thermal events are commonly used to identify decomposition windows for NiO formation from nickel precursors (De Jesus *et al.* 2005; Hong *et al.* 2021).

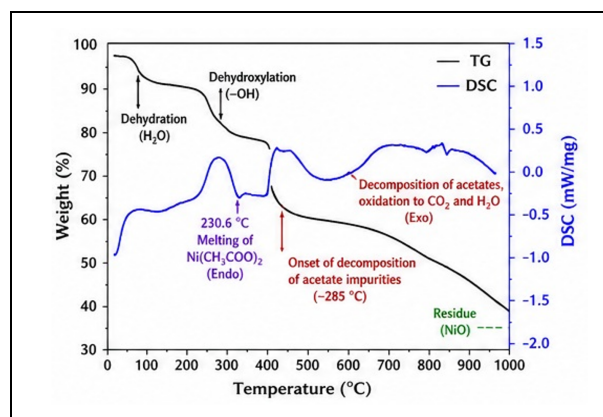


Fig. 1: TG/DSC thermogram of NiO precursor

The release of water vapour below 140 °C was also evidenced by the FTIR 3D evolved-gas

chromatographs (Fig. 2). An endothermic effect was observed at about 225 °C without significant mass loss, which was attributed to melting or rearrangement of residual precursor species. As the temperature increased to approximately 285 °C, the decomposition of precursor impurities yielded acetate-related fragments that underwent exothermic oxidation, resulting in the evolution of CO₂ and H₂O.

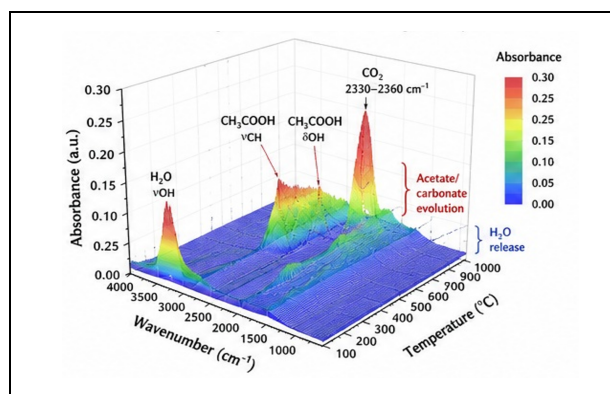


Fig. 2: 3D FTIR evolved-gas analysis

The FTIR spectra for the evolving gases exhibit normal CO₂ vibrational bands in the range 2330-2360 cm⁻¹ and absorption bands for acetic acid species at approximately 3540 cm⁻¹ (vOH), 2965 cm⁻¹ (vCH) and 1738 cm⁻¹ (δOH). At 280 °C in a multistep decomposition profile, hydroxy acetate intermediates such as Ni₂(OH)₂(CH₃COO)₂ were identified. The 2D thermal imagery showed that acetate decomposition and the formation and subsequent decomposition of carbonate species occurred within 390-610 °C. To ensure complete removal of contaminants and conversion to crystalline NiO nanoparticles, the sample was calcined at 950 °C. The FTIR spectrum shows Ni-O vibrations at 468 and 425 cm⁻¹ (Fig. 3), with no absorption bands for precursor residues, supporting the formation of phase-pure NiO nanoparticles (Hong *et al.* 2021; Haq *et al.* 2022).

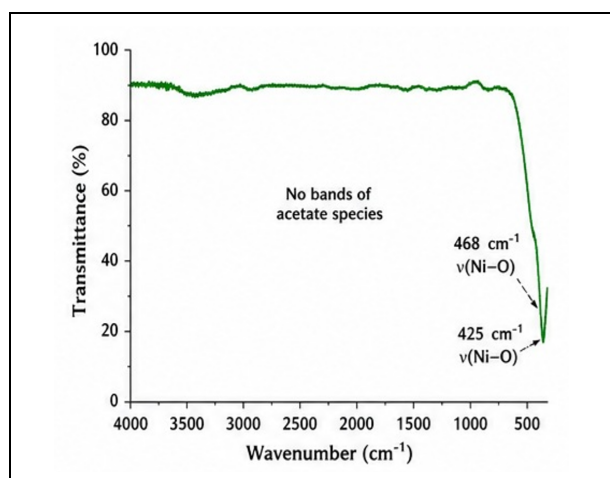


Fig. 3: FTIR spectrum of calcined NiO

A clear X-ray diffraction pattern, representative of a single-phase crystalline NiO structure, supports the presence of crystalline NiO, as shown in Fig. 4. The diffraction data were refined using the Rietveld method. Agreement parameters calculated from the fitted models ($R_{\text{expected}} = 9.0241$; $R_{\text{profile}} = 8.5627$; weighted $R_{\text{profile}} = 10.2148$; and $\chi^2 = 1.2874$) indicate satisfactory agreement between the experimental pattern and the cubic bunsenite phase of NiO (ICDD PDF #04-083-0640; International Centre for Diffraction Data, 2024). The refined lattice parameter ($a = b = c = 4.1786 \pm 0.0003$ Å) was determined with a unit-cell volume of approximately 72.94 Å³ and angles equal to $\alpha = \beta = \gamma = 90^\circ$.

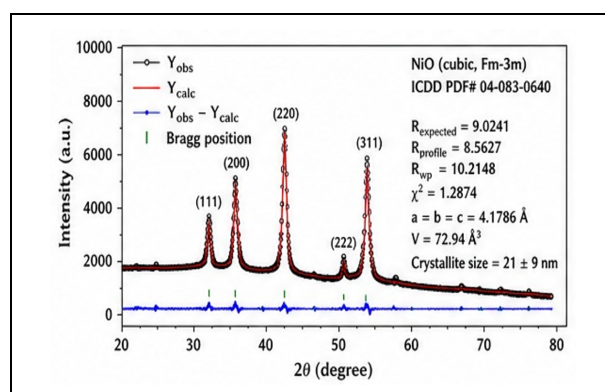


Fig. 4: XRD pattern and rietveld refinement of NiO nanoparticles

The March-Dollase value (0.836) indicates preferred orientation along the (200) crystallographic plane. Crystallite-size data produced through whole-pattern fitting suggest an average crystallite size of approximately 21 ± 9 nm. Low-magnification micrographs show that the NiO nanoparticles occur as irregular micrometer-sized agglomerates, as demonstrated by the SEM images (Fig. 5). In general, the NiO powder consists mainly of quasi-spherical to polyhedral nanoparticles with a relatively consistent size distribution, which is consistent with reported NiO nanoparticle morphologies (Hong *et al.* 2021).

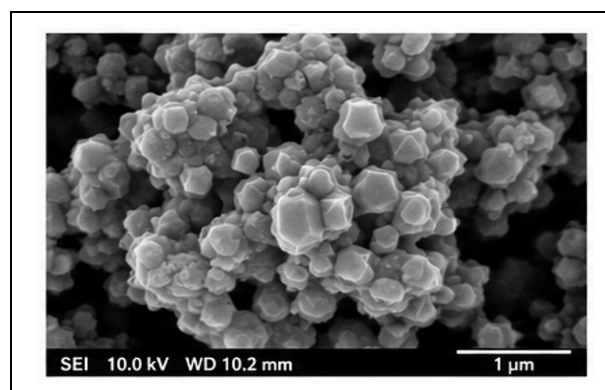


Fig. 5: SEM image of NiO nanoparticles

TEM investigations (Fig. 6) further indicated that the NiO powder comprises primarily polyhedral nanoparticles with dimensions ranging from 18 to 32 nm. Most particles appear to consist predominantly of single crystals, as inferred from the correlation between XRD and TEM results. The NiO nanoparticles also show a tendency to form uneven and discontinuous agglomerates. NiO nanoparticles have received significant attention because of their photocatalytic, biological and optical properties.

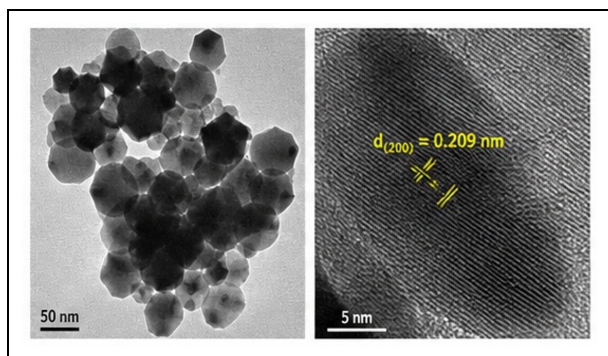


Fig. 6: TEM images of NiO nanoparticles

The photocatalytic activity of NiO nanoparticles was evidenced by the gradual reduction in the characteristic absorption peak of MO at around 465 nm (Fig. 7) with increasing irradiation time. After 160 min of light exposure, the degradation efficiency was approximately 89%, calculated from the decrease in absorbance relative to the initial dye concentration. This performance is consistent with recent reports showing strong visible-light methyl orange degradation by green-synthesized NiO-based photocatalysts (Kapil *et al.* 2026; Haq *et al.* 2022).

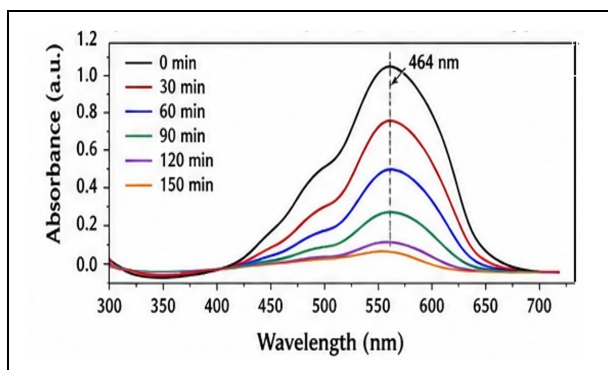


Fig. 7: UV-Visible spectra of MO during photocatalytic degradation by NiO nanoparticles

The photocatalytic degradation mechanism follows pseudo-first-order reaction kinetics for diluted dye solutions (Fig. 8), as defined by the following equation:

$$\ln(C_0/C) = k_{app} t \quad \dots (1)$$

where C_0 represents the initial concentration of methyl orange, C is the concentration at irradiation time t and k_{app} denotes the apparent first-order rate constant. The apparent rate constant (k_{app}) was 0.0151 min^{-1} , the removal efficiency at 150 min was 89%, and the regression coefficient was $R_2 = 0.9926$. These values support efficient photocatalytic degradation of MO by the synthesized NiO nanoparticles.

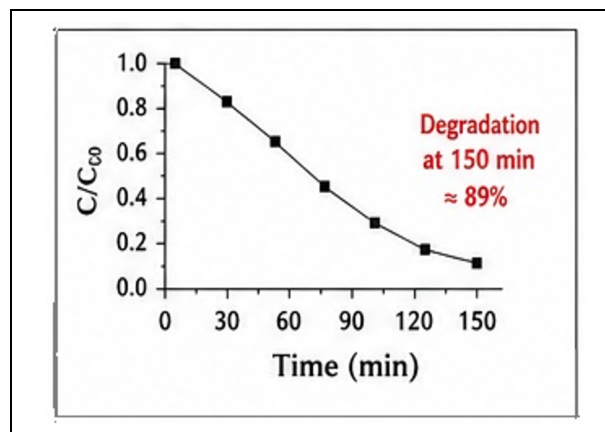


Fig. 8: Pseudo-first-order reaction kinetics for diluted dye solutions by NiO nanoparticles

3.2 Water Analysis

The NiO nanoparticles were studied for their potential application as microbial disinfectants and water-purification materials. Three water samples were collected from domestic wastewater, agricultural field water and industrial effluent. These samples were analyzed for antimicrobial and water-treatment efficacy and were labelled Sample 1, Sample 2 and Sample 3, respectively. Each sample was stored separately in sterile containers and transported to the laboratory for analytical testing.

To quantify microbial load, physicochemical and microbiological characteristics were analyzed, including pH, electrical conductivity (EC), total dissolved solids (TDS) and colony-forming units (CFU). For each site, a second set of water samples was collected and labelled Sample A, Sample B and Sample C. These samples were subjected to direct sunlight for approximately 6 h/day for 4 consecutive days. A third set from the same water sources was labelled Sample A', Sample B' and Sample C'. Samples B' and C' were stored in NiO nanoparticle-coated containers and exposed to sunlight under the same schedule. As shown in Tables 1 and 2, treatment with NiO nanoparticles plus sunlight produced superior performance compared with untreated samples or samples exposed to sunlight alone.

Table 1. Physicochemical analysis of water samples before and after NiO nanoparticle treatment

Sample	Water Source	pH	EC ($\mu\text{S}/\text{cm}$)	TDS (mg/L)	Treatment Condition
Sample 1	Domestic wastewater	7.82	912	598	Untreated
Sample 2	Agricultural field water	8.15	1045	672	Untreated
Sample 3	Industrial effluent	6.94	1288	825	Untreated
Sample A	Domestic wastewater	7.61	801	520	Sunlight exposed
Sample B	Agricultural field water	7.92	938	603	Sunlight exposed
Sample C	Industrial effluent	6.75	1162	742	Sunlight exposed
Sample A'	Domestic wastewater	7.28	612	401	NiO-coated + sunlight
Sample B'	Agricultural field water	7.54	704	458	NiO-coated + sunlight
Sample C'	Industrial effluent	6.98	875	563	NiO-coated + sunlight

Table 2. Microbial analysis of water samples treated with NiO nanoparticles

Sample	Total Bacterial Count (CFU/mL)	Viral/Protozoal Presence	Reduction Efficiency (%)	Treatment Condition
Sample 1	3.8×10^5	High	-	Untreated
Sample 2	4.5×10^5	Moderate	-	Untreated
Sample 3	6.2×10^5	High	-	Untreated
Sample A	2.4×10^5	Moderate	36.8	Sunlight exposed
Sample B	2.9×10^5	Moderate	35.5	Sunlight exposed
Sample C	4.1×10^5	Moderate	33.9	Sunlight exposed
Sample A'	5.2×10^4	Low	86.3	NiO-coated + sunlight
Sample B'	6.7×10^4	Low	85.1	NiO-coated + sunlight
Sample C'	9.5×10^4	Very low	84.7	NiO-coated + sunlight

Table 3. Comparative performance of untreated and NiO-treated water samples

Parameter	Untreated Samples	Sunlight-treated Samples	NiO-coated Samples
Average pH	7.64	7.43	7.26
Average EC ($\mu\text{S}/\text{cm}$)	1081	967	730
Average TDS (mg/L)	698	622	474
Average CFU Reduction (%)	-	35.4	85.4
Water Quality Improvement	Low	Moderate	Excellent

The results indicate that NiO nanoparticles can be used for microbial remediation and as a component of water-treatment processes. The comparison of untreated,

sunlight-exposed and NiO nanoparticle-coated water samples indicates that the coated containers improved the physicochemical and microbial properties of the water, as

shown in Table 3. Untreated water samples showed the highest microbial contamination, TDS and EC.

These relatively high contaminant levels indicate that waters from these sources are unsafe for domestic or environmental use because of pathogenic microbes, suspended pollutants and dissolved inorganic salts. Among the untreated samples, industrial wastewater contained the highest EC, TDS and CFU levels, probably because of organic waste, industrial pollutants and high nutrient concentrations that support microbial growth. A slight decrease in EC, TDS and microorganism count was observed in water samples exposed only to sunlight, likely due to solar disinfection and photochemical activity.

For the group treated with NiO nanoparticles, all testing parameters showed marked improvement. Electrical conductivity and total dissolved solids decreased, suggesting that NiO nanoparticles facilitated adsorption and removal of suspended pollutants and dissolved ionic species. The high surface area and surface reactivity of NiO nanoparticles likely improved interactions between pollutants and nanoparticle surfaces.

Reduced microorganism concentrations in NiO-treated samples demonstrated the most significant treatment impact, with bacterial CFU reductions greater than about 85%. The antibacterial effect may be associated with superoxide and hydroxyl radical formation under sunlight, membrane damage, release of nickel ions and disruption of enzymatic or metabolic activity. Similar ROS-associated antimicrobial mechanisms and bacterial-control performance have been reported for green-synthesized NiO and Ni/NiO nanoparticles (Etefa *et al.* 2023; Ali *et al.* 2022; Agalya *et al.* 2024).

The combined effect of solar irradiation and NiO nanoparticles appears to enhance water purification. Under light exposure, NiO nanoparticles can generate reactive oxygen species and accelerate the degradation of organic pollutants and microbes. The partial mineralization of organic contaminants and formation of intermediate oxidative species during photocatalysis may also explain the moderate pH decrease after treatment. Overall, the results suggest that NiO nanoparticles can provide a low-cost, eco-friendly approach for improving contaminated water quality, especially in settings without advanced filtration systems.

4. CONCLUSION

A simple and eco-friendly method was used to demonstrate how *Mentha arvensis* extract can be used to synthesize NiO nanoparticles. Characterization showed that the produced nanoparticles were crystalline, structurally stable and suitable for photocatalytic

application. The NiO nanoparticles degraded methyl orange efficiently and reduced microbial load in contaminated water when combined with sunlight exposure. These findings suggest that green-produced NiO nanoparticles could be an economically viable, sustainable and effective option for environmental remediation and wastewater treatment.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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