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"A Review On Influence Of Synthesis Routes On The Structural, Optical And Functional Properties Of WS_2 Nanostructures"

Dixita S. Parmar [a,c*], Yash N. Doshi [b], Piyush B. Patel [a], Hiren N. Desai [a]

[a] C.B. Patel Computer College and J.N.M. Patel Science College, Surat 395017, Gujarat, India

[b] Sciences and Humanities Department, School of Engineering, P P Savani University, Kosamba, Surat 394125, Gujarat. India

[c] Smt. Devkiba Mohansinhji Chauhan College of Commerce & Science (University of Mumbai), Silvassa 396230, UT of DNH & DD, India

Abstract

Tungsten disulfide (WS_2), a layered transition-metal dichalcogenide, exhibits tunable bandgap, rich surface chemistry, and strong environmental stability features that make it a versatile material for energy conversion and optoelectronic applications. In this comparative study, WS_2 nanoparticles were synthesized using seven distinct chemical routes: Chemical Vapor Synthesis (CVS), Hydrothermal, Sol–Gel, Solvothermal, Microwave-Assisted, Plasma-Enhanced Chemical Vapor Deposition (PECVD), and Polyol methods. The structural, morphological, and optical characteristics of the samples were investigated by X-ray diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and UV–Vis spectroscopy. Performance evaluations were conducted for photocatalytic activity, hydrogen evolution reaction (HER), photodetection, and supercapacitor behavior. Among all routes, hydrothermal WS_2 showed the best overall functionality, exhibiting an HER overpotential of 186 mV at 10 mA cm^{-2}, high photocurrent responsivity (1.12 A W^{-1}), and a specific capacitance of 284 Fg^{-1}. The results establish a clear relationship between synthesis environment, structural ordering, defect concentration, and resulting performance, providing a framework for tailoring WS_2 nanostructures for multifunctional energy devices.

Keywords: WS_2, Hydrothermal synthesis, PECVD, Photocatalysis, HER, Photodetector, Supercapacitor, Nanostructure morphology.

1. Introduction

Layered transition-metal dichalcogenides (TMDs) such as MoS_2 and WS_2 have attracted extensive research attention owing to their exceptional combination of semiconducting, catalytic, and mechanical properties. Among them, tungsten disulfide (WS_2) has emerged as a highly versatile material because of its tunable electronic structure, chemical stability, and rich defect chemistry (Stand et al., 2022). It exhibits a transition from an indirect to a direct band gap (~2.0 eV) as the number of layers decreases, accompanied by pronounced spin–orbit coupling and robust structural integrity (V. Jain, Doshi, Raval, et al., 2023). These characteristics make WS_2 suitable for a broad range of applications, including hydrogen evolution catalysis, photocatalysis, electrochemical energy storage, and photodetection (Nagaraju et al., 2018; Parmar et al., 2025).

Although mechanical or liquid-phase exfoliation techniques can produce high-quality monolayers, such methods are limited by low yield, lack of scalability, and difficulty in controlling layer number or lateral dimensions (V. Jain, Doshi, Shah, et al., 2023). In contrast, chemical synthesis routes such as hydrothermal, solvothermal, sol–gel, polyol, microwave-assisted, and vapor-phase approaches offer cost-effective and tunable alternatives for large-scale WS_2 production (Tan et al., 2017; Xu et al., 2017). Each method distinctly influences nucleation rate, growth dynamics, sulfurization efficiency, and defect formation, thereby governing the resulting particle morphology, crystallinity, and electronic characteristics (Bányai, 2018; B. Cao et al., 2018; Yang et al., 2013).

Understanding these synthesis–property correlations is essential for tailoring WS_2 nanostructures toward targeted functional applications. For instance, defect-rich or edge-exposed nanosheets are advantageous for catalytic hydrogen evolution, while highly crystalline films are preferred for photodetection and electronic devices (Y. N. Doshi et al., 2024; Tandel et al., 2024; Zhou et al., 2019). Similarly, nanostructures with large surface areas and stable layered frameworks exhibit superior charge storage and ion transport behavior, making them ideal for supercapacitors (Y. Doshi et al., 2024; Pathak, Doshi, Shah, Desai, Patel, Sahoo, et al., 2024).

Despite numerous reports on individual synthesis techniques, systematic comparative analyses under controlled precursor conditions remain limited (V. M. Jain et al., 2025; Kumari et al., 2023). Such studies are crucial to reveal how processing environments dictate the balance between crystallinity, defect density, and phase composition, which in turn affect optical absorption, charge transfer, and overall device performance (V. M. Jain et al., 2021; Tandel et al., 2025).

The present work addresses this gap by conducting a comparative experimental investigation of WS_2 nanoparticles synthesized via seven distinct chemical routes using identical tungsten and sulfur precursors. The study establishes direct correlations between synthetic parameters and the resulting structural, optical, and electrochemical characteristics. Specifically, it examines: **(a)** Photocatalytic and hydrogen evolution activity, **(b)** Optical and photodetection response, **(c)** Supercapacitor behavior, and **(d)** Structural–morphological correlations.

This comprehensive comparison aims to elucidate the mechanistic differences among synthesis routes and to identify the most effective approach for achieving high-performance, multifunctional WS_2 nanostructures suitable for energy conversion and optoelectronic applications.

2. Synthesis Procedures

2.1. Hydrothermal Route

For the hydrothermal synthesis, 0.5 mmol of tungsten hexachloride (WCl_6) and 1.5 mmol of thiourea were dissolved in 20 mL of deionized water under magnetic stirring until a clear solution was obtained. The precursor mixture was transferred to a 50 mL Teflon-lined stainless-steel autoclave and maintained at 200 °C for 24 h. After cooling to room temperature, the resulting black precipitate was centrifuged, repeatedly washed with ethanol and water, and then dried at 70 °C. Finally, the dried powder was annealed at 500 °C for 2 h under an Ar/H_2 (95 : 5) atmosphere. This approach yielded flower-like WS_2 nanosheets approximately 30–60 nm thick, characterized by highly exposed edge sites favorable for catalytic reactions (S. Cao et al., 2014; Desai et al., 2025; Pallikkarathodi Mani & Cyriac, 2020; Parmar et al., 2025).

2.2. Sol–Gel Route

In the sol–gel synthesis, tungsten ethoxide was slowly added to ethanol under constant stirring to form a transparent sol. Thiourea was then introduced as the sulfur source, initiating hydrolysis and condensation reactions. The resulting gel was aged for 12 h, dried at 70 °C to remove residual solvents, and subsequently annealed at 700 °C under argon for 3 h. The process generated uniformly distributed WS_2 nanoparticles (~40 nm) embedded within a lightly amorphous matrix, providing a high surface area and good particle connectivity (Tong et al., 2022).

2.3. Solvothermal Route

The solvothermal procedure followed the same stoichiometry as the hydrothermal route, except ethanol was used as the reaction medium to influence reduction kinetics and crystal growth. The homogeneous solution was sealed in a Teflon-lined autoclave and heated to 180 °C for 18 h. After cooling, the precipitate was collected, washed, and dried at 80 °C. This route produced highly crystalline WS_2 nanorods and microspheres, attributed to the slower diffusion and reduction rate in the organic solvent (Santhosh et al., 2023).

2.4. Polyol Route

In the polyol process, 0.5 mmol of WCl_6 and 1.5 mmol of thiourea were dissolved in 30 mL of ethylene glycol, which acted simultaneously as solvent, mild reducing agent, and stabilizer. The mixture was refluxed at 180 °C for 6 h, resulting in the formation of fine, spherical WS_2 nanoparticles with an average diameter of ~20 nm. The viscous nature of ethylene glycol suppressed agglomeration, leading to uniform particle distribution and improved crystallinity after mild annealing (Baričić et al., 2024).

2.5. Microwave-Assisted Route

For the microwave-assisted synthesis, a precursor solution containing WCl_6 and thiourea in ethanol was placed in a sealed Teflon vessel and subjected to microwave irradiation at 700 W for 20 min. Rapid and

uniform heating triggered instantaneous nucleation and growth of WS_2 nanostructures. The as-formed powder was rinsed and dried under vacuum. This route yielded porous WS_2 nanoflowers with a short synthesis time, high defect density, and increased active surface area suitable for photo- and electrochemical applications.(Panigrahi & Pathak, 2008)

2.6. Chemical Vapor Synthesis (CVS)

In the vapor-phase route, sulfurization of tungsten oxide was carried out in a dual-zone quartz-tube furnace. WO_3 powder was positioned in the central heating zone at 900 °C, while elemental sulfur was placed in an upstream zone at 250 °C. High-purity argon gas (100 sccm) transported sulfur vapors to the reaction zone, where sequential reduction and sulfurization of WO_3 occurred for 45 min. The reaction produced crystalline WS_2 layers with well-defined lamellar morphology and few structural defects (Y. Doshi et al., 2023; Y. N. Doshi et al., 2025).

2.7. Plasma-Enhanced Chemical Vapor Deposition (PECVD)

Ultrathin WS_2 films were also grown using plasma-enhanced chemical vapor deposition. Tungsten hexacarbonyl ($W(CO)_6$) and hydrogen sulfide (H_2S) gases were introduced into a low-pressure RF plasma reactor maintained at 400 °C and 150 mTorr. The plasma provided energetic radicals that accelerated sulfurization and facilitated the direct growth of 2–3 layer WS_2 sheets on silicon substrates. The resulting films exhibited high uniformity, minimal contamination, and excellent crystallinity suitable for optoelectronic device fabrication (Liu, 2019).

3. Results and Discussion

3.1 Structural and Morphological Characteristics

XRD confirmed the formation of the hexagonal (2H) phase of WS_2 across all samples. Hydrothermal and solvothermal routes showed sharp (002) peaks, suggesting preferential c-axis orientation. Sol–gel and microwave samples displayed broader reflections indicative of nanocrystalline domains (~8–12 nm).

SEM micrographs revealed distinct morphologies:

- Hydrothermal: Flower-like nanosheets with radial alignment.
- Solvothermal: Nanorods and microspherical clusters.
- Sol–Gel: Uniform nanoparticles in amorphous background.
- Microwave: Porous nanoflowers and nanosheets.
- Polyol: Monodisperse spherical nanoparticles.
- CVS/PECVD: Continuous films and triangular nanosheet domains.

EDS confirmed near-stoichiometric W:S ≈ 1:2, with minor oxygen traces only in sol–gel and microwave samples.

3.2 Raman and Optical Properties

Raman spectra displayed two characteristic modes: E_2g (~355 cm^{-1}) and A_1g (~420 cm^{-1}). Narrower linewidths for hydrothermal WS_2 indicated improved crystallinity.

Photoluminescence (PL) peaks centered between 1.95 and 2.01 eV confirmed semiconducting behavior. Redshifts (~30 meV) observed in microwave and sol–gel samples suggested sulfur vacancy-induced n-type

doping. The optical bandgaps varied from 1.85 eV (polyol) to 2.10 eV (PECVD), governed by quantum confinement and defect density.

3.3 XPS Analysis

The W 4f doublets at 32.6 eV (W^{4+} $4f_{7/2}$) and 34.7 eV (W^{4+} $4f_{5/2}$) and the S 2p peaks at 162.3/163.5 eV confirmed WS_2 formation. Weak shoulders at 35.9 eV indicated trace WO_3 residues in sol–gel samples. Hydrothermal WS_2 displayed the highest sulfurization completeness with S:W ≈ 2.04 .

3.4 Photocatalytic and HER Activity

Under visible light, WS_2 samples degraded methylene blue following pseudo-first-order kinetics ($\ln C_0/C = kt$). Hydrothermal WS_2 achieved ~92% degradation within 120 min due to large surface area (62 $m^2 g^{-1}$) and efficient charge separation between conduction and valence bands. HER polarization data revealed the following performance (Table 1):

Table 1: Performance of various methods from the HER polarization data

Method	Overpotential (mV @ 10 mA cm^{-2})	Tafel slope (mV dec^{-1})
Hydrothermal	186	68
Solvothermal	210	74
Sol–Gel	245	92
Microwave	232	88
Polyol	223	81
CVS	196	65
PECVD	172	59

The outstanding HER efficiency of PECVD and hydrothermal WS_2 stems from vertically aligned nanosheets that expose abundant active edges and enable rapid electron transfer. Electrochemical impedance spectroscopy confirmed minimal charge-transfer resistance (R_{ct} ≈ 9.6 Ω) for hydrothermal WS_2.

3.5 Photodetection Properties

All WS_2 samples exhibited measurable photocurrent under illumination (Jariwala et al., 2024). Hydrothermal WS_2 recorded the highest photocurrent density (0.82 µA cm^{-2}) and responsivity (1.12 A W^{-1}) with rise and decay times of 0.9 s and 1.3 s, respectively. PECVD films showed slightly lower responsivity (0.97 A W^{-1}) but faster transient response (~0.3 s) (Pathak, Doshi, Shah, Desai, Patel, & Sahoo, 2024). The dependence of photocurrent on light intensity ($I \propto P^{\alpha}$; $\alpha \approx 0.91$) confirmed trap-assisted photoconductive behavior (Kokni et al., 2025).

3.6 Supercapacitor Characteristics

Cyclic voltammetry of WS_2 electrodes (1 M Na_2SO_4, 0–0.8 V) showed nearly rectangular profiles for hydrothermal and solvothermal samples, indicating efficient ion transport. Table 2 shows specific capacitances obtained from GCD curves.

Table 2: obtained Specific Capacitances from GCD curves

Method	Specific Capacitance (F g^{-1} @ 1 A g^{-1})
Hydrothermal	284
Solvothermal	256
Microwave	228
Polyol	239
Sol–Gel	210
PECVD	245
CVS	231

Hydrothermal WS_2 retained 91% capacitance after 5000 cycles, attributed to its robust layered structure facilitating ion intercalation (V. M. Jain et al., 2021).

3.7 Structure–Property Correlation

Table 3: Correlation between the structural characteristics and resulting properties of WS_2 synthesized by different methods

Method	Morphology	Crystallinity	Defect Density	HER	Photocatal-ysis	Photodeti-on	Supercapactor
Hydrothermal	Flower-like nanosheets	High	Moderate	Excellent	Excellent	Excellent	Excellent
Solvothermal	Rods/clusters	High	Low	Good	Good	Moderate	Good
Sol–Gel	Nanoparticles	Medium	High	Fair	Moderate	Weak	Moderate
Microwave	Nanoflowers	Medium	High	Good	Good	Moderate	Moderate
Polyol	Spherical NPs	Moderate	Low	Good	Moderate	Moderate	Good
CVS	Triangular films	High	Low	Excellent	Moderate	Good	Good

PECVD	Few-layer films	Very High	Low	Excellent	Good	Very Good	Good

Defect engineering and crystal order were found to be decisive: hydrothermal WS_2 offered balanced crystallinity and defect density, ideal for catalytic and charge-storage applications, whereas PECVD produced highly crystalline thin films suited to fast photoresponse devices (JAIN et al., 2022; Raval et al., 2022).

4. Conclusion

This comprehensive comparison demonstrates that the physicochemical characteristics of WS_2 are strongly determined by the synthesis route. Hydrothermal synthesis produced defect-rich nanosheets with exceptional activity in HER, photocatalysis, photodetection, and energy storage. PECVD yielded few-layer WS_2 with superior crystallinity and fast photoresponse, while solvothermal and polyol routes offered scalable alternatives with tunable morphologies.

Overall, the interplay between synthesis temperature, solvent polarity, and sulfurization environment governs nucleation, defect evolution, and charge-transport properties. By optimizing these parameters, WS_2 nanostructures can be precisely tailored for next-generation catalytic and optoelectronic systems.

Credit Author statement

Dixita S. Parmar: Writing-original draft, Data curation, Investigation, Formal Analysis. **Yash N. Doshi:** Writing- review & editing, Validation, Conceptualization, Methodology. **Piyush B. Patel:** Supervision, Resources. **Hiren N. Desai:** Conceptualization**.**

Data availability

The dataset used and analyzed during the current study are available from the corresponding author on reasonable request.

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