

Study of The Effect of Calcination Temperature on the Phase Composition of ZnO Powder Synthesized via The Sol-Gel Method

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ABSTRACT

This study investigated the effect of calcination temperature on the phase composition and crystal size of zinc oxide powders synthesised by the sol-gel method. Zn powder, HCl and NaOH were used as precursors in a multi-step process involving dissolution, titration, gel formation, leaching, drying and calcination at temperatures ranging from 300°C to 700°C for 2 hours. Rietveld analysis of X-ray diffraction (XRD) data using MAUD and Rietica software determined phase composition and crystal size. Initial analysis identified a single simonkolleite phase ($Zn_5(OH)_8Cl_2$) prior to calcination, which disappeared at 500°C. Wurtzite (ZnO) appeared at 300°C, accompanied by secondary phases ($NaCl$ and $ZnCl_2$). The wurtzite content increased to 81.42 wt% at 700°C. Calcination temperature also influenced crystal size, which ranged from 27.34 nm to 110.61 nm for wurtzite at different temperatures. The results highlight the dynamic changes in phase composition and crystal size with different calcination temperatures, providing valuable insights into tailoring zinc oxide properties for various applications.

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1. INTRODUCTION

Semiconductors are a class of materials that possess electrical conductivity characteristics positioned between those of metals and nonmetals (Hossain et al., 2023). Unlike metals, which are excellent conductors of electricity, and nonmetals, which are typically insulators, semiconductors occupy a unique middle ground. They have the remarkable ability to facilitate the flow of electric current under certain conditions. This conductivity arises from the ability of semiconductors to manipulate the behavior of electrons within their atomic structures. The significance of semiconductors in electronics is profound. Their ability to be modified or tailored through different treatments, like doping with specific elements or altering their crystal structures, allows engineers and scientists to finely tune their electrical properties (Feng et al., 2019; Kousseff et al., 2021). This level of control over conductivity properties is a cornerstone of modern electronics.

The increasing interest in researching ZnO is driven by the continuous advancements in processing techniques that have made it possible to produce both undoped and doped ZnO of exceptionally high quality, suitable for a wide range of electronic material applications (Seid & Dejene,

2020; Ayoub et al., 2022). ZnO is classified as a semiconductor material, which means it possesses unique electrical properties. However, its utility goes beyond just its semiconducting characteristics. Materials properties and performance can be significantly influenced by factors such as its morphology (structure), shape, and particle size (Sulciute et al., 2021; Ayoub et al., 2022; Clarke & Ghandi, 2023). Researchers have found that by controlling these aspects, they can fine-tune ZnO's behavior for various applications. ZnO is particularly appealing for research due to its stable chemical structure and relatively straightforward synthesis methods (Verma et al., 2021; Raha & Ahmaruzzaman, 2022). This stability is crucial for ensuring the reliability and longevity of electronic devices and materials that incorporate ZnO. Additionally, ZnO has high electron mobility, which means that electrons can move effectively through the material, making it valuable for electronic and optoelectronic applications (Das & Basak, 2021; Sharma et al., 2022). Due to its unique combination of properties, ZnO has found applications in various fields. These include photocatalysis (using light to drive chemical reactions), gas sensing (detecting specific gases in the environment), light-emitting devices (such as LEDs), and photovoltaic cells (for converting sunlight into electricity) (Seid & Dejene, 2020). Researchers are continually exploring new ways to leverage ZnO's properties in these and other areas, making it a versatile and sought-after material for scientific investigation.

Materials containing impurities have the potential to exert profound effects on their physical attributes, including electrical, thermal, and optical properties (McCluskey & Haller, 2018). An essential aspect of identifying and understanding the impact of impurities in a material involves an analysis of its inherent structure. By scrutinizing the arrangement and composition of the material, we can gain insights into the presence and influence of impurities. Consequently, it is necessary to conduct research on the synthesis of ZnO and explore how varying calcination temperatures during the synthesis of ZnO can influence its crystal structure. These studies are pivotal as they have the potential to elucidate how this parameter can lead to precise control over the properties and performance of ZnO. Such understanding may open up a wide array of applications for ZnO in fields ranging from electronics to optics, making it a valuable subject of ongoing scientific inquiry.

ZnO can be synthesized using various techniques, each offering unique advantages and tailoring the material's physical characteristics for specific applications. These synthesis methods include biosynthesis (Septiana et al., 2022), sol-gel (Chung et al., 2015), hydrothermal (Nakate et al., 2022), precipitation (Rajan et al., 2023), etc. Among these techniques, the sol-gel method stands out as a commonly chosen approach, particularly for producing nano-sized ZnO materials. This method is favored for several reasons. First, it is known for its simplicity, user-friendliness, and speed, making it accessible and efficient for researchers and industry practitioners alike. Additionally, the sol-gel method excels in producing ZnO materials with minimal impurities or additional compounds, ensuring a high level of material purity (Bokov et al., 2021). Furthermore, the sol-gel method encompasses various parameters that can influence the crystal structure of the resulting ZnO material, with calcination temperature being one of the crucial factors in this regard (Bokov et al., 2021; Khan & Dar, 2021). By carefully adjusting the synthesis method and its parameters, researchers can tailor the properties of ZnO materials to meet the specific requirements of diverse scientific and industrial applications. This flexibility in synthesis approaches provides a valuable toolkit for customizing ZnO materials according to desired characteristics, which is essential in materials science and engineering.

Calcination is a critical process in materials science that plays a crucial role in enhancing the purity of materials by effectively removing impurities. The temperature at which calcination takes place is a pivotal factor that influences several key aspects of the resulting material, including its purity, size, and structure (Nurlaila et al., 2017; Elsandika et al., 2019). For instance, in the case of ZnO crystals, they can begin forming at relatively low temperatures, typically below 400°C (Borysiewicz, 2019). Any adjustments made to the calcination temperature can have a profound impact on the crystal structure and overall quality of the material (Nurlaila et al., 2017; Elsandika et al., 2019). This underscores the importance of precise control and optimization of the calcination temperature in materials science. By doing so, researchers can tailor the properties and characteristics of the final material to align with specific applications and achieve desired outcomes. In essence, the calcination temperature serves as a

powerful tool for fine-tuning material properties to meet the unique requirements of various scientific and industrial applications.

The information presented underscores the significance of conducting research that centers on the synthesis of ZnO using the sol-gel method, with a specific focus on investigating the impact of calcination temperature on the crystal structure. The sol-gel technique facilitates the synthesis of ZnO powder within a temperature range of 300°C to 700°C. Subsequently, the synthesized ZnO powder undergoes X-ray diffraction (XRD) analysis to elucidate its crystal structure response to temperature variations. These findings, in turn, provide valuable insights into how the material behaves under different temperature conditions, offering a deeper understanding of its characteristics.

2. METHOD

Zinc oxide (ZnO) powders were synthesized using the sol-gel method, a precise procedure that relied on zinc powder, hydrochloric acid (HCl), and sodium hydroxide (NaOH) as crucial precursors. The process commenced with the dissolution of zinc powder in an HCl solution, leading to the creation of an acid solution known as ZnCl₂. This resulting ZnCl₂ solution was then carefully titrated with a NaOH solution until it reached a neutral pH level of 7. This critical step initiated the formation of a gel-like substance, which subsequently underwent a series of sequential treatments, including leaching, drying, and calcination, carried out over a duration of 2 hours at temperatures ranging from 300°C to 700°C. X-ray diffraction (XRD) patterns of the synthesized powders were collected using a Cu-K_α Bruker Advance D8 X-ray diffractometer covering an angle range of 10° to 120° with a step size of 0.02°. These patterns were subjected to comprehensive qualitative and quantitative analysis employing specialized software tools, namely *Match!3*, *Rietica*, and *MAUD*. This analysis was performed to identify phases, determine phase compositions, and assess crystallite sizes with precision. Notably, adjustments for instrument peak broadening were meticulously applied to ensure accurate estimations of phase composition and crystallite size.

3. RESULTS AND DISCUSSION

Figure 1 provides the X-ray diffraction patterns of all samples. These patterns offer insights into the structural changes that occur in the materials both before and after undergoing calcination at temperatures ranging from 300°C to 700°C. The qualitative analysis of these patterns was conducted using *Match!3* software, which provides a comprehensive elucidation of the material's phase identification. Initially, the X-ray diffraction pattern of the powder before calcination reveals the presence of a single compound identified as simonkolleite (zinc chloride hydroxide monohydrate, Zn₅(OH)₈Cl₂·H₂O). However, as the calcination temperature increases, distinct alterations in the diffraction pattern become evident. At 300°C and 400°C, the intensity of the simonkolleite phase peaks begins to decrease, coinciding with the emergence of recognizable peaks corresponding to the wurtzite (ZnO) phase. Upon reaching a calcination temperature of 500°C, a significant change occurs within the material. The simonkolleite phase is no longer discernible in the diffraction pattern, but new phases emerge, most notably ZnCl₂ (zinc chloride) and NaCl (halite). These phases become increasingly prominent as the temperature rises to 700°C, with the diffraction pattern of the ZnO phase continuing to intensify. The appearance of the zinc chloride peak within the temperature range of 500°C to 700°C can be attributed to sub-optimal holding times during the calcination process. This inadequacy hinders the complete decomposition of compounds, allowing zinc chloride to persist within the material. Simultaneously, the presence of the NaCl phase at these higher temperatures suggests that the washing process was also less than optimal, resulting in residual salt compounds in the synthesized material. The formation of these phases can be explained in more detail in the next passage.

The synthesis process initiated with the reaction of Zn with HCl, resulting in the formation of ZnCl₂ (Equation (1)). Subsequently, the ZnCl₂ solution is titrated with NaOH, leading to the formation of Zn₅(OH)₈Cl₂ and NaCl (Equation (2)). Following titration, the next step involved a washing procedure designed to remove any remaining NaCl content from the sample. Notwithstanding the washing procedure, the research has identified the enduring presence of NaCl content that remains incompletely

eradicated from the sample. H₂O was used as the solvent for washing due to the polar nature of both NaCl and H₂O, allowing NaCl to be dissolved into Na⁺ and Cl⁻ ions in this solvent environment. In the subsequent stage, the sample underwent a drying process with the objective of removing any residual water or washing solution that might have still adhered to the sample after the completion of the washing process. Furthermore, a crucial step involved subjecting the sample to calcination at various temperatures. This procedure aims to facilitate the decomposition of the compound Zn₅(OH)₈Cl₂ present in the sample while simultaneously removing any impurity compounds that may still be present. The ultimate goal of this calcination process is to obtain ZnO compounds, as outlined in Equation (3). This calcination process holds significant importance as it substantially contributes to the purification of the sample and is a prerequisite for conducting further structural analysis and applications within the context of this research.

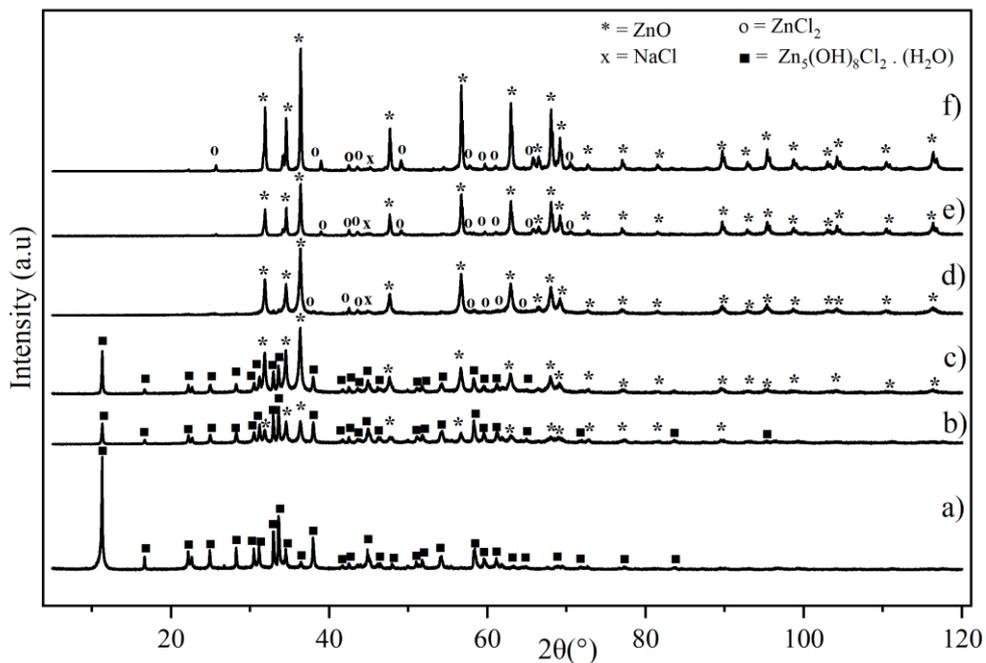
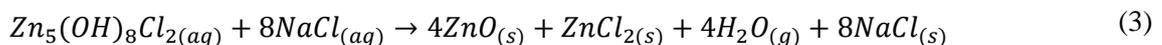
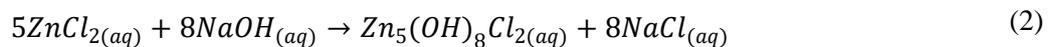
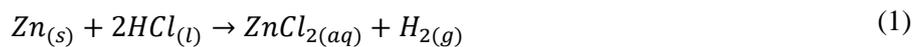


Figure 1. XRD patterns (CuK_α) of ZnO (a) before calcination and after calcination with temperatures (b) 300°C, (c) 400°C, (d) 500°C, (e) 600°C, and (f) 700°C.



During the calcination process, the variation in temperature becomes a critical factor that exerts a substantial influence on the final characteristics of the sample being processed. Specifically, elevated temperatures possess the capacity to initiate more efficient decomposition reactions of specific compounds that are present in the sample, such as Zn₅(OH)₈Cl₂. As the temperature rises, several important effects as follows come into play. Higher temperatures provide the energy needed for a more efficient and thorough decomposition of the target compound (in this case, Zn₅(OH)₈Cl₂) (Taglieri et al., 2023). This implies that at higher temperatures, the compound is more prone to decompose into its constituent elements or simpler compounds. Increasing the temperature also enhances the likelihood of eliminating impurity compounds or undesired byproducts that may be present in the sample. These impurities can include any substances that are not part of the desired material composition. Given these temperature-dependent effects, maintaining precise control over the calcination temperature is of utmost

importance. It allows researchers to fine-tune the decomposition and purification processes, ensuring that they achieve the intended results. The precise control of calcination temperature is crucial for obtaining the desired material properties and characteristics. Whether the goal is to generate a purer form of the material, eliminate impurities, or induce specific structural changes, the variation in temperature during calcination plays a pivotal role in achieving desired outcomes. Consequently, this control over temperature constitutes a critical aspect of the research framework, enabling the tailoring of the sample's properties to effectively meet the objectives of the study.

During the calcination process, where varying temperatures were employed, significant changes in the phases present within the sample were observed. These changes were evident through shifts in the peaks detected in the X-ray diffraction pattern, indicating alterations in the phase composition and structure. At lower temperatures (300°C and 400°C), the peaks corresponding to the simonkolleite phase decreased in intensity, while peaks indicative of the wurtzite phase began to emerge. This suggests that the simonkolleite phase was starting to decompose, giving way to the formation of wurtzite. At the calcination temperature of 500°C, the simonkolleite phase became undetectable in the diffraction pattern, indicating that it had completely been decomposed. In its place, other phases such as zinc chloride and NaCl became noticeable. This suggests that the calcination process at 500°C led to the elimination of simonkolleite and the emergence of these new phases. During the temperature intervals of 500°C and 700°C, the peaks associated with wurtzite became more pronounced. This intensification may hint at the persistence of undissolved NaCl from the gel washing process, which can affect the purity of the final wurtzite. The emergence of the zinc chloride peak was attributed to insufficient holding time during the calcination process. Incomplete decomposition of compounds, such as $Zn_5(OH)_8Cl_2$, can occur when the calcination duration is inadequate, resulting in the presence of zinc chloride in the final material (Moezzi et al., 2016). In summary, the variations in calcination temperature led to phase changes within the sample, affecting the presence and intensity of specific phases like simonkolleite, wurtzite, zinc chloride, and NaCl. These changes indicate the complex chemical reactions and transformations taking place during the calcination process, and they highlight the importance of precise temperature control and sufficient holding times to achieve the desired material composition and structure.

Initially, before any calcination process, the material is entirely in the "simonkolleite" phase. As the calcination temperature increases, changes in the material's composition are observed. With a gradual increase in calcination temperature, the presence of simonkolleite starts to decrease. For instance, at 300°C, it has diminished to 90.10%, indicating that a phase transformation has begun. As the calcination temperature raises further, typically in the range from 500°C to 700°C, the simonkolleite phase disappears completely, reaching 0%. This means that the material has completely transformed into a different phase or composition. Simultaneously, as the calcination temperature reaches 300°C, a new phase identified as wurtzite, starts to emerge, constituting 9.90% of the material's composition at this temperature. The presence of the wurtzite phase continues to increase as the calcination temperature is further elevated. It suggests that this new phase becomes more dominant with higher calcination temperatures.

The intriguing phase transition is initiated by subjecting the material to high temperatures during the calcination process. The key driver behind this transition is the excess release of OH^- ions. The excess OH^- ions play a crucial role in accelerating the reaction kinetics for the formation of zinc oxide (ZnO). This excess of ions facilitates the chemical reactions necessary for the conversion of the initial simonkolleite compound into ZnO. This transformation is attributed to the decomposition of simonkolleite at high calcination temperatures, leading to the production of ZnO compounds (Taglieri et al., 2023). The calcination process serves a dual purpose. Firstly, it acts as an effective means to eliminate impurities and extraneous phases from the material. This purification process results in obtaining purer compounds. Secondly, calcination also plays a pivotal role in transforming the crystal phases within the material. This phase transformation fosters homogeneity within the material (Lhimr et al., 2021). It is important to note that at calcination temperatures ranging from 500°C to 700°C, the synthesized material is composed of three distinct phases: zinc oxide, halite, and $ZnCl_2$. This composition reflects the dynamic interplay between temperature, chemistry, and phase changes that

govern the behavior of materials during calcination. The coexistence of these phases at high temperatures demonstrates the intricate and multifaceted nature of materials as they undergo complex transformations.

Table 1 Phase composition and lattice parameter analysis of the ZnO powder samples using *Rietica* software with the Rietveld method

| Sample | Phase | Phase Composition (wt%) | Lattice Parameter (Å) | | |
|--------------------|---------------|-------------------------|-----------------------|-----------|-----------|
| | | | <i>a</i> | <i>b</i> | <i>c</i> |
| Before Calcination | Simonkolleite | 100 (0) | 6.32 (4) | 6.32 (4) | 23.67 (2) |
| 300°C | Simonkolleite | 90.10 (2) | 6.34 (3) | 6.34 (3) | 23.63 (1) |
| | Wurtzite | 9.90 (3) | 3.25 (2) | 3.25 (2) | 5.21 (9) |
| 400°C | Simonkolleite | 56.50 (1) | 6.34 (1) | 6.34 (1) | 23.65 (3) |
| | Wurtzite | 43.50 (1) | 3.25 (5) | 3.25 (5) | 5.21 (7) |
| 500°C | Wurtzite | 44.53 (2) | 3.25 (1) | 3.25 (1) | 5.21 (1) |
| | Halite | 15.18 (2) | 5.73 (3) | 5.73 (3) | 5.73 (3) |
| | Zinc Chloride | 40.29 (2) | 6.28 (9) | 11.17 (8) | 12.42 (1) |
| 600°C | Wurtzite | 64.45 (3) | 3.24 (1) | 3.24 (1) | 5.19 (4) |
| | Halite | 6.99 (7) | 5.71 (0) | 5.71 (0) | 5.71 (0) |
| | Zinc Chloride | 28.56 (5) | 6.04 (1) | 14.99 (8) | 14.21 (3) |
| 700°C | Wurtzite | 81.42 (3) | 3.24 (4) | 3.24 (4) | 5.19 (7) |
| | Halite | 4.22 (1) | 5.71 (0) | 5.71 (0) | 5.71 (0) |
| | Zinc Chloride | 14.22 (3) | 6.54 (0) | 11.31 (0) | 12.33 (0) |

The X-ray diffraction patterns displayed in Figure 1 reveal structural changes in the ZnO material as a result of varying calcination temperatures. This analysis is crucial for understanding how the material's structure evolves under different conditions. The findings emphasize the importance of precise control and optimization of calcination parameters. The passage notes that alterations in the phase composition of ZnO are not the only factors at play. Changes in the structural properties of the material also significantly impact its characteristics. This can include factors like crystal size. To determine the crystal size of ZnO, quantitative analysis is employed using the MAUD software. This software is a tool for analyzing X-ray diffraction data to extract information about the crystal structure, including size. Table 2, which is derived from the quantitative analysis using MAUD software, provides essential insights into the crystal size of ZnO at various calcination temperatures. It reveals that higher calcination temperatures result in increasing of the crystal size. This expansion is attributed to the high thermal energy introduced at elevated temperatures. The high thermal energy, which is inherent at higher calcination temperatures, plays a pivotal role in facilitating the diffusion of constituent atoms within the material. This diffusion process accelerates crystal growth and orchestrates the precise arrangement of atoms within the crystal lattice. In essence, the increased thermal energy allows atoms to move more freely, leading to larger crystal sizes.

At elevated calcination temperatures, the crystals within the material tend to exhibit a pronounced fusion or merging with neighboring crystals. This fusion occurs because the presence of grain boundaries diminishes, resulting in a larger crystal size as the calcination temperature continues to increase (Pomeroy, 2021). In simpler terms, the individual crystals in the material tend to grow larger and fuse together. Grain boundaries are the interfaces between individual crystal grains within a material. These boundaries can influence various properties of the material. When calcination temperatures are high, the presence of grain boundaries decreases. As a result, the individual crystals have more freedom to grow and merge with adjacent crystals, contributing to an increase in crystal size. In materials science, thermodynamics pertains to the study of energy and stability, while kinetics focuses on the speed of reactions. The calcination process involves both aspects – thermodynamic stability and the kinetics of crystal growth. The choice of calcination temperature influences how these processes interact and, consequently, determines the material's characteristics. The passage underscores that calcination plays a pivotal role in determining several critical aspects of the material, including phase

composition, crystal size, and agglomeration (Musyarofah et al., 2017). These factors are all crucial for optimizing the material's properties for specific applications. The choice of calcination temperature can be used as a tool to tailor the material to meet specific requirements.

Table 2 The crystal size of ZnO powder calcined at 300°C–700°C using MAUD software

| Sample | Crystal Size (nm) |
|--------|-------------------|
| 300°C | 27.342 (3) |
| 400°C | 29.599 (0) |
| 500°C | 36.382(1) |
| 600°C | 60.925 (4) |
| 700°C | 110.61 (1) |

4. CONCLUSION

The calcination temperature a significant role in influencing the crystal structure of ZnO powder, specifically its phase composition and crystal size. Prior to the calcination process, the phase composition of ZnO crystals is characterized by the simonkolleite phase. However, when subjected to calcination at temperatures 300°C and 400°C, the resulting material exhibits a combination of two phases, specifically simonkolleite and zinc oxide. The higher calcination temperatures within the range of 500°C–700°C, the material exhibits a more complex composition, consisting of three phases: zinc oxide, halite, and ZnCl₂. Additionally, the calcination temperature exerts a notable influence on the crystal size of the ZnO powder, leading to an increase in crystal size with elevated calcination temperatures. Samples subjected to calcination at 300°C, 400°C, 500°C, 600°C, and 700°C display ZnO crystal sizes measuring 27.342 nm, 29.599 nm, 36.382 nm, 60.925 nm, and 110.61 nm, respectively.

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