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EFFECT OF POLYOL SYNTHESIS PARAMETERS ON PARTICLE SIZE AND CRYSTAL SIZE OF ZnO: A SYSTEMATIC REVIEW

Kormil Saputra, Rizka Ayu Fitri, Dian Wijaya Kurniawidi
University of Mataram, Mataram, Indonesia
kormil.saputra.fmipa@staff.unram.ac.id; diankurnia@unram.ac.id

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Abstract

Zinc oxide nanoparticles (ZnO NPs) are semiconductor oxide materials that have been widely developed because of their high chemical stability, relatively low synthesis cost, and broad potential applications in photocatalysis, sensors, pigments, catalysts, optical materials, and nanofluid systems. Particle size and crystallite size are critical parameters because they directly influence surface area, crystallinity, morphology, charge transfer, and the functional performance of ZnO. This study aims to examine the effects of synthesis parameters in the polyol method on the particle size and crystallite size of ZnO-based nanomaterials through a systematic literature review. Relevant literature published between 2021 and 2026 was collected from Scopus, ScienceDirect, and Google Scholar using Boolean search strategies. The selected articles were screened based on predetermined inclusion and exclusion criteria and analyzed using a descriptive-comparative approach. The review findings indicate that the polyol method influences the morphostructural characteristics of ZnO through several synthesis variables, including the type of polyol, precursor ratio, water content, base concentration, reaction temperature, reaction time, surfactants, stabilizers, and post-synthesis treatment. Ethylene glycol and diethylene glycol tend to support the formation of ZnO with smaller crystallite size and more controlled morphology, whereas prolonged reaction time and high-temperature calcination may increase crystal growth. In addition, doping and the use of surfactants can restrict crystal growth and reduce agglomeration. This review concludes that the polyol method is effective for controlling the particle size and crystallite size of ZnO; however, simultaneous optimization of synthesis parameters is required to obtain ZnO nanomaterials with morphostructural characteristics suitable for functional applications.

Keywords: ZnO Nanoparticles; Polyol Synthesis; Crystallite Size; Particle Size; Morphology

INTRODUCTION

Zinc oxide nanoparticles (ZnO NPs) are one of the metal oxide nanomaterials that are widely developed because they have good chemical stability, relatively low synthesis costs, distinctive semiconductor properties, and application flexibility in photocatalysis, sensors, pigments, optical materials, plasmonic nanofluids, and metal oxide-based catalysts(Hassan et al., 2026; Sadiq et al., 2021). In the last five years, the development of ZnO is no longer limited to pure ZnO, but has been directed at doping, the formation of hybrid materials, and combinations with other oxides or metals to improve optical, catalytic, electrochemical, and photochromic functions. For example, Co-doped ZnO has been developed as a green nano-pigment HosseiniKia & Norouzbeigi, (2024), Ho-doped ZnO was used for photodegradation of methylene blue under visible light (Phuruangrat et al., 2022), Ag-ZnO/ethylene glycol was developed as a plasmonic hybrid nanofluid (Sengwa & Saraswat, 2025), while ZnO/MoO_3 was investigated as a Schottky interface-based photochromic material (Andron et al., 2021).

The performance of ZnO is strongly influenced by its morphostructural characteristics, particularly particle size, crystallite size, crystallinity, morphology, surface area, and the presence of lattice defects (He et al., 2022). Smaller particle size generally increases the active surface area and increases the contact of the material with the reaction environment, while crystallite size and crystallinity determine the structural regularity and charge transfer dynamics (Kong et al., 2020). In photocatalytic systems, Ho-doped ZnO showed that increasing Ho content can decrease the particle size and crystallite size, while simultaneously increasing the degradation of methylene blue; a composition of 3% Ho-doped ZnO produced the highest degradation efficiency of 96.66% in 150 minutes

(Phuruangrat et al., 2022). Similar findings regarding the importance of crystallite size were also seen in Co-doped ZnO, where optimal conditions resulted in a wurtzite structure with a crystallite size of 11 nm and a strong green color (HosseiniKia & Norouzbeigi, 2024).

The synthesis method is a key factor in controlling the particle size and crystallite size of ZnO. Various approaches such as sol–gel, hydrothermal, precipitation, combustion, and polyol have been used to produce ZnO with different characteristics (A. Barzinjy, 2020; Navas et al., 2020). Among these methods, the polyol method has advantages because polyol can act as a solvent, reaction medium, reducing agent, and particle growth controller. Ethylene glycol, propylene glycol, and diethylene glycol are often used because they can support the formation of more homogeneous particles and control the nucleation process and crystal growth (Gallo-Cordova et al., 2021; Sun et al., 2021). HosseiniKia & Norouzbeigi, (2024) showed that ethylene glycol is more effective than propylene glycol in producing co-doped ZnO with smaller crystallite sizes and better crystallinity. Meanwhile, Andron et al., (2021) used diethylene glycol in the polyol route to produce ZnO and MoO_3 with morphological characteristics and surface properties that influence the photochromic effect of ZnO/MoO_3.

In addition to the type of polyol, synthesis parameters such as precursor ratio, reaction temperature, reaction time, water content, base concentration, surfactant, stabilizer, and post-synthesis treatment also play an important role in determining the final characteristics of the material. In the synthesis of Co-doped ZnO, variations in the Co:Zn ratio, water content, NaOH, reaction time, surfactant, and the ratio of surfactant to Zn have been shown to affect the crystallite size and the percentage of the amorphous phase HosseiniKia & Norouzbeigi, (2024). In the Ag-ZnO/EG system, ethylene glycol plays a role in the in-situ reduction of Ag^+ to Ag nanoparticles, while PVP functions as a stabilizing agent that strengthens the stabilization of Ag NPs and enhances the plasmonic optical response (Sengwa & Saraswat, 2025). This shows that the influence of the polyol method cannot be understood solely from the type of solvent, but must be seen as an interaction between the reaction medium, precursor composition, thermal conditions, and post-synthesis treatment.

Although various studies have addressed the synthesis of ZnO and ZnO-based materials, information on the relationship between polyol synthesis parameters and particle and crystallite sizes remains scattered across different application contexts. Some studies emphasize pigment applications and colorimetric properties (HosseiniKia & Norouzbeigi, (2024), while others focus on photocatalysis (Phuruangrat et al., 2022), methane oxidation catalysis (Ahledel et al., 2024), plasmonic nanofluids (Sengwa & Saraswat, 2025), and ZnO/MoO_3 photochromic materials (Andron et al., 2021). This situation indicates the need for a systematic review that not only summarizes the synthesis results but also compares how the type of polyol, precursor ratio, temperature, reaction time, surfactant, and treatment affect the morphostructural characteristics of ZnO. Therefore, this research was conducted through a Systematic Literature Review (SLR) approach to analyze the effect of the polyol method synthesis parameters on the particle size and crystal size of ZnO-based nanomaterials.

METHODS

1. Research Design and Problem Formulation

This study uses a Systematic Literature Review (SLR) approach to analyze the effect of polyol method synthesis parameters on particle size and crystal size of ZnO-based nanomaterials. The problem formulation used in this review is: how do polyol method synthesis parameters affect particle size and crystal size of ZnO nanomaterials? This question is used as a basis for determining the literature search strategy, article selection criteria, data extraction variables, and the direction of comparative analysis between studies.

2. Data Sources and Literature Search Strategy

Literature was collected from the scientific databases Scopus, ScienceDirect, and Google Scholar. The search focused on research articles published between 2021 and 2026, available in full text, and relevant to the synthesis of ZnO or ZnO-based materials. The search strategy was carried out using a combination of Boolean-based keywords ("ZnO nanoparticles" OR "zinc oxide nanoparticles") AND ("polyol method" OR "polyol synthesis") AND (crystallinity OR "crystal size" OR morphology).

3. Inclusion and Exclusion Criteria

Article selection was performed based on inclusion and exclusion criteria to ensure that only articles that were relevant, comparable, and had adequate characterization data were included in the analysis.

Table 1. Article inclusion and exclusion criteria

Inclusion Criteria	Exclusion Criteria
Research articles 2021–2026	Review, proceedings, book chapter, or editorial
Discussing ZnO or ZnO-based materials	Not relevant to ZnO or ZnO-based oxides
Using the polyol method or related chemical synthesis approaches	Does not explain the synthesis method clearly
Report synthesis parameters such as polyol/solvent type, precursor, temperature, reaction time, or surfactant.	There is no comparable synthesis parameter data.
Presenting characterization data such as XRD, SEM, TEM, particle size, crystal size, or morphology	Incomplete characterization data
Full text available and in English	Full text is not available or not in English

4. Article Selection Process

The article selection process follows the PRISMA process: identification, screening, eligibility assessment, and final article determination. In the identification stage, articles were collected from databases using predetermined keywords. Next, articles were screened based on title and abstract to eliminate duplication and studies that did not align with the research focus. Articles that passed the initial stage were then fully reviewed to assess their compliance with the inclusion and exclusion criteria. Articles that met all criteria were included in the data extraction and analysis stage.

5. Data Extraction

Data were systematically extracted from each selected article. Information collected included article identity, research objectives, materials, type of polyol or solvent, type and concentration of precursor, reaction temperature, reaction time, use of surfactant or stabilizer, treatment, morphology, particle size, crystallite size, and main results. This extraction was performed to facilitate comparisons between studies and to assess the relationship between synthesis parameters and the characteristics of the resulting materials.

6. Data Analysis and Synthesis

Data were analyzed using a descriptive-comparative approach. Descriptive analysis was used to describe the general characteristics of the articles, synthesis parameters, and material characterization results. Comparative analysis was conducted by comparing the effects of polyol type, reaction temperature, reaction time, precursor ratio, surfactant usage, and post-synthesis treatment on particle size, crystallite size, and ZnO morphology. The analysis results were then synthesized to identify general patterns, trends, and research gaps that still require further study.

RESULTS

1. Characteristics of Included Studies

Figure 1 shows the systematic article selection process based on the PRISMA flow. During the identification stage, 353 articles were obtained from two databases: ScienceDirect (337 articles) and Scopus (16 articles). Before entering the screening stage, 15 duplicate articles were removed using an automated tool, resulting in 338 articles being further processed. In the next stage, screening was conducted based on document type. Of the 338 articles, 152 were excluded because they did not meet the publication type criteria. These included 1 conference abstract, 83 review articles, 4 encyclopedia articles, and 64 book chapters. After this process, 186 articles were selected for screening based on title and abstract.

During the title and abstract screening stage, 130 articles were eliminated because they did not match the study focus. Thus, 56 articles were further assessed in the eligibility stage. At this stage, articles were evaluated based on full-text availability, topic suitability, keyword suitability, and publication year range. A total of 43 articles were excluded, as follows: 5 articles did not have a PDF, 34 articles were off-topic, 1 article was off-keyword, and 3 articles were not within the 2021–2026 year range. After this stage, 13 articles remained for further eligibility assessment. In the final stage, 8 articles were again excluded because their content did not include key characterization data such as XRD and SEM/TEM. Thus, the final number of articles that met all inclusion criteria and were used in the SLR analysis was 5 articles.

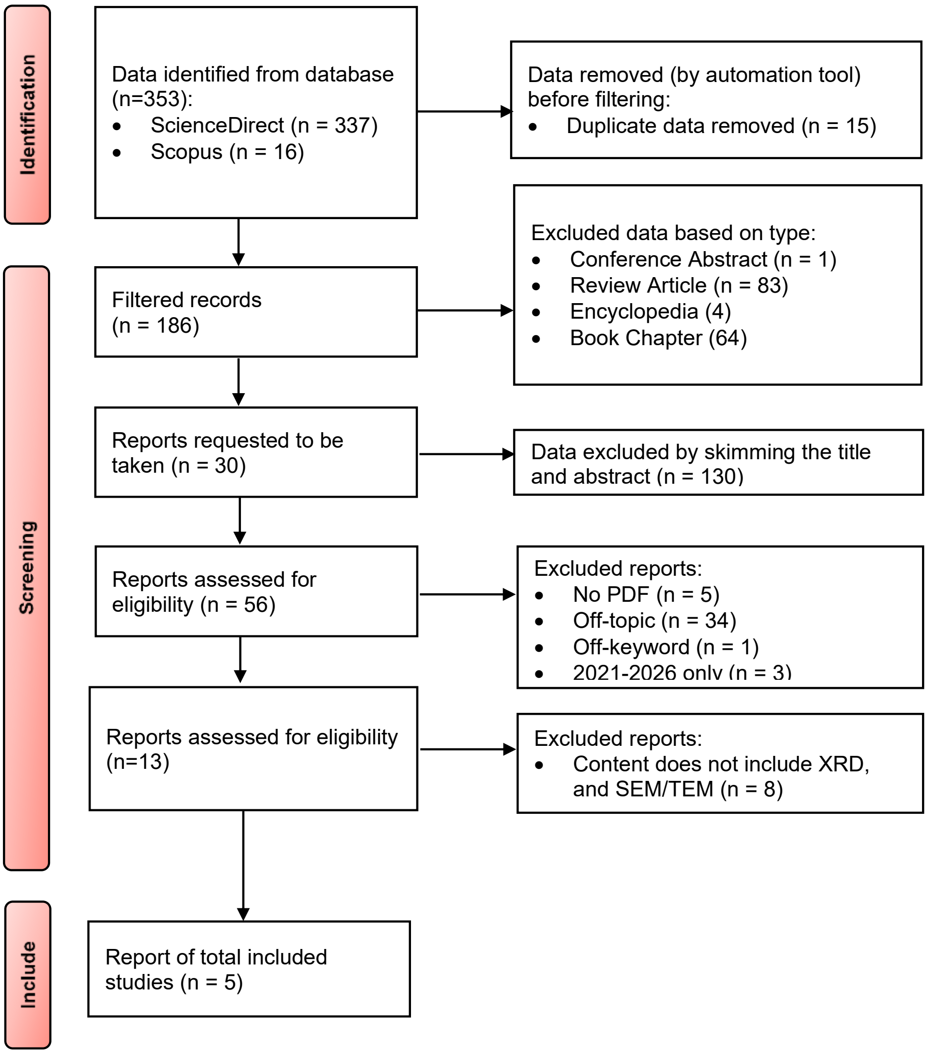

Figure 1. PRISMA Table

Articles that have passed the selection stage are then analyzed based on their bibliographic characteristics, including the country of origin of the research, journal name, publisher, keywords, and number of citations, as presented in Table 1.

Table 1. Bibliographic characteristics of the reviewed studies

Ref.	Country	Journal Name	Publisher	Keywords	Cit.
(HosseiniKia & Norouzbeigi, 2024)	Iran	Results in Engineering	Elsevier	Nano-pigment, Polyol, Taguchi, Nanoparticle, Colorimetric	7
(Ahledel et al., 2024)	Canada	Journal of Catalysis	Elsevier	Electrochemical characterization, Palladium-based catalyst, Methane oxidation, Electrochemical promotion of catalysis (EPOC)	0

(Phuruangrat et al., 2022)	Thailand	Desalination and Water Treatment	Elsevier	Ho-doped ZnO; Photocatalytic property; Combustion synthesis	5
(Sengwa & Saraswat, 2025)	India	Next Nanotechnology	Elsevier	Ag-ZnO nanohybrid; Ethylene glycol; Plasmonic hybrid nanofluids; Energy bandgaps; Plasmonic coupling	0
(Andron et al., 2021)	France	Materials Advances	Royal Society of Chemistry	ZnO/MoO_3; Photochromism; Schottky barrier; Polyol synthesis; Self-bleaching	8

Based on Table 1, the reviewed articles totaled 5 studies with a publication range of 2021–2025. The distribution of publication years shows that 2024 was the most dominant year, with 2 articles or 40% of the total studies, while 2021, 2022, and 2025 each comprised 1 article or 20%. This pattern indicates that studies related to ZnO-based materials, polyol synthesis, metal oxides, photocatalysis, catalysis, and optical properties have remained actively developed themes in recent years. The article by Andron et al., (2021) represents the earliest study in the table with a focus on enhancing the photochromic effect in the ZnO/MoO_3 system, while Sengwa & Saraswat, (2025) is the most recent article that studies ethylene glycol-based Ag-ZnO hybrid nanofluids and plasmonic coupling. From the geographical distribution aspect, the five articles come from 5 different countries, namely Iran, Canada, Thailand, India, and France, so that each country contributes 1 article or 20%. This distribution shows that research on ZnO materials and metal oxide systems is not concentrated in one particular region, but is developing in various research centers in Asia, Europe, and North America. Iran contributes through the development of Co-doped ZnO nano-pigment, Canada through the study of Pd-MOx/YSZ for methane oxidation, Thailand through Ho-doped ZnO for methylene blue degradation, India through Ag-ZnO/EG plasmonic hybrid nanofluid, and France through ZnO/MoO_3 photochromic combined material.

Based on publishers, Elsevier dominated with 4 articles or 80%, namely through Results in Engineering, Journal of Catalysis, Desalination and Water Treatment, and Next Nanotechnology. Meanwhile, the Royal Society of Chemistry contributed 1 article or 20% through Materials Advances. Elsevier's dominance indicates that most of the reviewed studies were published in international journals covering the fields of materials engineering, catalysis, nanotechnology, and environmental technology. On the other hand, the presence of Materials Advances broadens the scope of studies towards materials chemistry, specifically on the ZnO/MoO_3 interface mechanism and Schottky barrier formation as the basis for photochromic response. From a keyword perspective, Table 1 shows that the most consistent main theme is ZnO-based materials engineering and metal oxides. HosseiniKia & Norouzbeigi, (2024) emphasize the keywords nano-pigment, polyol, Taguchi, nanoparticle, and colorimetric, which indicate an orientation towards optimizing the synthesis and color character of Co-doped ZnO. Ahledel et al., (2024) has a different orientation through the keywords electrochemical characterization, palladium-based catalyst, methane oxidation, and EPOC, but remains relevant because it involves metal oxides such as ZnO, SnO_2, and FeOx as Pd catalyst promoters. Phuruangrat et al., (2022) place Ho-doped ZnO, photocatalytic property, and combustion synthesis as the main focus, while Sengwa & Saraswat, (2025) highlight Ag-ZnO nanohybrid, ethylene glycol, plasmonic hybrid nanofluids, energy bandgaps, and plasmonic coupling. Andron et al., (2021) focused on ZnO/MoO_3, photochromism, Schottky barrier, polyol synthesis, and self-bleaching. The variety of keywords indicates that the reviewed studies can be mapped into three main clusters: material synthesis and optimization, catalysis/photocatalysis, and optical, photochromic, and plasmonic properties.

Bibliometrically, the total citations of the five articles are 20, with an average of 4 citations per article and a median of 5 citations. The article with the highest citations is Andron et al., (2021) with 8 citations or 40% of the total citations, followed by HosseiniKia & Norouzbeigi, (2024) with 7 citations or 35%, and Phuruangrat et al., (2022) with 5 citations or 25%. Meanwhile, Ahledel et al., (2024) and Sengwa & Saraswat, (2025) have not received any citations based on the data in the table, so each contributes 0% to the total citations. This difference indicates that the impact of citations is not only influenced by the quality or relevance of the topic, but also by the age of the publication and the time available for the article to be absorbed by the scientific community. Article Andron et al., (2021), for example, has a temporal advantage because it has been available for longer and discusses the ZnO/MoO_3 mechanism that is relevant for the development of reversible photochromic.

1. Relationship between Synthesis Parameters and Formation Mechanism and Morphostructural Characteristics of ZnO-Based Materials

After the bibliographic characteristics are presented in Table 1, the analysis continues by reviewing the main substance of each reviewed article. The information reviewed includes the research objectives, the types of materials developed, the synthesis methods used, the mechanisms of formation or action of the materials, and the main results obtained. This summary is important to show the research direction, synthesis approach, and contribution of the findings of each study, as presented in Table 2.

Table 2. General Characteristics of Articles Based on Research Objectives, Materials, Synthesis Methods, Mechanisms, and Results

Ref.	purposes	Material	Mechanism	Results
(HosseiniKia & Norouzbeigi, 2024)	Synthesizing and optimizing cobalt-doped ZnO nanoparticles as green nano-pigments using the polyol method.	Co-doped ZnO nanoparticles	In situ Co doping onto ZnO lattice in ethylene glycol medium with NaOH, water, and surfactant control.	The optimum sample has a wurtzite structure, a crystallite size of 11 nm, a uniform morphology, and produces a bright green color with L* = 59.89, a* = −22.82, and h° = 166.89.
(Ahledel et al., 2024)	Examining the role of MOx in enhancing Pd catalysts for methane oxidation.	Pd-ZnO, Pd-SnO_2, Pd-FeOx/YSZ.	Oxygen ion transfer from MOx to Pd	Pd activity increases; Pd-ZnO excels under O_2-rich conditions, Pd-FeOx excels under reductive conditions.
(Phuruangrat et al., 2022)	Testing Ho-doped ZnO for methylene blue degradation under visible light.	ZnO and Ho-doped ZnO	Ho^{3+} acts as an electron sink, suppressing e^-/h^+ recombination, and enhancing the formation of active radicals	3% Ho-doped ZnO gave the best activity with 96.66% MB degradation in 150 min and stable up to 5 cycles
(Sengwa & Saraswat, 2025)	Examining the in situ growth of Ag NPs and coupling of SPR with ZnO in EG nanofluid	Ag-ZnO/EG and Ag-ZnO/EG/PVP hybrid nanofluids	$AgNO_3$ is reduced by EG through a polyol process to form Ag NPs which are coupled with ZnO; PVP stabilizes the Ag NPs	The absorbance increased up to 340% at 800 nm; after PVP, the Ag SPR appeared at 440 nm and the band gap was at 2.0–4.4 eV
(Andron et al., 2021)	Enhancing the photochromic effect at the ZnO/MoO_3 interface.	ZnO/MoO_3, Al-doped ZnO/MoO_3	Electron and oxygen ion transfer through the Schottky barrier at the ZnO–MoO_3 interface	The combination of Al-doped ZnO and MoO_3 polyol results in the highest coloring effect and can undergo self-bleaching

Based on Table 2, the study of HosseiniKia & Norouzbeigi, (2024) focused on the development of Co-doped ZnO nanoparticles as green nano-pigments through a polyol synthesis approach optimized using the Taguchi design. The main objective of this study is not only limited to material synthesis, but also on controlling process parameters to obtain nanoparticles with optimal crystal characteristics, morphology, and color. The selection of Co-doped ZnO is relevant because the substitution of Co ions in the ZnO structure can change the optical characteristics of the material through changes in the crystal environment and crystal field splitting, so that the color of ZnO from white can shift towards green or blue-green. The synthesis mechanism in this study shows that Co ions are directly incorporated into the ZnO lattice during the nanoparticle formation process in ethylene glycol medium. Ethylene glycol acts as a polyol medium that supports the formation of more uniform particles, smaller crystallite sizes, and better crystallinity compared to propylene glycol. In addition, the use of water and NaOH functions to regulate the hydrolysis process and the formation of ZnO, while surfactants help control particle growth, agglomeration, and the final morphology of the nanoparticles. The synthesis factors analyzed include the type of polyol, the molar ratio of cobalt nitrate to zinc acetate, the water ratio, the NaOH ratio, the reaction time, the type of surfactant, and the ratio of surfactant to zinc acetate.

The main results show that the optimum conditions produce Co-doped ZnO with a wurtzite structure with an average crystallite size of 11 nm and an amorphous phase of approximately 14%. This value indicates that the polyol method is capable of producing small crystallites with a fairly good level of structural regularity. Morphologically, the optimum sample shows a relatively uniform shape, while EDX analysis confirms the distribution of cobalt on the sample surface and FTIR indicates the formation of Zn–O bonds. Thus, the characterization results support that Co ions are successfully integrated into the ZnO structure. From the aspect of color performance, the optimum sample shows green pigment character with $L^* = 59.89$, $a^* = -22.82$, $b^* = 5.30$, $C^* = 23.45$, and $h° = 166.89$. The negative a^* value indicates a color tendency towards green, while the L^* value indicates a fairly good level of pigment brightness. This study also confirms that the increase in the presence of Co in the ZnO structure contributes to the increase in greenness of the color, while the increase in crystallite size does not directly determine the brightness. On the contrary, the percentage of amorphous phase affects the decrease in brightness because it increases light scattering and absorption.

After the objectives, materials, mechanisms, and main results of each article are summarized in Table 2, the subsequent analysis focuses on the synthesis parameters and morphostructural characteristics of the resulting materials. The parameters compared include the type of polyol or solvent, precursor concentration and type, reaction temperature and time, surfactant use, and post-synthesis treatment. Furthermore, material characteristics such as morphology, particle size, and crystallite size are also presented to examine the relationship between synthesis conditions and material structural properties. A summary of these parameters is shown in Table 3.

Table 3. Synthesis Parameters and Morphostructural Characteristics of ZnO/Metal Oxide-Based Materials in the Reviewed Articles

Ref.	**Types of polyols**	**Precursor concentration**	**Precursor type**	**Reaction temp. (°C)**	**Reaction time**	**Surfactant Conc.**	**Treatment**	**Morph.**	**Particle Size (nm)**	**Crystal Size (nm)**
(Hosseini Kia & Norouzbeigi, 2024)	EG & PG	Co:Zn molar ratio (0.01–0.10)	Cobalt nitrate & Zinc acetate	120	5,7 and 9 hours	Molar ratio of surfactant :Zn (0; 0.02; 0.20)	Polyol and calcination 500 °C	Uniform rod-like nanoparticles	500–1.000	11-34,2
	EG	Pd:MOx = 50:50 at.%	$PdCl_2$; $Fe(NO_3)_3.9H_2O$; $Zn(CH_3CO_2)_2.2H_2O$; $SnCl_2$	190	NR	NA	Two-step polyol reduction and deposition on YSZ	Dominantly spherical	10–20	NR
(Phuruangrat et al., 2022)	Ethanol	$Zn(NO_3)_2.6H_2O$ = 0,01 mol; $Ho(NO_3)_3.6H_2O$ = 0–5 wt%	$Zn(NO_3)_2.6H_2O$; $Ho(NO_3)_3.6H_2O$; tartaric acid; NaOH	600	2 hours	NA	Polyol, drying 80 °C, and calcination 600 °C	Nanoparticles with irregular shapes	15,78 – 65,35	27–36
(Sengwa & Saraswat, 2025)	EG	ZnO = 0,01 wt%; $AgNO_3$ = 1,0 wt%; PVP = 1,0 wt%	ZnO nanopowder; $AgNO_3$; PVP	25	1-72 hours	1.0 wt%	Stirring, sonication, in situ polyol reduction, aging, PVP stabilization	NA	72 – 100	NA
(Andron et al., 2021)	DEG	0,1 mol.L^{-1}	$ZnCl_2$; $AlCl_3$; $(NH_4)_2MoO_4$	180	3 hours	NA	Polyol, annealing, and mixing of ZnO/MoO_3 50:50 wt%	ZnO: spherical aggregates; MoO_3: platelets	100–1.000	7–25

Based on Table 3, the synthesis approaches for ZnO-based materials and metal oxides in the reviewed articles show quite wide variations, both in terms of reaction medium, precursor type, temperature, reaction time, and post-synthesis treatment. Of the five studies, four articles involved polyol-based approaches or polyol mediums, namely the use of ethylene glycol (EG), propylene glycol (PG), and diethylene glycol (DEG), while one article used ethanol in the combustion method. This indicates that the term polyol in the table needs to be used carefully, because ethanol in the synthesis of Ho-doped ZnO acts as a combustion solvent, not a synthetic polyol.

Nominally, the study of HosseiniKia & Norouzbeigi, (2024) used EG and PG as polyol medium with a Co:Zn molar ratio of 0.01–0.10, a reaction temperature of 120 °C, and a reaction time of 5, 7, and 9 hours. This synthesis also involved variations in the surfactant to Zn ratio of 0; 0.02; and 0.20, followed by calcination at 500 °C. The results showed that the synthesis conditions were able to produce Co-doped ZnO with a uniform rod-like morphology and a crystallite size of 11–34.2 nm, with the optimum sample having a crystallite size of 11 nm. In the study of Ahledel et al., (2024), the synthesis used EG through two-step polyol reduction to produce a Pd-MOx catalyst with a nominal composition of Pd:MOx = 50:50 at.%. The precursors used included $PdCl_2$, $Fe(NO_3)_3.9H_2O$, $Zn(CH_3CO_2)_2.2H_2O$, and $SnCl_2$, with a reaction temperature of 190 °C. Different from the first study, this research did not focus on surfactants, but on the formation of bimetallic nanoparticles and their deposition on YSZ solid electrolyte. The particle morphology was reported to be predominantly spherical with a particle size of around 10–20 nm, while the numerical crystallite size was not specifically reported.

The study by Phuruangrat et al., (2022) differs methodologically because it did not use a polyol method, but rather tartaric acid-assisted combustion synthesis. Ho-doped ZnO material was synthesized from 0.01 mol of $Zn(NO_3)_2.6H_2O$ and 0–5 wt% $Ho(NO_3)_3.6H_2O$, with ethanol as the solvent. The precursor gel was dried at 80 °C for 12 hours, then calcined at 600 °C for 2 hours. Morphologically, the particles were irregular in shape, but the particle size decreased with increasing Ho doping, from 65.35 nm in pure ZnO to 15.78 nm in 5% Ho-doped ZnO. The crystallite size also decreased from 36 nm to 27 nm, indicating that Ho doping plays a role in limiting ZnO crystal growth. In Sengwa & Saraswat, (2025), the synthesis was carried out in EG medium at a low temperature of around 25 °C, with a composition of ZnO = 0.01 wt%, $AgNO_3$ = 1.0 wt%, and PVP = 1.0 wt%. This system did not produce dry powder, but plasmonic hybrid nanofluids of Ag-ZnO/EG and Ag-ZnO/EG/PVP. The treatments used included stirring, sonication, in situ polyol reduction, aging for 1–72 hours, and stabilization using PVP. The morphology and crystallite size were not directly characterized by SEM/TEM/XRD, but the size of Ag nanoparticles was estimated to be around 72 nm based on the Ag SPR peak at 440 nm. Therefore, the morphology and crystallite size columns for this article are safer to write NA/NR.

Meanwhile, Andron et al., (2021) used DEG as a polyol medium to synthesize ZnO, Al-doped ZnO, and MoO_3. The precursor concentration was set at 0.1 mol L^{-1}, with ion sources consisting of $ZnCl_2$, $AlCl_3$, and $(NH_4)_2MoO_4$. The reaction was carried out at 180 °C for 3 hours, then part of the sample was annealed and mixed as a 50:50 wt% ZnO/ MoO_3 system. Morphologically, the ZnO from the polyol formed spherical aggregates, while MoO_3 tended to form platelets. The crystallite size of the ZnO from the polyol ranged from 7–25 nm, depending on the doping and annealing conditions, while the particle/aggregate size ranged from hundreds of nanometers to micrometers.

DISCUSSION

Based on the reviewed articles, the polyol method affects the particle size and crystallite size of ZnO by controlling the nucleation process, crystal growth, surface stabilization, and post-synthesis treatment. In the synthesis of Co-doped ZnO, HosseiniKia & Norouzbeigi, (2024) showed that the use of ethylene glycol (EG) resulted in better crystallinity and smaller crystallite size compared to propylene glycol, with an optimum crystallite size of 11 nm. This finding is consistent with Walunj et al., (2023), who reported that ZnO synthesized based on EG has a wurtzite phase, nanosphere/aggregate morphology, and a crystallite size of approximately 18.09 nm; this indicates that the polyol medium is able to regulate the formation of crystal nuclei and limit particle growth. Geleta, (2024) also emphasized that the structure and optical characteristics of ZnO from polyol are strongly influenced by the synthesis conditions, so that the crystallite size cannot be separated from the interaction between the precursor, polyol medium, and reaction conditions.

Precursor ratio, water content, and base are parameters that directly affect particle size and crystallite size. In Co-doped ZnO, variations in the ratios of Co:Zn, H_2O:Zn, NaOH:Zn, and reaction time affect crystallite size and the percentage of amorphous phase (HosseiniKia & Norouzbeigi, 2024). Mechanistically, increasing the base accelerates the formation of hydroxide species that are the core of ZnO formation, but too high OH^- concentrations can accelerate aggregation and particle growth. Abdulqodus et al., (2025) confirmed that the pH of the growth solution affects the nucleation, morphology, size, and structural integrity of ZnO, while Ejsmont & Goscianska, (2023) showed that pH adjustment can control the crystallinity, particle size, and morphology of ZnO. Thus, the effect of the polyol method on ZnO size is not independent, but is strengthened by the balance between hydrolysis, pH, and crystal growth rate.

Surfactants and stabilizers are also important variables in controlling particle morphology and size. In the study of HosseiniKia & Norouzbeigi, (2024), Triton X100 played a role in controlling the growth of Co-doped ZnO, thus obtaining a uniform rod-like morphology. In a different system, Walunj et al., (2023) used TBAB as a capping agent in the synthesis of polyol-based ZnO, which helped produce particles with a size of 10–25 nm based on FESEM. Meanwhile, Sengwa & Saraswat, (2025) used PVP to stabilize Ag nanoparticles in the Ag-ZnO/EG/PVP system, thus demonstrating that organic agents can control the stability of dispersion and particle growth in polyol-based systems. At the application level, Baruah et al., (2024)showed that various ZnO morphologies, such as nanoparticles, microspheres, belts, and triangular structures, resulted in different performance responses in DSSCs; this confirms that morphology is not merely a characterization result, but a parameter that determines the function of the material.

Post-synthesis treatments such as calcination and annealing have a dual role. On the one hand, calcination increases crystallinity, removes organic residues, and enhances the formation of the wurtzite phase. On the other hand, high temperatures can enlarge the crystallite size due to atomic diffusion, sintering, and the coalescence of small crystallites into larger ones. Sharma et al., (2023) showed that increasing the calcination temperature from 400–600 °C increased the crystallite size of ZnO and decreased the band gap from 3.15 to 3.05 eV. Saidani et al., (2024)also reported that increasing the calcination temperature can change the size, surface area, shape, and optical behavior of ZnO; at higher temperatures, crystal growth increases and the surface area decreases. Therefore, increasing crystallinity through calcination must be balanced with crystallite size control to ensure the material maintains a high active surface area.

Comparison between polyol and non-polyol studies shows that the final size of ZnO is not only determined by the synthesis method, but also by the dopant and treatment. In Phuruangrat et al., (2022), Ho-doped ZnO synthesized by combustion and calcination at 600 °C for 2 hours showed a decrease in crystallite size from 36 nm in pure ZnO to 27 nm in 5% Ho-doped ZnO. This pattern indicates that dopants can inhibit crystal growth through lattice distortion and changes in surface energy. Rafaie et al., (2021) showed that Al-doped ZnO can also be used to modify the structural and photocatalytic properties of ZnO, while Sa-nguanprang et al., (2020)showed that rare-earth doping in ZnO enhances photocatalytic activity through defect formation and charge trapping. This means that the effect of the polyol method on particle size should be analyzed together with the effect of the dopant, because the dopant can reduce or enlarge the crystallites depending on its ionic radius, concentration, and compatibility in the ZnO lattice.

Critically, the reviewed articles suggest that the term “polyol method” needs to be used more selectively. Ahledel et al., (2024); Andron et al., (2021); HosseiniKia & Norouzbeigi, (2024); Sengwa & Saraswat, (2025) do involve polyols such as EG or DEG, but Phuruangrat et al. (2022) uses ethanol in a combustion method, making it more appropriately positioned as a comparative study. Furthermore, Sengwa & Saraswat, (2025) focused more on plasmonic nanofluids and the in situ growth of Ag nanoparticles, rather than on direct measurements of ZnO crystal size. This limitation is important because an overly broad SLR can confound the effects of polyol, doping, calcination, and hybrid formation. Thus, the analysis must distinguish between the influence of the polyol medium as a nucleation and growth controller, the influence of the dopant as a lattice modifier and defects, and the influence of the thermal treatment as a controller of crystallinity and crystal enlargement.

The polyol method influences the particle size and crystal size of ZnO through four main mechanisms. Polyol controls nucleation through metal ion coordination and precursor solubility regulation, as seen in the use of EG in the synthesis of Co-doped ZnO and polyol-mediated ZnO (HosseiniKia & Norouzbeigi, 2024; Walunj et al., 2023). Polyol limits crystal growth through viscosity, hydroxyl groups, and surface stabilization, as seen in the DEG system in ZnO/MoO_3 (Andron et al., 2021). Furthermore, particle size is influenced by pH, water, base, and surfactant because these parameters determine the rate of hydrolysis, aggregation, and morphology formation(Abdulqodus et al., 2025; Ahmed et al., 2023). Then, the crystallite size can increase after calcination or annealing because small crystallites merge into larger crystallites (Saidani et al., 2024; Sharma et al., 2023). Thus, the polyol method can produce small-sized ZnO with more controlled morphology, but only when the type of polyol, precursor ratio, water content, pH, temperature, reaction time, surfactant, and post-synthesis treatment are optimized simultaneously.

CONCLUSION

Based on the results of a systematic study, the polyol method affects the particle size and crystallite size of ZnO-based nanomaterials through controlling the nucleation process, crystal growth, surface stabilization, and post-synthesis treatment. The type of polyol, precursor ratio, water content, base concentration, reaction temperature, reaction time, surfactant, stabilizer, and calcination/annealing are proven to be important parameters that determine the morphostructural character of ZnO. In general, the use of polyol media such as ethylene glycol and diethylene glycol can produce more homogeneous particles and smaller crystallite sizes if the synthesis conditions are properly controlled. However, increasing the reaction time and excessive thermal treatment can increase the crystallite size due to grain growth, atomic diffusion, and agglomeration. Thus, the polyol method is effective for controlling the particle size and crystallite size of ZnO, but its effectiveness is highly dependent on the simultaneous optimization of the reaction medium, precursor composition, synthesis conditions, and post-synthesis treatment.

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