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Conference paper

Sol-Gel and Hydrothermal Synthesis of NiO Nanoparticles: A Comparative Study for Efficient Photocatalytic Wastewater Treatment

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Abstract

Nickel oxide nanoparticles (NiO-NPs) have recently attracted considerable attention as low-cost, chemically stable photocatalysts for the remediation of dye-contaminated wastewater. In this work, NiO-NPs were synthesized from nickel acetate by two widely used routes, sol-gel (SG) and hydrothermal (HT) synthesis, followed by calcination at 450 °C for 2 h, in order to evaluate how the synthesis pathway governs their structural, morphological and photocatalytic properties. X-ray diffraction confirmed the formation of pure, single-phase cubic NiO (space group Fm-3m) for both routes, but revealed markedly different crystallite sizes, with HT-NiO (≈ 41.7 nm) more crystalline than SG-NiO (≈ 30.2 nm). The photocatalytic performance of the two powders was assessed through the degradation of methylene blue (10 mg/L) under UV irradiation. HT-NiO degraded about 75% of the dye within one hour, outperforming SG-NiO, which degraded about 65% of the dye under the same conditions. This enhanced activity is attributed to the larger crystallite size and superior crystallinity of the hydrothermally synthesized particles, which favour a more efficient separation and migration of photogenerated charge carriers and limit electron-hole recombination, despite a comparatively lower specific surface area. These results indicate that crystalline quality, rather than particle size or surface area alone, is a decisive factor governing the photocatalytic efficiency of NiO nanoparticles. Overall, the hydrothermal route appears as a promising, scalable and environmentally friendly strategy for producing NiO-based photocatalysts suitable for the treatment of dye-polluted industrial effluents, in line with current efforts toward sustainable, carbon-neutral wastewater treatment.

Keywords: NiO nanoparticles; Photocatalysis; Hydrothermal synthesis; Sol-gel synthesis; Methylene blue degradation.

1. Introduction

The growing contamination of water resources by synthetic dyes discharged from the textile, paper and leather industries has made the development of efficient, low-cost remediation technologies a pressing environmental priority. Among the various strategies proposed, heterogeneous photocatalysis using semiconductor metal oxides has emerged as one of the most attractive approaches, owing to its ability to mineralise a wide range of organic pollutants without generating secondary waste [1,2]. Within this family of materials, nickel oxide nanoparticles (NiO-NPs) have received growing interest because they combine a large specific surface area, a tunable band gap, low production cost and remarkable chemical and thermal stability.

Beyond wastewater treatment, NiO nanoparticles are exploited across a wide range of technological fields as a direct consequence of these properties. Their high reactivity and large surface area make them attractive catalysts for numerous chemical transformations, while their good electrochemical behaviour has led to their incorporation into lithium-ion battery electrodes to improve performance and cycling stability. Their sensitivity to environmental changes is also exploited in gas, temperature and humidity sensors, and their electrochromic response is used to regulate light transmission in smart windows. In the biomedical field, the antimicrobial and antioxidant character of NiO-NPs has opened avenues for early disease detection and targeted therapy [1].

In the specific context of photocatalytic water treatment, several complementary mechanisms explain the efficiency of NiO-based materials, as summarised in Table 1. Their high specific surface area promotes the breakdown of hydrocarbons, aromatic compounds and phenolic derivatives, while under irradiation they generate reactive oxygen species (ROS) capable of mineralising organic pollutants into harmless by-products. Physical capture further contributes to the removal of hydrocarbons, phenols, fats and oils, and the strong affinity of NiO surfaces for heavy-metal cations allows the simultaneous elimination of toxic species such as lead, cadmium and mercury. When NiO nanoparticles are incorporated into filtration membranes, the capture efficiency toward particles, colloids and macromolecules is further improved.

Table 1 Main properties of NiO nanoparticles and their effect on pollutant removal.

Property of NiO-NPs	Effect on pollutant removal
High specific surface area	Enhanced breakdown of hydrocarbons, aromatic compounds and phenols
Reactive oxygen species (ROS) generation	Breakdown of organic pollutants into harmless by-products
Physical capture	Effective removal of hydrocarbons, phenols, fats and oils
Strong affinity for heavy metals	Capture and elimination of toxic metals (Pb, Cd, Hg)
Incorporation into membranes	Improved capture efficiency of particles, colloids and macromolecules

The controlled synthesis of NiO nanoparticles is a decisive step for optimising their catalytic performance, since particle size, crystallinity and surface chemistry are all strongly dependent on the preparation route. Among the many synthesis strategies available, the sol-gel technique remains one of the most widely used because of its simplicity, low cost and good control of stoichiometry [3]. Hydrothermal synthesis, based on the crystallisation of precursors in an aqueous medium under pressure, has proven particularly suitable for producing nanocrystalline oxides with well-defined morphology, as the crystallite size and shape can be tuned through the reaction temperature, pressure, pH and duration [4].

Although both routes are frequently reported in the literature, direct comparisons of sol-gel- and hydrothermally-synthesized NiO nanoparticles prepared from the same precursor and evaluated under identical photocatalytic conditions remain relatively scarce. The present study addresses this gap by synthesizing NiO-NPs from nickel acetate via sol-gel and hydrothermal routes under comparable conditions, and by systematically comparing their structural and morphological properties with their photocatalytic activity toward methylene blue degradation under UV irradiation. The aim is to clarify how the synthesis method governs the structure-activity relationship of NiO nanoparticles and to identify the most promising route for scalable, environmentally friendly wastewater treatment.

2. Materials and methods

2.1. Materials

Nickel(II) acetate tetrahydrate was used as the nickel precursor for both synthesis routes. Analytical-grade reagents, absolute ethanol, sodium hydroxide and distilled water were used throughout without further purification. Methylene blue (MB) was used as the model organic pollutant for the photocatalytic degradation tests.

2.2. Synthesis of NiO nanoparticles by the sol-gel method

For the sol-gel (SG) route, a stoichiometric amount of nickel acetate was dissolved in absolute ethanol under continuous magnetic stirring. A few drops of concentrated nitric acid were added to catalyse the hydrolysis and condensation reactions. The resulting sol was aged at room temperature for 24 h until gelation occurred, and the resulting gel was dried at 80 °C to remove residual solvent. The dried xerogel was then ground into a fine powder and calcined in air at 450 °C for 2 h to decompose organic residues and crystallise the NiO phase (sample denoted SG-NiO).

2.3. Synthesis of NiO nanoparticles by the hydrothermal method

For the hydrothermal (HT) route, an aqueous solution of nickel acetate was prepared and the pH was adjusted to the alkaline range with sodium hydroxide to induce the precipitation of a nickel hydroxide precursor. The resulting suspension was transferred to a Teflon-lined stainless-steel autoclave and heated under autogenous pressure to promote crystallisation of the precursor. After natural cooling to room temperature, the precipitate was recovered by centrifugation, washed repeatedly with distilled water and ethanol, and dried. The dried precursor was finally calcined in air at 450 °C for 2 h under the same conditions as the SG sample, to allow a direct comparison between the two routes (sample denoted HT-NiO).

2.4. Characterization

The crystal structure and phase purity of the calcined powders were investigated by X-ray diffraction (XRD) on a diffractometer using Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). Phase identification was performed by comparison with standard ICDD data. The average crystallite size was estimated from the full width at half maximum (FWHM) of the principal diffraction peak using the Debye-Scherrer equation, and the lattice parameter was refined

from the corresponding interplanar spacing. The morphology of the NiO nanoparticles was examined by electron microscopy to evaluate particle shape, agglomeration state and surface texture for both synthesis routes.

2.5. Photocatalytic activity evaluation

The photocatalytic activity of SG-NiO and HT-NiO was evaluated through the degradation of methylene blue (10 mg/L) under UV irradiation. In a typical experiment, the photocatalyst was dispersed in the dye solution and stirred in the dark for 30 min to reach adsorption-desorption equilibrium prior to irradiation. The suspension was then exposed to UV light, and aliquots were withdrawn at regular time intervals over one hour, centrifuged to remove the catalyst, and analysed by UV-Vis spectrophotometry at the characteristic absorption maximum of methylene blue (≈ 664 nm). The degradation efficiency was calculated from the relative decrease in dye absorbance with irradiation time. Control experiments performed in the absence of catalyst or without light confirmed that the observed colour removal was due to photocatalytic action rather than simple adsorption or photolysis.

3. Results and discussion

3.1. Structural analysis by XRD

The XRD patterns recorded for the calcined NiO nanoparticles obtained by the sol-gel and hydrothermal routes are shown in Figure 1. Both samples display the characteristic diffraction pattern of pure, single-phase cubic NiO (space group Fm-3m), with no detectable secondary phase or unreacted precursor, confirming that calcination at 450 °C for 2 h is sufficient to achieve complete crystallisation for both synthesis routes.

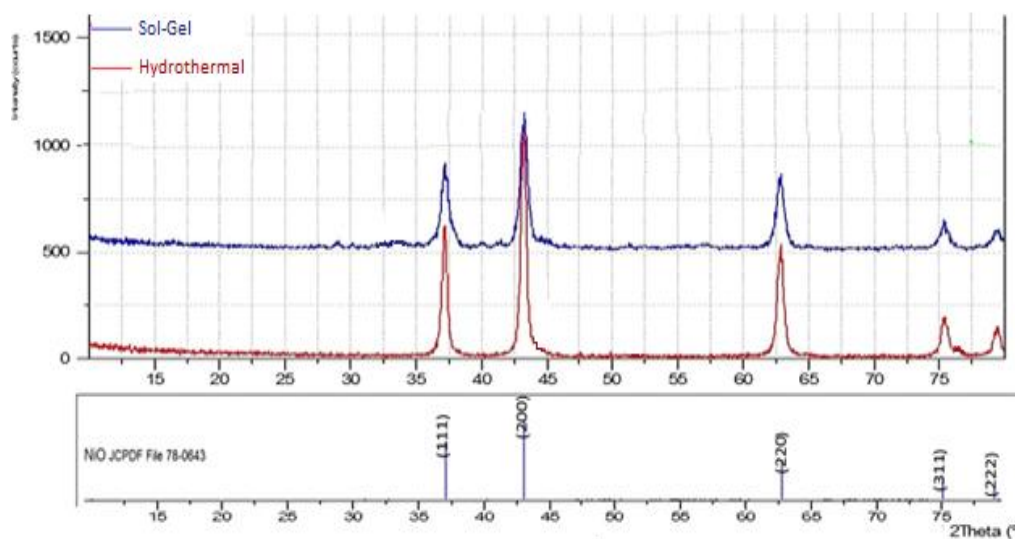


Figure 1 XRD patterns of NiO nanoparticles synthesized by the sol-gel (SG-NiO, blue) and hydrothermal (HT-NiO, red) routes, indexed to the cubic NiO phase (JCPDS file 78-0643)

As summarised in Table 2, the position of the main (200) reflection is nearly identical for both samples (43.13–43.17°), confirming that the cubic NiO lattice is preserved regardless of the synthesis route, with lattice parameters of 4.192 Å and 4.188 Å for HT-NiO and SG-NiO, respectively, in good agreement with the reference value for bulk NiO. The key difference between the two samples lies in the peak broadening: the narrower FWHM measured for HT-NiO (0.216°) compared with SG-NiO (0.299°) translates, through the Scherrer equation, into a markedly larger average crystallite size for the hydrothermally synthesized sample (≈ 41.7 nm) than for the sol-gel one (≈ 30.2 nm). This indicates that the hydrothermal route promotes a slower, more controlled crystal growth, yielding larger and better-crystallised NiO domains, whereas the sol-gel process, involving rapid gelation and drying, tends to produce smaller and comparatively more disordered crystallites.

Table 2 Structural parameters of NiO nanoparticles obtained by Rietveld/Scherrer analysis of the XRD patterns.

Sample	Phase (hkl)	2θ (°)	d _{hkl} (Å)	Lattice param. a (Å)	FWHM (°)	Crystallite size D (nm)
HT-NiO	NiO (200)	43.13	2.096	4.192	0.216	41.74
SG-NiO	NiO (200)	43.17	2.094	4.188	0.299	30.2

3.2. Morphological analysis

Electron microscopy images of the two powders, presented in Figure 2, corroborate the structural trends deduced from XRD. The sol-gel sample consists of fine, loosely agglomerated nanoparticles with a relatively narrow size distribution, consistent with its smaller crystallite size. The hydrothermally synthesized sample instead shows more clearly faceted, better-defined particles with a comparatively coarser texture, in agreement with the larger crystallite size and higher crystallinity inferred from the sharper XRD reflections. These morphological differences are consistent with the distinct nucleation and growth mechanisms operating in each synthesis route: the fast, kinetically controlled gelation of the sol-gel process versus the slower, thermodynamically driven crystallisation that takes place inside the autoclave during hydrothermal treatment.

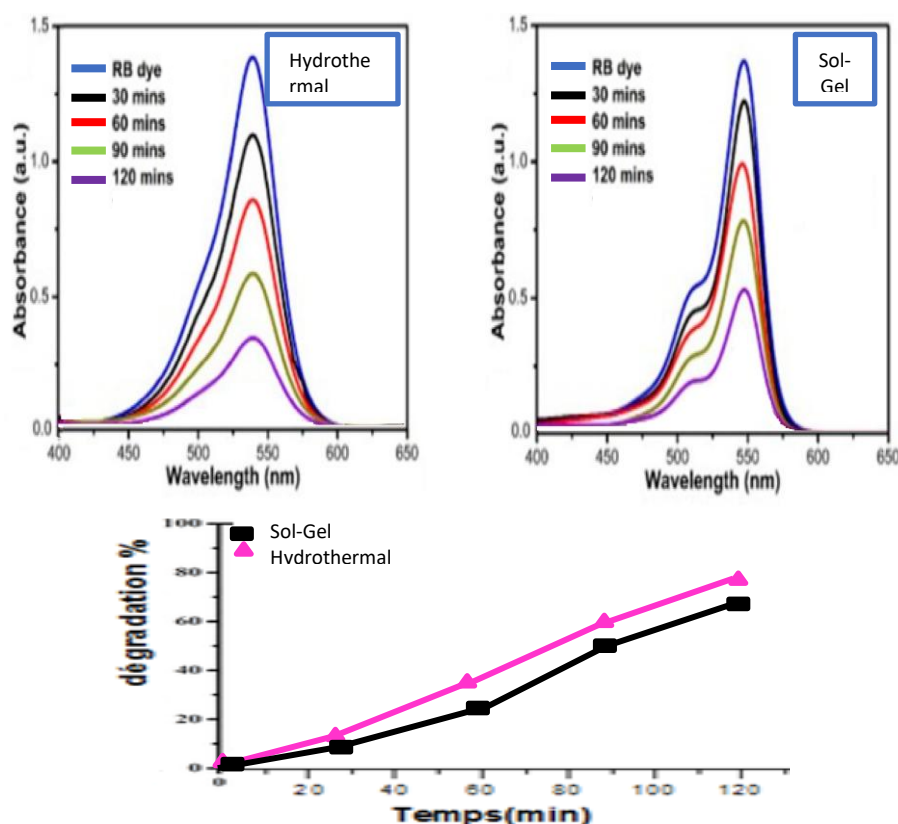


Figure 2 Electron micrographs of NiO nanoparticles synthesized by the sol-gel (top) and hydrothermal (bottom) routes.

3.3. Photocatalytic degradation of methylene blue

The photocatalytic performance of SG-NiO and HT-NiO was evaluated by monitoring the degradation of methylene blue (10 mg/L) under UV irradiation over one hour. Under these conditions, HT-NiO degraded approximately 75% of the dye, compared with about 65% for SG-NiO. This difference, although moderate in absolute terms, is reproducible and consistent with the structural characterisation described above, confirming that the hydrothermal route yields a more efficient photocatalyst despite requiring the same calcination treatment as the sol-gel sample. Control experiments performed in the absence of catalyst or without light confirmed that the observed colour removal was due to photocatalytic action rather than simple adsorption or photolysis.

This result may at first appear counter-intuitive, since a larger crystallite size is generally associated with a smaller specific surface area and, therefore, with fewer surface-active sites available for pollutant adsorption. However, several complementary factors can explain the superior activity of HT-NiO. First, the higher crystallinity of the hydrothermal sample reduces the density of structural defects that otherwise act as recombination centres for photogenerated electron-hole pairs, thereby extending charge-carrier lifetime. Second, hydrothermally grown NiO surfaces are typically richer in surface hydroxyl groups, which favour the trapping of photogenerated holes and the subsequent generation of hydroxyl radicals. Third, the improved separation and migration of charge carriers in a more ordered crystal lattice outweighs the moderate loss in surface area, so that photocatalytic efficiency is ultimately governed more by crystalline quality than by particle size alone.

These findings are consistent with the broader literature on NiO-based photocatalysts, summarised in Table 3. Using a green combustion route, Kumar et al. [5] reported an outstanding 99.6% degradation of methylene blue under UV light, while Ali et al. [6], working with green Ni/NiO nanoparticles under visible light, achieved 95% removal of crystal violet. Mustafa et al. [7] showed that biosynthesized NiO-NPs can simultaneously decolorize several azo dyes under sunlight, with efficiencies of 93.6% for Congo red, 82.4% for methylene blue and 91.1% for malachite green, while Jagan et al. [8] obtained 92.8% and 98.0% degradation of safranin and methyl orange, respectively, by tuning the alkaline activator used during

synthesis. Somewhat lower, but still substantial, efficiencies were reported by Sundaresan et al. [9] for rhodamine B (85%) and by Ahmad et al. [10] for acridine orange (84%) using plant-mediated NiO-NPs. Fatimah et al. [11] demonstrated that green-synthesized NiO-NPs can also target emerging pollutants, achieving 99.4% oxidation of the antibiotic tetracycline, and Sethuraman et al. [12] reported 91.7% degradation of the azo dye allura red using NiO-NPs obtained via a *Trachyspermum ammi*-mediated green route. Taken together, these studies show that degradation efficiencies ranging from about 82% to over 99% have been reported for NiO-based photocatalysts, depending on the precursor, irradiation source and dye considered. The performance obtained here for HT-NiO, while somewhat lower than the highest values reported under optimised conditions, was achieved without any surfactant, template or green-synthesis additive, underlining the intrinsic potential of a simple, scalable hydrothermal route.

Table 3 Comparison of the photocatalytic degradation efficiency of NiO nanoparticles reported in the literature and obtained in this work.

Precursor	Irradiation	Target pollutant	Efficiency (%)	Reference
Ni(NO ₃) ₂ ·6H ₂ O	UV light	Methylene blue	99.6	Kumar et al. [5]
NiCl ₂ ·6H ₂ O	Visible light	Crystal violet	95	Ali et al. [6]
Ni(NO ₃) ₂ ·6H ₂ O	Sunlight	Congo red / MB / malachite green	93.6 / 82.4 / 91.1	Mustafa et al. [7]
Ni(NO ₃) ₂ ·6H ₂ O	UV light	Safranin / methyl orange	92.8 / 98.0	Jagan et al. [8]
Ni(NO ₃) ₂ ·6H ₂ O	UV light	Rhodamine B	85	Sundaresan et al. [9]
Ni(NO ₃) ₂ ·6H ₂ O	UV light	Acridine orange	84	Ahmad et al. [10]
NiCl ₂ ·2H ₂ O	UV light	Tetracycline	99.4	Fatimah et al. [11]
NiSO ₄ ·6H ₂ O	UV light	Allura red	91.7	Sethuraman et al. [12]
Ni(NO ₃) ₂ ·6H ₂ O	UV light	Methylene blue	75 (HT) / 65 (SG)	Present study

3.4. Proposed photocatalytic mechanism

The mechanism responsible for dye degradation over NiO nanoparticles is summarised schematically in the graphical abstract (Figure 3). Upon UV irradiation, photons with energy equal to or greater than the NiO band gap promote electrons from the valence band to the conduction band, generating electron-hole pairs. The photogenerated holes react with adsorbed water molecules or surface hydroxyl groups to form highly reactive hydroxyl radicals ($\bullet\text{OH}$), while the photogenerated electrons are scavenged by dissolved oxygen to form superoxide radical anions ($\text{O}_2\bullet^-$). Both reactive

species act as strong, non-selective oxidants that progressively attack the methylene blue chromophore, ultimately mineralising the dye into carbon dioxide, water and simple inorganic by-products [13]. Within this framework, any structural feature that limits the recombination of electron-hole pairs - such as the improved crystallinity of HT-NiO - directly increases the concentration of reactive species available for pollutant degradation, which is fully consistent with the higher photocatalytic efficiency measured for the hydrothermal sample.

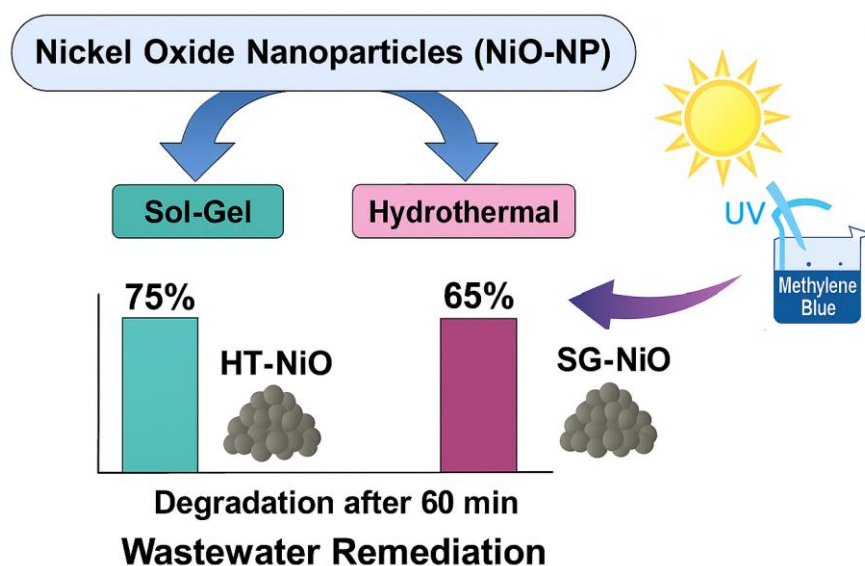


Figure 3 Graphical overview of the sol-gel and hydrothermal synthesis routes and of the photocatalytic mechanism of NiO nanoparticles.

4. Conclusion

This study demonstrates that the synthesis route strongly influences the structural, morphological and photocatalytic properties of NiO nanoparticles prepared from nickel acetate. XRD analysis confirmed the formation of pure, single-phase cubic NiO by both the sol-gel and hydrothermal routes, but revealed a markedly larger crystallite size and higher crystallinity for the hydrothermally synthesized sample (≈ 41.7 nm) compared with its sol-gel counterpart (≈ 30.2 nm). This structural advantage translated into a superior photocatalytic performance, with HT-NiO degrading about 75% of methylene blue under UV light within one hour, against about 65% for SG-NiO, despite a presumably lower specific surface area. These results show that crystalline quality, rather than nanoparticle

size alone, can be the decisive factor governing photocatalytic efficiency, by favouring more efficient separation and migration of photogenerated charge carriers. Overall, the hydrothermal method emerges as a promising, scalable and environmentally friendly strategy for producing high-performance NiO photocatalysts for the treatment of dye-polluted wastewater, such as effluents from the textile and oil-refining industries, in support of ongoing efforts toward sustainable and carbon-neutral pollution-control technologies. Future work should extend this comparison to a broader range of dyes and real industrial effluents, and evaluate the reusability and long term stability of HT-NiO under repeated photocatalytic cycles.

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