

Synthesis of Zinc Oxide Nanoparticles Through Polymeric Precursor Route and their Integration into Functional Nanocomposites

Puja Singh¹, S.V.A.R. Sastry^{2,*}

Abstract

A polymer precursor approach for creating zinc oxide (ZnO) nanoparticles with regulated size and shape is described in this work. By stabilising and capping particles, the polymer-assisted pathway efficiently reduces particle agglomeration while facilitating controlled nucleation and growth. The surface shape and dispersion of the produced ZnO nanoparticles were assessed using the scanning electron microscopy (SEM). When compared to traditional synthesis techniques, the addition of polymer greatly enhanced surface homogeneity and particle dispersion. By encouraging the breakdown of organic components and oxide production, thermal treatment of the polymer-metal precursor allowed the synthesis of well-defined ZnO nanoparticles. The suggested technique provides a straightforward, affordable, and scalable way to create high-quality ZnO nanoparticles, which makes it appropriate for use in sensing, catalysis, and other cutting-edge technologies. Furthermore, ZnO-based nanocomposites with improved mechanical, thermal, and functional properties can be created by successfully incorporating the synthesised ZnO nanoparticles into polymer matrices. These composites increase ZnO's use in cutting-edge coatings, electronics, and energy-related systems. Furthermore, ZnO nanoparticles' performance in composite systems is significantly influenced by their surface features and structural attributes. In order to achieve superior composite characteristics, the inclusion of nanoscale particles with increased dispersion improves interfacial bonding inside the polymer matrix. This study's polymer precursor pathway naturally promotes uniform particle distribution, which lessens phase separation problems that are frequently seen with traditional mixing methods. Better control over particle matrix interactions is another benefit of this method, which can have a big impact on the final materials' functional efficiency, thermal resistance, and mechanical durability.

Keywords: ZnO nanoparticles, polymeric precursor method, ZnO-based nanocomposites, surface morphology, polymer nanoparticle integration

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INTRODUCTION

Nanoparticles are gaining popularity due to their unique chemical and physical properties, which make them ideal for a wide range of technological applications in biotechnology, electronics, optics and electronics [1]. Technically speaking, they are useful in a variety of applications due to their ability to modify their optical and electrical properties by methodically adjusting particle size and form. One important feature of nanomaterials is that the ratio of atoms on the surface to total atoms decreases with their size [2].

The material rises in bulk. The physical and

electrical properties of these materials are significantly altered as a result. By modifying the particle size or surface, chemists can readily change the electrical and chemical properties of materials [3]. Numerous important industrial sectors, such as food production, agriculture, and pharmaceutical distribution, have been transformed by nanoparticles (NPs). Emulsion solvent extraction, double emulsion and evaporation, salting out, emulsion diffusion, and solvent displacement/precipitation are some of the chemical synthesis methods for nanoparticles. However, a new type of pollution has entered the environment as a result of the large-scale production of nanoparticles [4].

Reducing the environmental impact of chemical pollution requires the development of innovative and practical methods to overcome the drawbacks of chemical treatments, increase production, and lower costs. Because of their potential applications in solar cells, gas sensors, photocatalysts, and nanomedicines, zinc oxide (ZnO) nanoparticles (NPs) have drawn a lot of attention recently [5]. Zinc oxide (ZnO) nanoparticles have attracted significant attention in areas such as electronics, optics, and catalysis because of their distinctive physicochemical properties. They possess a wide band gap of around 3.37 eV, along with high exciton binding energy and good electron mobility, which make them suitable for optoelectronic applications like UV detectors, light-emitting devices, and solar cells. In addition, their large surface area and active surface sites enhance their effectiveness in catalytic and photocatalytic processes, particularly in environmental and energy-related applications.

The characteristics of zinc oxide (ZnO) vary depending on the size, shape, and morphology of its particles. In particular, nanocrystalline ZnO has improved chemical and physical properties because of its changed electronic structure and increased surface area [6]. The development of cutting-edge technologies like ultraviolet lasers, nanogenerators, solar cells, and chemical and biological sensors could be greatly aided by zinc oxide (ZnO), a well known semiconductor with a large energy gap. ZnO-based energy harvesting devices called nano generators are made up of arrays of piezoelectric ZnO nanorods or nanowires positioned between two electrodes on flexible substrates. The electrical output signals produced by the mechanical deformation of ZnO nanorods or nanowires can be stored in energy storage devices or used right away [7]. Because of its piezoelectric and pyroelectric properties, ZnO can be used for a variety of purposes, including sensing, converting, producing energy, and serving as a photocatalyst in the production of hydrogen. It is an essential component in the ceramics industry because of its rigidity, low toxicity, and piezoelectric constant [8]. Due to their remarkable performance in the domains of electronics, optics, and photonics, zinc oxide (ZnO) nanowires are highly sought after for numerous applications. UV lasers, light-emitting diodes, solar cells, nanogenerators, gas sensors, photodetectors, and photocatalysts are some examples of these applications [9].

Wurtzite nanoparticles with a high degree of crystallinity were produced by the solvothermal method at low temperatures [10]. These spherical nanoparticles measured 8.4 ± 1.3 nm in diameter. With a recovery period of 22 seconds, the synthetic specimen demonstrated the capacity to detect butanol at concentrations as high as 500 parts per million (ppm). The developed sensor had a number of advantages, including a quick and effective reaction time, a short recovery period, and outstanding stability when exposed to n-butanol gas. Scanning electron microscopy (SEM) was used to analyse the generated ZnO sample [11].

The increasing need for high-performance nanomaterials has raised interest in creating synthesis methods that provide accurate control over particle properties while preserving affordability and scalability. The polymeric precursor pathway has become a viable substitute for traditional synthesis techniques in this setting. This method creates a homogenous polymer-metal combination that, when heated, produces uniformly distributed metal oxide nanoparticles [12]. In the polymeric precursor route, the polymerization step is essential for developing a uniform precursor resin, where metal ions become embedded within a three-dimensional polymer network. This arrangement allows the metal ions to be evenly distributed at the molecular level, which helps in achieving controlled nucleation during heat treatment and reduces the chances of early-stage particle agglomeration. Polymer chains are essential

for controlling nucleation and development, which stops unchecked aggregation and improves nanoparticle dispersion [13].

Chelation of metal ions within the polymer matrix plays an important role in improving the uniformity of the resulting nanoparticles. By holding the metal ions in a coordinated manner throughout the precursor, it ensures an even distribution and avoids localized concentration differences. This uniform arrangement supports consistent nucleation and growth during calcination, leading to better control over particle size and overall structural uniformity. The polymeric precursor approach offers notable benefits in terms of compositional homogeneity and structural stability in addition to managing particle size and shape [14]. Highly pure and defect-controlled nanostructures can be formed more easily when precursors are mixed at the molecular level. Additionally, this process enables the tweaking of ZnO nanoparticles' optical, electrical, and catalytic properties by including functional additives or dopants during synthesis. Because of its adaptability, this method is ideal for creating materials that are specifically suited for cutting-edge applications. The incorporation of nanoparticles into functional matrices to create nanocomposites is a significant facet of current nanotechnology research. ZnO nanoparticles can be added to polymeric, ceramic, or hybrid matrices to greatly improve the final material's overall performance. These nanocomposites display higher electrical conductivity, UV shielding, antibacterial activity, and mechanical strength and thermal stability. The effectiveness and stability of these composite systems are significantly influenced by the interfacial interaction between ZnO nanoparticles and the host matrix. Furthermore, ZnO-based nanocomposites have shown great promise in cutting-edge fields like smart coatings, environmental remediation, and energy storage. For example, they can be used in photocatalytic systems to effectively degrade organic contaminants when exposed to UV or visible light. Similarly, the development of wearable electronics and self-powered gadgets has been made possible by their inclusion into flexible substrates [15]. In addition to increasing performance, the synergistic coupling of ZnO nanoparticles with appropriate matrices expands the range of feasible applications.

Despite tremendous advancements, issues like large-scale manufacturing, synthesis repeatability, and particle agglomeration still require attention. In order to better regulate the properties of nanoparticles, ongoing research efforts are concentrated on improving synthesis parameters and investigating new strategies [16]. In this sense, the polymeric precursor approach is a workable option to get over these restrictions and create next-generation nanomaterials with improved dependability and performance.

MATERIALS AND METHODS

Materials Required

Polyvinyl Chloride (PVC) and $\text{Zn}(\text{NO}_3)_2$ (Merck) were utilised exactly as supplied. Throughout the entire experiment, doubly distilled water and analytical-grade chemicals were utilised.

Synthesis of ZnO Nanoparticles and Nanocomposite Formation

Zinc nitrate [$\text{Zn}(\text{NO}_3)_2$] was added to a polyvinyl chloride (PVC) matrix at the proper concentration in order to create ZnO nanoparticles using the polymer precursor method. Using a magnetic stirrer, the resultant solution was first mixed uniformly for 45 minutes at about 80 °C. In order to encourage the precursor's synthesis, a refluxing procedure was then conducted for roughly 90 minutes at a temperature close to 550 °C. To separate the nanoparticles, the reaction mixture was then centrifuged for 15 minutes at 6000 rpm. To get rid of any remaining contaminants, the precipitate was repeatedly cleaned using ethanol and deionised water. To guarantee a consistent particle size distribution, the purified material was subsequently dried, crushed into a fine powder, and run through a 100 µm mesh screen. This altered process is in line with widely documented polymer precursor techniques for metal oxide nanoparticle production [17]. The in-situ creation of ZnO-based nanocomposites, where residual or partially dissolved polymer phases may interact with ZnO particles, impacting their dispersion and interfacial properties, is also made possible by the presence of the polymer matrix during synthesis.

The synthesis procedure of ZnO nanoparticles using the polymer precursor method is illustrated schematically in Figure 1

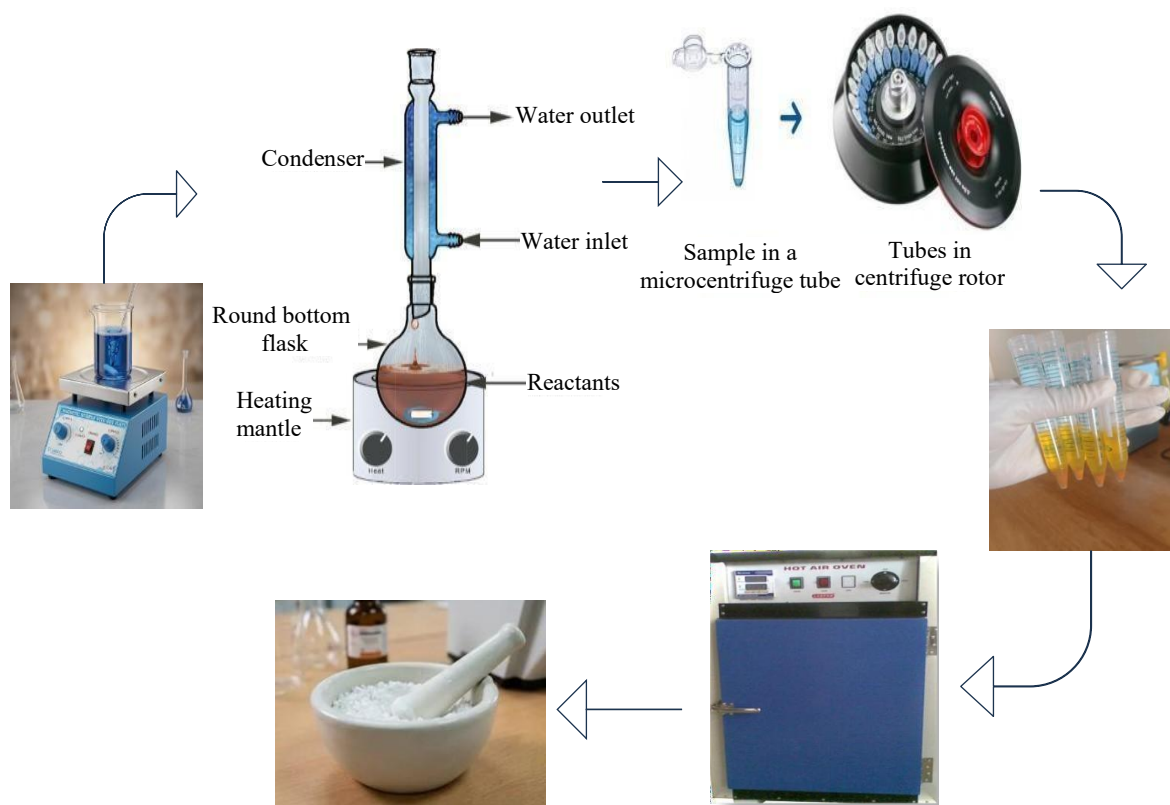


Figure 1. Schematic representation of the synthesis of ZnO nanoparticles via the polymer precursor method.

RESULTS AND DISCUSSIONS

The SEM pictures were taken using ZEISS (SUPRA) equipment. To avoid charging effects, the sample was drop cast onto a silicon wafer and then lightly coated with gold or platinum. The samples were then placed inside the chamber [18].

The surface appearance, particle dispersion, and microstructural properties of ZnO nanoparticles produced using the polymer precursor technique were investigated using scanning electron microscopy (SEM). Figure (2a–d) shows SEM images of ZnO nanoparticles and offers thorough understanding of the prepared material's morphological development and aggregation behaviour.

The morphology at a very high magnification, equivalent to a field view of about 2 μm , is shown in Figure 2a (50,000 $\times$), where small nanoscale particles are seen linked to larger flake-like structures. These granular characteristics verify ZnO production at the nanoscale, yet strong interparticle interactions clearly show partial agglomeration [19].

A region of around 10 μm is covered by Figure 2b and 2c (10,000 $\times$), which further demonstrates plate-like, non-uniform structures with a broad size distribution. This implies that during synthesis and post-processing, smaller nanoparticles have aggregated into bigger secondary structures[20]. The surface texture is more clearly visible in Figure 2d (25,000 $\times$), which corresponds to a region of roughly 4 μm . The surface texture is rough and heterogeneous, with small particles attached to larger compact sections. The conditions of synthesis have a significant impact on the observed morphology [21]. Initially, the PVC polymer matrix controls the nucleation and development of ZnO nanoparticles by acting as a stabilising agent. On the other hand, irregular and flake-like structures are formed after high-temperature treatment ($\sim 550^\circ\text{C}$) due to the polymer's breakdown [22]. Furthermore, procedures including centrifugation, washing, drying, and grinding aid in the aggregation of nanoparticles. The successful creation of ZnO nanoparticles with an agglomerated, non-spherical, plate-like morphology

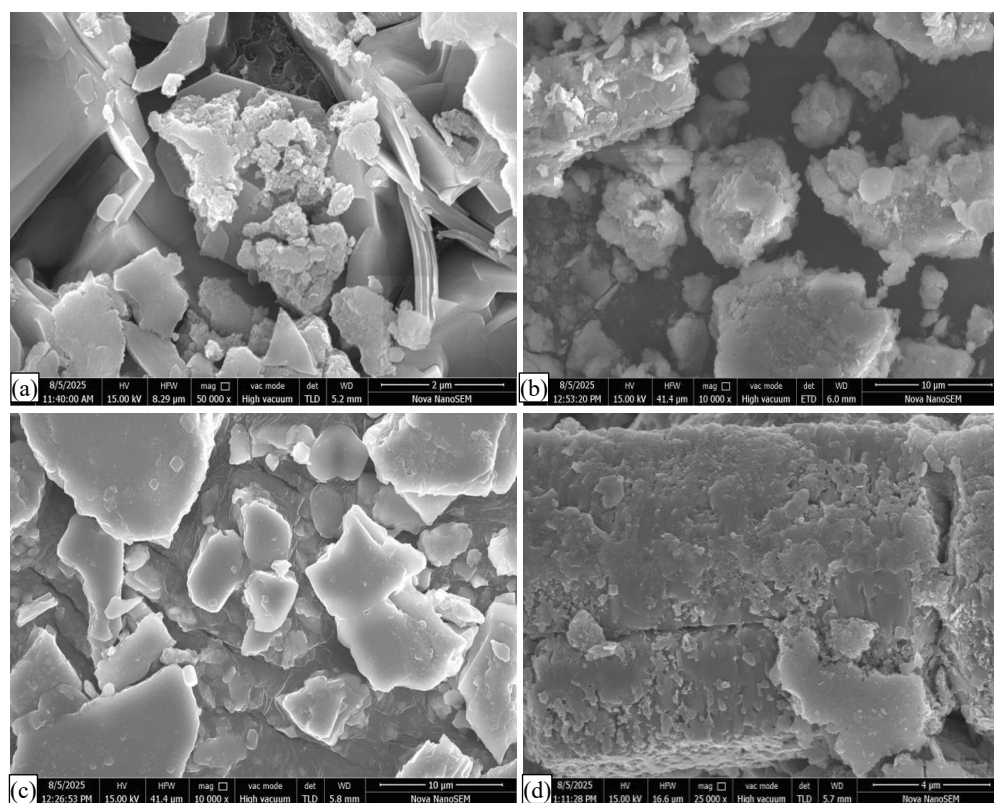


Figure 2. (a–d) SEM images of ZnO nanoparticles synthesised via the polymer precursor method.

made up of nanoscale substructures is generally confirmed by the SEM investigation [23]. Applications needing high surface activity benefit from this shape, which has more surface roughness.

Strong interfacial interactions between ZnO nanoparticles and the polymer precursor may also be indicated by the observed agglomerated and flake-like morphology. The production of ZnO-based nanocomposites, where uniform dispersion and interfacial adhesion are critical factors in determining material performance, benefits from such interactions [24]. The structural properties found in SEM indicate that the synthesized material can serve as an effective filler in polymer matrices for composite construction. Figure 2(a–d) presents the SEM micrographs of ZnO nanoparticles synthesized via the polymer precursor method at different magnifications.

CONCLUSIONS

Zinc nitrate was used as the metal source and polyvinyl chloride (PVC) as the polymer matrix in this study's effective synthesis of ZnO nanoparticles using a polymer precursor technique. ZnO nanoparticle generation was successfully facilitated by the synthesis technique that was used, which included controlled magnetic stirring, refluxing, centrifugation, repeated washing, and subsequent thermal treatment. In addition to influencing the particles' ultimate morphology and aggregation behaviour, the methodical processing conditions guaranteed appropriate particle nucleation and growth.

The produced ZnO is made up of nanoscale primary particles that are organised into irregular, flake-like, and plate-shaped structures, creating bigger agglomerates in the micrometre range, according to SEM examination. The intrinsic high surface energy of ZnO nanoparticles, which promotes particle clustering during drying and calcination, is responsible for the occurrence of such agglomeration.

Furthermore, diverse and rough surface textures are formed as a result of the polymer matrix's breakdown at high temperatures ($\sim 550^\circ\text{C}$), suggesting a significant impact of the polymer precursor on particle growth and structural evolution. The resulting morphology shows a hierarchical structure with

increased surface roughness, which is very advantageous for surface- driven processes, even though discrete spherical nanoparticles were not seen.

The polymer precursor method presented in this work is a feasible strategy for producing ZnO nanoparticles on a large scale because of its many benefits, which include simplicity, affordability, and scalability. According to recent research, these morphologically varied ZnO nanostructures are ideal for uses in gas sensing, photocatalysis, and environmental remediation, where more surface area and active sites improve performance. Additionally, because of their biocompatibility and capacity to produce reactive oxygen species, ZnO nanoparticles have demonstrated encouraging potential in biomedical applications, particularly in antibacterial activity and drug delivery systems. Their use in coatings, electronic gadgets, and UV-blocking materials highlights their significance in technology. This work emphasises the potential of the polymer precursor technique in the creation of ZnO-based nanocomposites in addition to nanoparticle synthesis. ZnO nanoparticles' interaction with the polymer matrix can be used to create composite materials with improved physicochemical characteristics. For use in high-performance coatings, dielectric materials, sensors, and biomedical devices, these composites show great promise. Future research might concentrate on improving the creation of composites and assessing their functional qualities in practical settings.

Overall, the results of this investigation show that the polymer precursor approach is a successful way to create ZnO nanoparticles with desired structural properties. Despite aggregation, the observed morphology offers substantial benefits for a range of cutting-edge applications, making these nanoparticles attractive options for further study and practical use. The results of this work further emphasise the importance of synthesis-structure interactions in defining the overall performance of ZnO-based materials, in addition to the findings already provided. The morphological features that have been observed, especially the creation of hierarchical and agglomerated structures, indicate that surface qualities can be optimised for specific applications through controlled adjustment of synthesis parameters. Future studies may be able to better regulate particle dispersion and structural uniformity by fine-tuning processing parameters such precursor concentration, thermal treatment profile, and polymer composition. Furthermore, the incorporation of ZnO nanoparticles into functional nanocomposites creates new possibilities for creating cutting-edge materials with many uses. In addition to strengthening interfacial bonding, the robust interaction between ZnO and the polymer matrix also improves the composite system's stability, durability, and load transmission. These qualities are especially crucial for applications that need to function for an extended period of time under situations of mechanical, thermal, or environmental stress. The produced ZnO nanoparticles and their related nanocomposites show great promise for use in next-generation technology. They are good choices for energy-related devices, sensitive sensing platforms, and high-efficiency catalytic systems due to their increased surface activity and structural flexibility. Specifically, the capacity to customize particle distribution and surface shape offers a major benefit in increasing functional efficiency in a variety of industries.

Future research might concentrate on reducing particle aggregation using sophisticated surface modification or dispersion methodologies. Furthermore, thorough characterisation using methods like transmission electron microscopy (TEM), X-ray diffraction (XRD), and spectroscopic analysis may offer more profound understanding of phase composition, interfacial interactions, and crystallinity. These materials' practical viability and commercial relevance will be further established by assessing their performance under actual operational settings.

Overall, the current study highlights the potential of the polymer precursor approach for ZnO nanoparticle synthesis to advance the creation of high-performance nanocomposite systems in addition to confirming its efficacy. Future studies aiming at creating nanostructured materials with better functioning, increased stability, and a wider range of applications can build on the knowledge gathered from this work.

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