زانکۆی سەلاحەدین - هەولێر
Salahaddin University-Erbil

# Green Synthesis of ZnO Nanoparticles Using Red Radish leaf Extract

Research Project
Submitted to the department of Physics in partial fulfillment of the
requirements for the degree of BSc. in Physics


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Supervisor Certificate

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# LIST OF ABBREVIATIONS

| Abbreviations | Definition |
|---|---|
| ZnO | Zinc oxide |
| NPs | Nanoparticles |
| UV-Vis | Ultraviolet-visible spectroscopy |
| XRD | X-ray diffraction |
| SEM | Scanning electron microscope |
| EDX | Energy dispersive X-ray |

# Summary

Nanotechnology is a rapidly developing field that involves the manipulation and utilization of materials at the nanoscale level. Nanoparticles (NPs), which are typically less than 100 nm in size, are a fundamental building block of nanotechnology. NPs can be synthesized using two different approaches, top-down and bottom-up, but the chemical and physical methods used for their production are often expensive and potentially hazardous to both the environment and the user. To address these issues, researchers have turned to biological ingredients such as plant extracts, bacteria, fungi, algae, and yeasts to synthesize NPs. Among these options, plant extracts are particularly promising due to their unique capacity for metal ion reduction within a relatively short period of time. However, plant extracts require a longer cultivation time compared to other microorganisms. In this study, the researchers utilized Red Radish leaf extract to produce zinc oxide (ZnO) NPs. This process is a one-pot, inexpensive, and green method, meaning it does not involve the use of toxic materials. The ZnO NPs were characterized using various techniques to examine their structure, size, morphology, chemical composition, and optical properties. The investigation revealed that Red Radish leaf extract is a suitable environment for producing nanosize ~27 nm, spherical, monodisperse, highly crystalline, and 1:1 Zn to O ratio ZnO NPs. The mechanism of formation of ZnO NPs from Red Radish leaf extract was also explained in detail. Overall, this study demonstrates the potential of using plant extracts as a safe and cost-effective method for producing NPs, which could have significant implications for the development of nanotechnology applications.

# CHAPTER ONE
# GENERAL INTRODUCTION

## 1.1 Introduction

The study of nanotechnology is not new; it has been around since the turn of the past century. Nobel Laureate Richard P. Feynman introduced the concept of "nanotechnology" in his 1959 speech "There's Plenty of Room at the Bottom" (Feynman, 1960), and since then, several breakthrough advancements have been achieved in the sector (Khan et al., 2019a). Expanding fields of study, nanoscience and nanotechnology comprise the study of materials, technologies, and systems whose atomic arrangement on the 1-100 nm scale confers distinctive features and functions. It is possible to apply nanotechnology to any area of study, including but not limited to chemistry, biology, physics, materials science, and engineering. Catalysis, biomedicines, pharmaceutics, healthcare, food technology, the textile industry, optics, optoelectronic devices, and many more industries can all benefit from its use (Barzinjy et al., 2020). Objects in the nanometer (nm) range, or less than 1 nm, can be considered nanoparticles because of the potential differences in properties between them and the bulk material. Copper, zinc, titanium, magnesium, gold, alginate, and silver are just some of the metals that are being utilized in the creation of these novel nanostructures at now. Nanoparticles are being used into a broad variety of common items, from cosmetics and clothing to medicinal treatments and industrial productions like solar and oxide fuel batteries for energy storage (Hasan, 2015). There is a wide variety of industries and consumer goods that benefit from zinc oxide nanoparticles (ZNPs), making them one of the most widely utilized nanomaterials. ZNPs have been found to selectively kill cancer cells, suggesting that they may be developed as a new type of anticancer drug (Roy et al., 2014). The mineral zincite in the earth's crust contains zinc oxide (ZnO), however the vast majority of ZnO used in industry is synthetically manufactured (Mirzaei and Darroudi, 2017)**.** Top-down and bottom-up strategies are the two most common methods used to get NPs ready. Creating NPs is a common practice, and it is done both from the top down and the bottom up. The mechanical processes that gradually break down large materials into nanosized constructs are crucial to the top-down techniques, which rely heavily on size reduction. On the other hand, molecular structures are built up from the atomic level up in bottom-up techniques. To this end, NPs are constructed from the ground up, with smaller molecules atop larger ones, by combining atoms, molecules, and even smaller structures. NPs are created when the nanostructure and building components have been formulated. Chemical and biological processes are employed as the bottom-up strategy to creating metallic NPs. **Figure 1** depicts the two primary methods for synthesizing metallic nanomaterials: the top-down method and the bottom-up method (Baig et al., 2021).

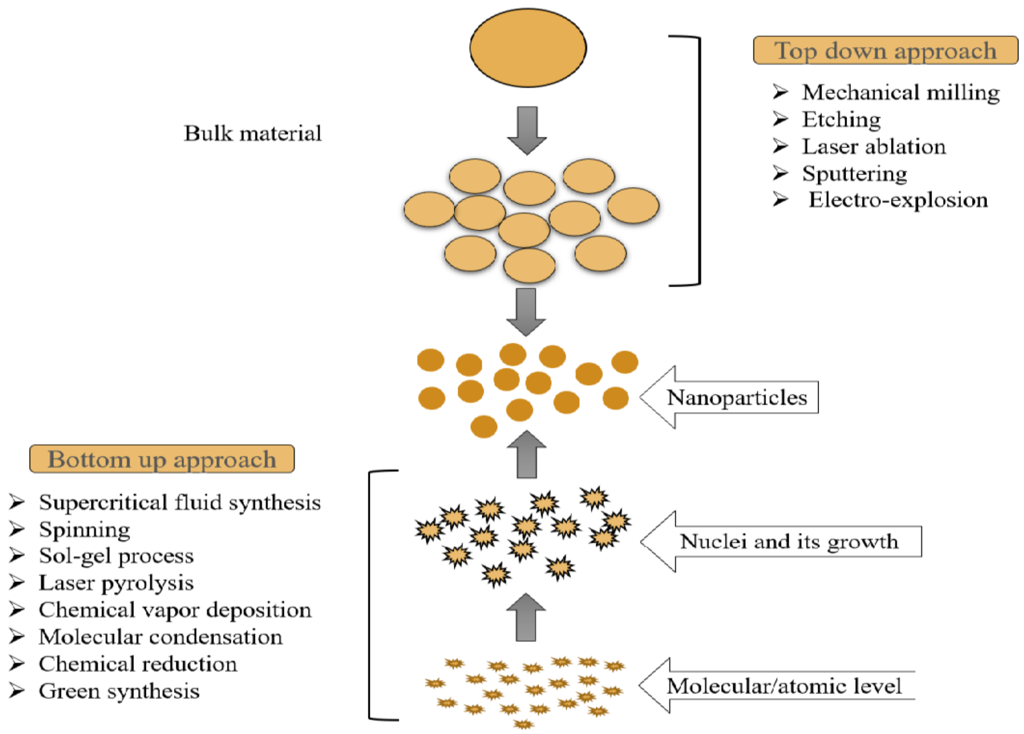


**Figure. 1:** Top-down and bottom-up approaches for synthesizing nanomaterials.

And **figure 2**: displays the grouping of dissimilar nanoparticle production methods (Barzinjy and Azeez, 2020).

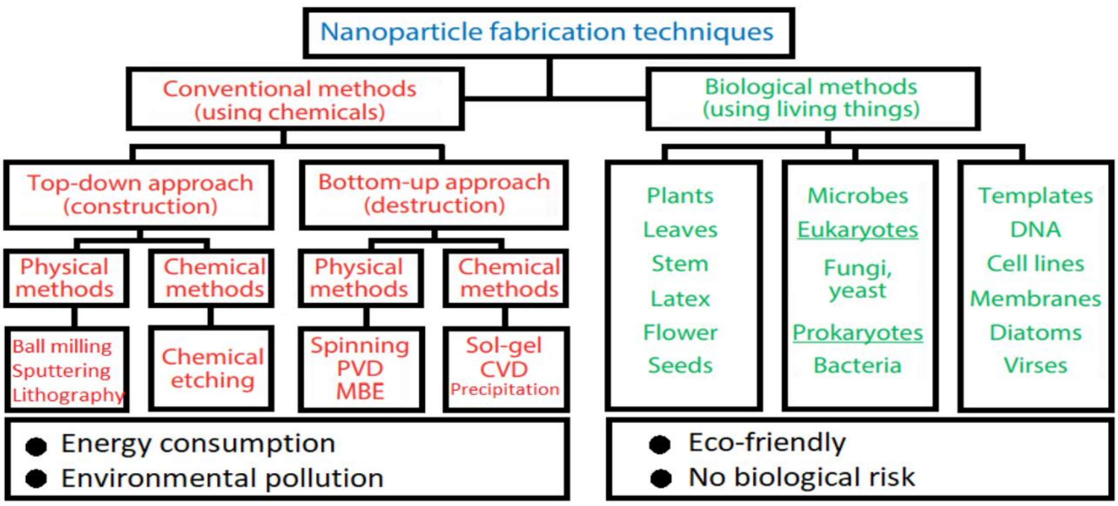


**Figure 2:** Classification of several methods for creating nanoparticles.

Further, we can affirm that nanotechnology has seen extensive use in the global food, biomedical, and agricultural industries (Rizwan et al., 2017). Contrary to popular belief, medical nanotechnology does not constitute "nanomedicine."

Although the term "nanomedicine" has been popular for a few years, it is more accurate to speak of "nanotechnology enabled medicine" when discussing specific medical specialties like diagnosis, treatment, or monitoring (Boisseau and Loubaton, 2011). It has been suggested that zinc oxide might be used as a practical means of preventing damage to the natural world. When it comes to cutting-edge research and development (Arora et al., 2014).

## 1.2 Zinc oxide nanoparticles

Zinc oxide (ZnO) nanoparticles attract a lot of attention due to their widespread use and nanotechnological properties and are produced by researchers in various ways (Gur et al., 2022). Zinc oxide is the most promising inorganic oxide, which is extensively being used for fabrication of devices and other applications (Dulta et al., 2022). ZnO nanoparticles have attracted worldwide attention in industry, medicine, and agriculture, because of their non-toxicity, antimicrobial activity, and low cost (Khan et al., 2019b). Zinc oxide nanoparticles (ZnO NPs), as one of the most important metal oxide nanoparticles, are popularly employed in various fields due to their peculiar physical and chemical properties (Jiang et al., 2018). Zinc oxide is of great economic and industrial interest due a wide range of properties that allows its application in many different areas, such as the rubber industry, biomedical field and metal surface treatment (Bandeira et al., 2020). Having these unique properties, ZnO NP is widely applied in various fields like solar cells, health, beauty products, sensors, agriculture, and photocatalytic (Batbual et al., 2023). Green synthesis of ZnO NPs has the advantage over other physicochemical processes because bio-inspired processes are relatively cheap, eco-friendly and renewable, compatible for pharmaceutical and biomedical applications, and can be easily scaled up for large-scale synthesis. Most importantly such processes can be carried out at relatively low pressure and temperature (Purkait et al., 2023).

# CHAPTER TWO

# MATERIALS AND METHOD

## 2.1 Materials

Zinc acetate dehydrate $C_4H_6O_4Zn.2H_2O$, molecular weight 219.50 g/mol, as well as NaOH Molecular weight 40 g/mol have been used in this study. The heating supply for synthesizing ZnO NPs was a Hot Plate Stirrer. PAN analytical X' Pert PRO (Cu Kα =1.5406 $A^o$) was used to perform X-ray diffraction (XRD) analyses. The scanning percentage was 1°/min in the 2θ assortment was from $20^o$ to $80^o$. A double-beam UV spectrophotometer (Super Aquarius spectrophotometer) was utilized to record UV–Vis spectral analysis to confirm the ZnO NPs production. Scanning electron microscopy (SEM) (Quanta 4500) was utilized to evaluate morphology and particle dispersion. EDX (Energy Dispersive X-ray analysis) in SEM was utilized to determine the elemental configuration of the produced nanostructures, while FT-IR analysis was done on a Perkin Elmer. FTIR spectrophotometer with a steadfastness of 4 $cm^{-1}$ was utilized for the examination of the available functional groups in the utilized plant extract and the NPs correspondingly.

## 2.2 Methods

### 2.2.1 Plant extract preparation

*Red radish* is one of the most important vegetable crops belongs to Brassicaceae family. It grows well worldwide; especially in both tropical and subtropical regions (Mahmoud et al., 2019). In this study, *Red Radish* leaf extract was used for the preparation of ZnO NPs. Fresh leaves of *Red Radish*, were washed several times by the drinking water, and formerly by means of deionized water to eliminate dirt subdivisions. About 30g of the utilized plant was mixed with 150 ml of DI water, and then the mixture put on a hot plate with a magnetic stirrer for 15 min at 35°C to extract the phytochemicals from the utilized plant. The plant extract was then filtered with filter paper twice to obtain a pure extract. A pH meter was used to monitor the addition of 2 moles of NaOH solution drop by drop to keep the pH of the combination at ~pH 8. The pH selection was not chosen at random since, according to the literature, smaller NPs can be formed at pH higher than 8 (Barzinjy and Hamadamen, 2022).

### 2.2.2 ZnO NPs Synthesis

*Red Radish* leaf extract was used to produce ZnO NPs utilizing the biological synthesis process. After preparing the plant extract as previously stated, 50 mL of the DI water and 2g of Zinc acetate dehydrate salt were put into a beaker and heated gradually on a hot plate within a magnetic stirrer, and then 50 ml of the *Red Radish* leaf extract was gradually added to the mixture. The temperature was maintaining at 60°C for 30 min, or until the combination turns greenish paste, the paste was blazed for about 2 hours at 500°C in a furnace. The residual was washed many times with ethanol distilled water. After that, the powder was dried at 100°C. Then, after obtaining ZnO NPs, the NPs were ready for the essential's characterizations. The producing process of ZnO NPs using *Red Radish* leaf extract and zinc acetate dehydrate is shown in **Figure 3**.

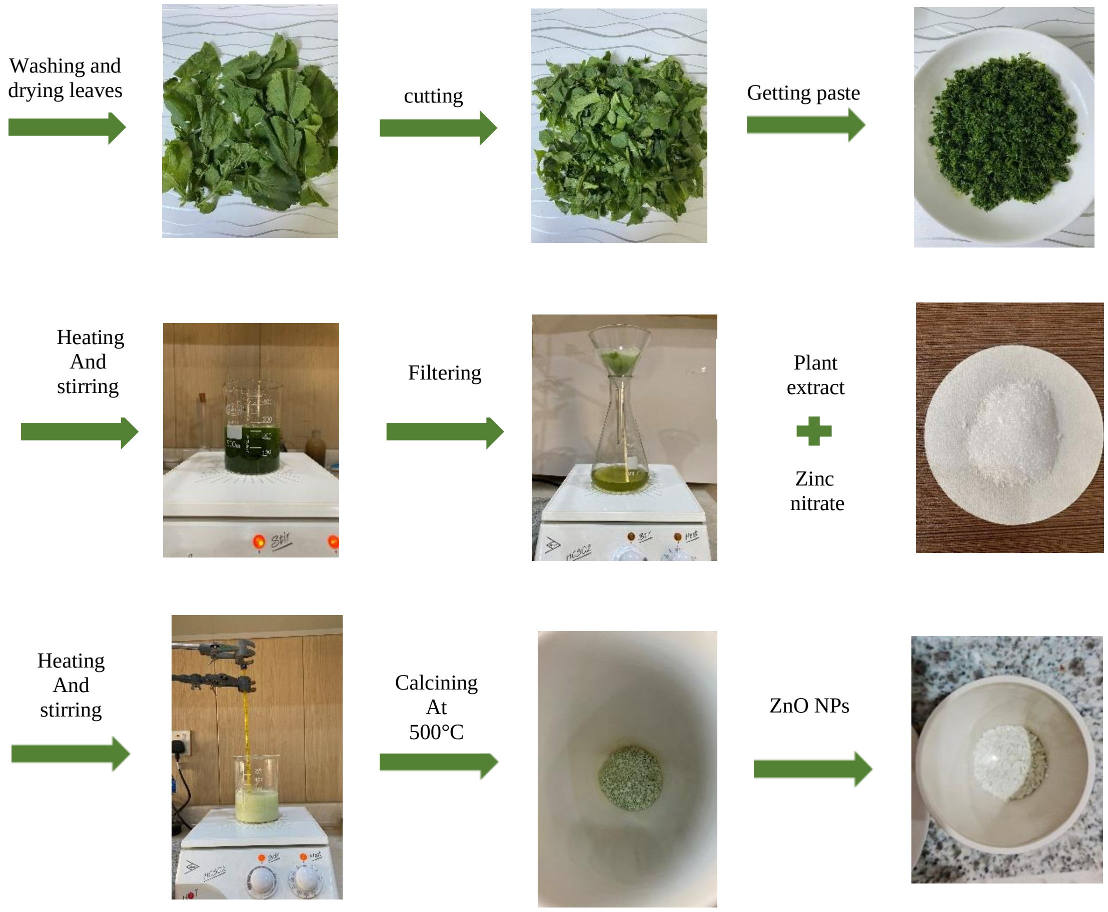


**Figure 3:** Graphical demonstration of ZnO NPs formation process utilizing red radish leaf extract and zinc acetate dehydrate.

# CHAPTER THREE

# RESULTS AND DISCUSSION

## 3.1 Characterization of Red Radish leaf extract

### 3.1.1 UV–Vis analysis

It sounds like you're referring to a process called green synthesis of nanoparticles, which involves using plant extracts to reduce metallic ions to metallic nanoparticles (NPs). Plant phytochemicals act as reducing agents, capping agents, and stabilizing agents all at once. This approach is eco-friendly and sustainable, and it offers several advantages over traditional methods of NP synthesis. UV-Vis spectroscopy is a commonly used technique for monitoring the progress of the reaction, as it can detect the surface plasmon resonance (SPR) of the newly formed NPs. SPR arises from the collective oscillation of conduction band electrons in response to electromagnetic waves, and it is a characteristic feature of metallic NPs. By observing the SPR peak in the UV-Vis spectrum, researchers can determine the size and morphology of the NPs and track the progress of the reaction. Overall, green synthesis of NPs using plant extracts and UV-Vis spectroscopy is a promising approach for producing NPs with controlled size and morphology while minimizing the environmental impact of traditional synthesis methods(Arora et al., 2014). Red Radish leaf extract possess one peak namely 296 nm as shown in **Figure (3.1)**.

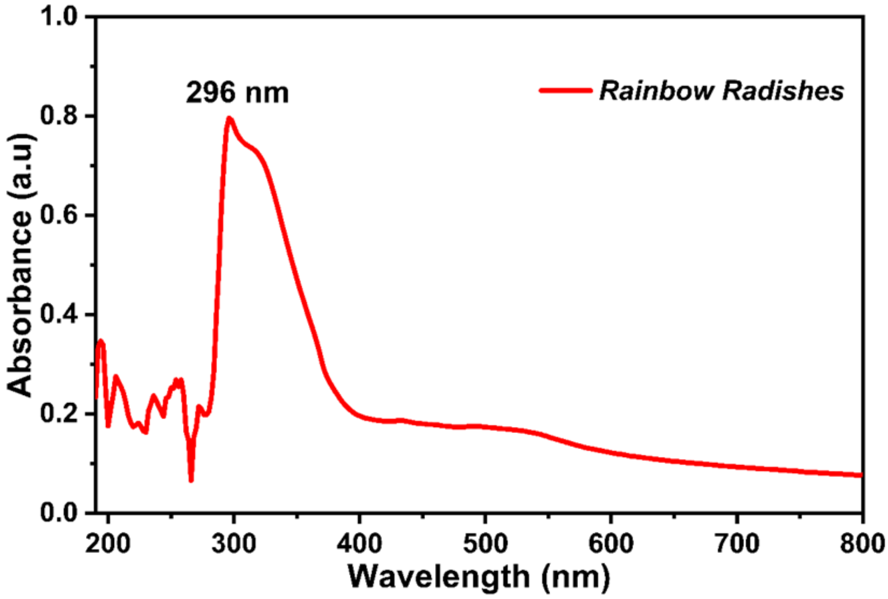


**Figure 3.1:** UV-Vis spectra of *Red radish* leaf extract.

## 3.2 Mechanism of formation of ZnO NPs

It is true that many investigations in the field of nanoparticle (NP) synthesis are focused on developing alternative reducing and stabilizing agents that are eco-friendly and do not pose health hazards. Plant extracts have emerged as a promising alternative to chemical agents due to the presence of various phytochemicals that can serve as reducing, capping, and stabilizing agents during the synthesis of NPs. Typically, a solution of plant extract is mixed with a metallic salt, such as zinc salts, and then heated at an optimal temperature and for a suitable time to form metallic ions that can be reduced by the phytochemicals present in the extract. However, to obtain the highest yields of NPs, it is crucial to optimize various parameters such as pH, temperature, time, and extract concentration, as well as the type of salts used It is important to note that the choice of plant extract used for NP synthesis can also impact the size and stability of the resulting NPs(Arora et al., 2014). For example, in the case of dill, the available biomolecules in the extract may be destroyed at higher temperatures, leading to agglomeration of ZnO NPs and the formation of larger particles. Overall, the use of plant extracts for NP synthesis offers a promising avenue for developing eco-friendly and sustainable methods of nanoparticle production. However, careful optimization of various parameters and selection of appropriate plant extracts are essential to ensure optimal yields and particle sizes. The pH value of the solution plays a crucial role in determining the characteristics of ZnO NPs synthesized from plant extracts. As stated by Alias et al., maintaining an alkaline pH level of 8 or 9 during the biosynthesis process is critical to producing homogenous particles with excellent nanostructure and minimal aggregation. This is because acidic and neutral pH levels in the Zn(OH)2 colloidal solution during the biosynthetic process can lead to particle agglomeration. On the other hand, an alkaline pH above 8 provides an adequate concentration of OH ions, which facilitates the rapid formation of ZnO NPs. It is worth noting that the pH level is not the only parameter that affects the synthesis of ZnO NPs from plant extracts. Other factors, such as temperature, time, and the concentration of the plant extract and metallic salts used, should also be optimized to achieve the desired particle size, morphology, and stability. The intensity of surface plasmon resonance (SPR) can increase in alkaline conditions, such as those created by the ionization of the OH groups in biomolecules present in plant extracts. SPR is a phenomenon that occurs when light interacts with metal nanoparticles, causing electrons to oscillate at a particular frequency and leading to the absorption of light at specific wavelengths. The production of NPs through SPR can be observed visually as a change in color, but it is essential to use a UV-Vis spectrophotometer for more accurate verification. Different phytochemical compounds present in plant extracts can react differently with metal ions, resulting in NPs with varying sizes, shapes, and crystallinity in each extract. In the study mentioned, the color of the metal ion solution with Red Radish extract changed to pale brown, indicating the formation of nanoparticles. However, further characterization is necessary to determine the size and stability of the particles and to confirm their crystalline nature. Based on the information provided, it appears that the process of synthesizing ZnO nanoparticles from Red Radish leaf extract involves the formation of a complex compound between the Zn2+ ion and phytochemicals present in the extract, which decomposes during calcination to form ZnO nanoparticles. The hydroxyl groups present in the extract are able to participate in redox reactions, reducing the Zn2+ ion to zero-valent Zn0, which then recombines during the nucleation phase to form clusters of nanoparticles(Arora et al., 2014). It is important to note that this process is complex and may involve multiple steps and factors that influence the final outcome of the synthesis. Further research and experimentation would be needed to fully understand and optimize the process for the production of ZnO nanoparticles from Red radish leaf extract.

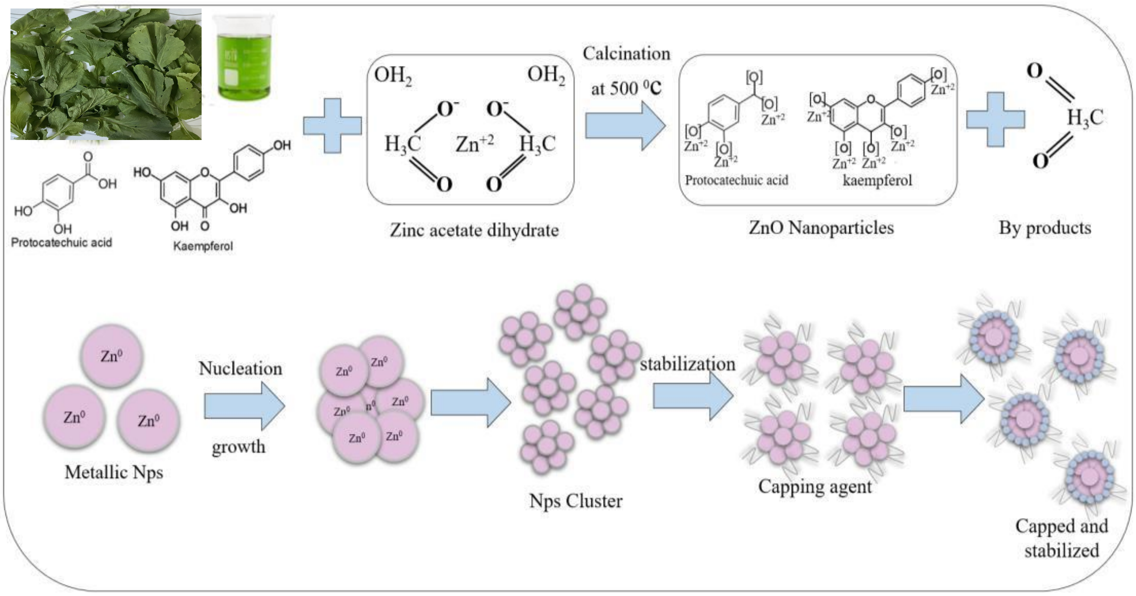


**Figure 3.2**: The plausible mechanism of synthesizing of ZnO NPs using *Red radish* leaf extract.

### 3.3 Characterization of ZnO NPs

Zinc oxide nanoparticles (ZnO NPs) were investigated in this study using various approaches to characterize their construction, morphology, and optical properties. Some of the techniques that may have been used to study these nanoparticles are:

### 3.3.1 UV–Vis analysis

UV-Vis spectroscopy: This technique can provide information about the optical properties of ZnO NPs, including their band gap and absorbance. UV-Vis spectroscopy measures the absorption of light in the UV and visible regions of the electromagnetic spectrum(Arora et al., 2014). The UV-Vis spectrum observed in this study shows a distinct peak at 321 nm, which is a characteristic absorption peak for ZnO nanoparticles. This peak is attributed to the surface plasmon resonance (SPR) of the nanoparticles. The sharper peak indicates the formation of mono-dispersed ZnO NPs, meaning that the nanoparticles are of uniform size and shape. The absorption peak maxima for ZnO NPs typically range between 300 and 380 nm, with variations depending on the size and shape of the particles. The observed peak at 321 nm falls within this range, and its value is lower than the 380 nm value for bulk ZnO. This blue-shift in excitonic absorption is due to the quantum confinement effect, which occurs when the size of the nanoparticles is smaller than the exciton Bohr radius. Overall, the UV-Vis spectrum analysis provides important information about the optical properties of ZnO nanoparticles and confirms the successful synthesis of mono-dispersed ZnO NPs with a distinct SPR peak at 321 nm.

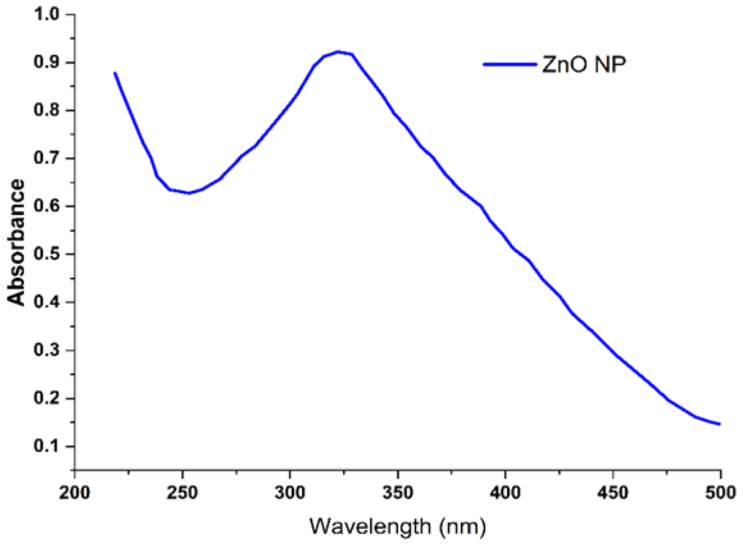


**Figure 3.3:** UV-Vis spectra of ZnO NPs prepared using *Red radish* leaf extract.

### 3.3.2 XRD analysis

X-ray diffraction (XRD) analysis: This technique can determine the crystal structure and phase of ZnO NPs. XRD can be used to calculate the average particle size of the nanoparticles by analyzing the diffraction peaks. The X-ray diffraction (XRD) investigation of biologically synthesized ZnO nanoparticles (NPs) shows the diffraction pattern of the crystal structure of the NPs(Arora et al., 2014). The diffraction pattern is characterized by peaks corresponding to different crystal planes, which can be used to identify the crystal structure and orientation of the NPs. In this study, the XRD pattern shows the presence of 11 different planes at specific angles. These peaks indicate the presence of a hexagonal wurtzite crystal structure of ZnO NPs, which is the most common crystal structure observed in ZnO NPs. The intensity and position of the diffraction peaks can be used to estimate the average crystallite size of the NPs using the Scherrer equation. The full width at half maximum (FWHM) of the diffraction peaks can also be used to estimate the crystal size and the degree of crystallinity of the NPs. XRD analysis is a powerful tool for characterizing the crystal structure and orientation of biologically synthesized ZnO NPs, providing important information for understanding their physical and chemical properties.

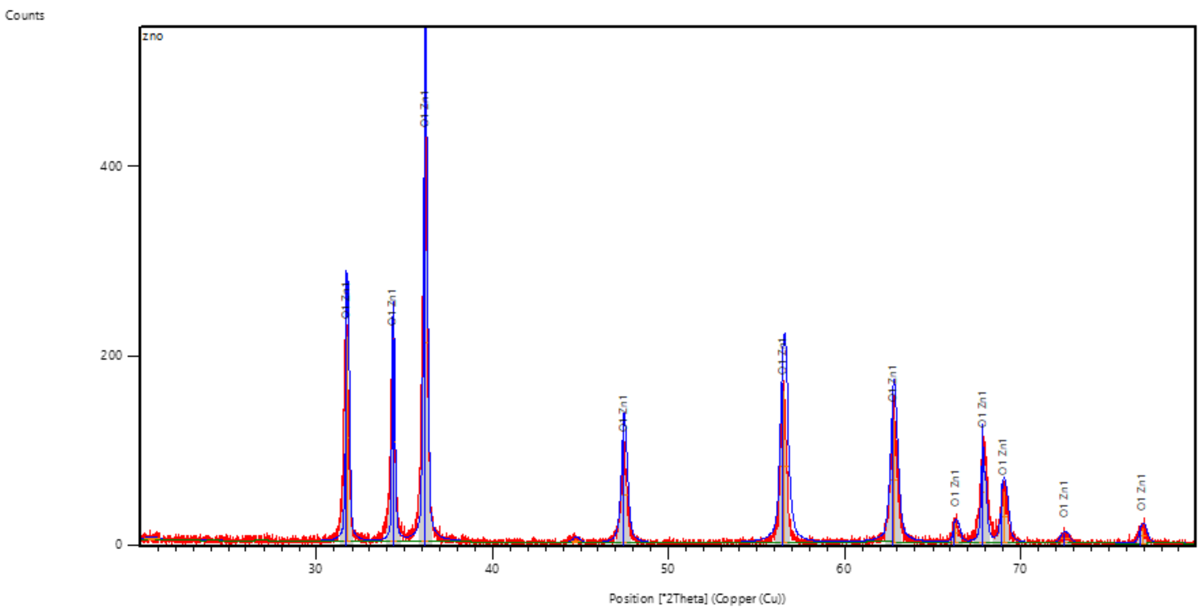


**Figure 3.4:** XRD patterns 0f Zno NPs synthesized using *red radish* leaf extract.

It seems like you are referring to a scientific research paper or report that describes the characterization of zinc oxide nanoparticles (ZnO NPs). The report describes the use of X-ray diffraction (XRD) analysis to determine the crystalline structure and size of the ZnO NPs. The diffraction peaks observed in the XRD pattern of the ZnO NPs matched with the standard JCPDS Card No. 89-0510, which is a database of XRD patterns for various materials. This indicates that the ZnO NPs have a crystalline structure similar to the one in the database. The report mentions the use of the Debye-Scherrer equation to calculate the crystalline size of the ZnO NPs. This equation relates the crystalline size (D) of a material to the X-ray wavelength (λ), the full-width at half maximum (FWHM) of the diffraction peak (β), the diffraction angle (θ), and a shape factor (k) which is typically assumed to be 0.9. The crystalline size of the ZnO NPs can be calculated using this equation, provided that the values of $\lambda$, $\beta$, and $\theta$ are known. It is worth noting that the characterization of ZnO NPs using XRD analysis is a common method in materials science research. By analyzing the XRD pattern, researchers can obtain information about the crystal structure, crystallite size, and degree of crystallinity of the material. This information can be used to understand the properties and potential applications of the material.

### 3.3.3 SEM

It appears that the study used SEM (Scanning Electron Microscopy) analysis to investigate the surface morphology of ZnO NPs that were synthesized using a green synthesis method(Arora et al., 2014). The results of the analysis are shown in a figure. The study observed that the majority of the ZnO NPs were nanometer-sized and spherical, with an average grain size of 33 nm. Interestingly, most of the NPs were of equal size, and there were no large particles present. The study also observed that the ZnO NPs were somewhat agglomerated, which is a common characteristic of nanoparticles synthesized using green methods. This is because green synthesized NPs have a higher surface area, and their attractive forces over time can cause aggregation. The study suggests that environmental factors can significantly impact the stability and agglomeration of NPs, which can lead to the formation of asymmetrical clusters during the nanoparticle production process. The synthesis of ZnO NPs is dependent on various growth factors, including the biomass content of the plant extract used, salt concentration, reaction time, temperature, and pH of the solution. Therefore, standardization of these growth factors is crucial in obtaining the desired size and shape of NPs, which is necessary for their optimal use and application. Overall, SEM analysis is a powerful tool in characterizing the surface morphology of NPs and understanding their physical properties.

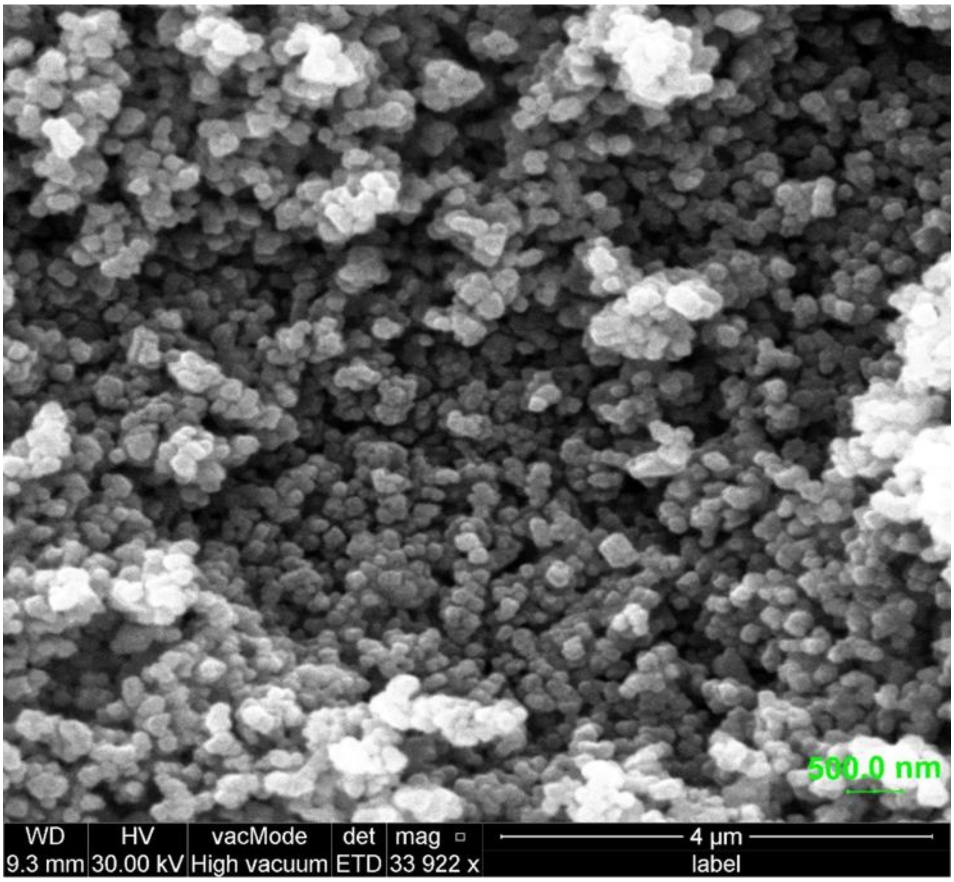


**Figure 3.5:** SEM analysis of ZnO NPs using *Red radish* leaf extract.

### 3.3.4 EDX

The paragraph describes the use of energy dispersive X-ray (EDX) analysis to examine the elemental composition of ZnO NPs (nanoparticles). The EDX analysis was used to provide additional information about the topographies (surface features) of the ZnO NPs(Arora et al., 2014). The results of the EDX analysis showed that the sample consisted mainly of zinc, oxygen, and gold. The gold was present because the sample had been coated with a thin layer of gold to enhance SEM (scanning electron microscopy) images. The EDX spectra showed strong peaks for zinc and oxygen, indicating that the sample was mainly ZnO. The weight percentage of zinc in the sample was 78.60%, while the weight percentage of oxygen was 21.40%. However, the atomic percentage of zinc was 47.30%, and the atomic percentage of oxygen was

52.70%, which gave a ratio of roughly 1:1 for Zn and O. This 1:1 ratio is desirable for many applications, such as water treatment, cosmetics, catalysis, and solar cells. In summary, the use of EDX analysis provided additional information about the elemental composition of the ZnO NPs sample, showing that it consisted mainly of zinc and oxygen with a desirable 1:1 ratio.

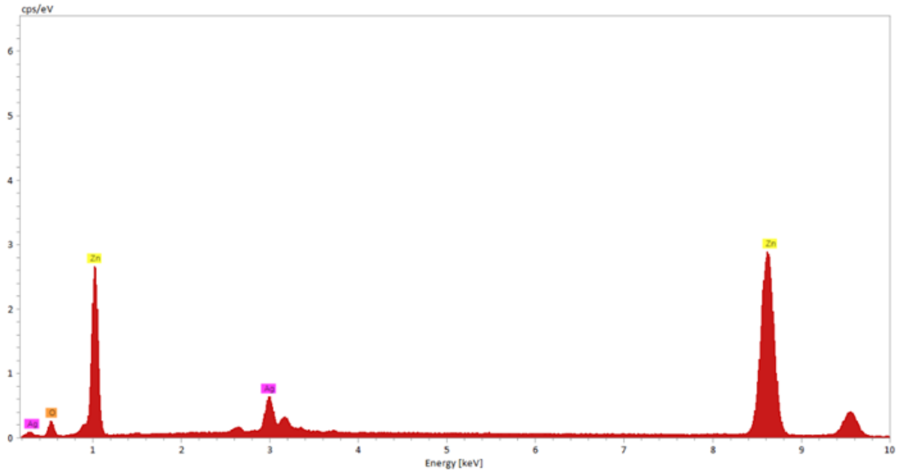


**Figure 3.6:** EDX Analysis of ZnO NPs using *red radish* leaf extraxt.

# CHAPTER FOUR
# CONCLUSION AND FUTURE WORK

## 4.1 Conclusion

The study describes a method to synthesize highly crystalline and monodisperse ZnO nanoparticles (NPs) using Red Radish leaf extract. This method is easy, quick, inexpensive and environmentally friendly compared to traditional chemical and physical methods. The resulting ZnO NPs are characterized by their monodispersity, high crystallinity, and direct band gap. The average particle size of the synthesized NPs is around 27 nm, which is within the range of many biomedical and cosmetic applications. The study also identified the phytochemicals responsible for the formation of ZnO NPs, namely protocatechuic acid and kaempferol. This finding suggests that Red Radish leaf extract could be used as a natural source for the synthesis of ZnO NPs. EDX analysis revealed that the atomic ratio of Zn to O in the synthesized NPs is 1:1, which is an important factor for many applications, especially in the biomedical and cosmetic industries. Overall, this study presents a promising approach for the synthesis of ZnO NPs with potential applications in various fields, including biomedicine and cosmetics. The use of Red Radish leaf extract as a green and sustainable source for the synthesis of NPs is also a noteworthy aspect of this study.

## 4.2 Future works

The study highlights the potential of Red Radish leaf extract as a green and sustainable source for the synthesis of ZnO NPs. This opens up possibilities for using other plant extracts with similar properties for synthesizing various metallic or metallic oxide NPs. Moreover, the biosynthesized ZnO NPs from Red Radish leaf extract can be further utilized for various applications, such as waste water treatment, sunblock, cosmetic, and solar cell applications. The unique properties of ZnO NPs, including their high surface area, biocompatibility, and photocatalytic activity, make them suitable for a wide range of applications. Therefore, future studies can explore the potential of using biosynthesized ZnO NPs for other applications, including drug delivery, antibacterial and antifungal agents, and environmental remediation. Overall, the study provides a promising approach for the synthesis of ZnO NPs and opens up possibilities for further research in this area.

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