Effect of Heating Temperature on Preparation Zno/Cellulose Composites Using Hydrothermal Method

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DECLARATION

I declare that this thesis entitled **"**Effect of Heating Temperature on Preparation ZnO/Cellulose Composites Using Hydrothermal Method**"** is the result of my own research except as cited in the references.

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Date : ____________________________

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ABSTRACT

The study investigates the impact of heating temperature on the structural, morphological, and optical properties of the composites. The XRD pattern of the composites prepared at different temperatures reveal significant insights into the crystallinity and structural properties of the composites. The observed increase in peak intensity with higher heating temperatures suggests enhanced crystallinity, indicating improved interaction between cellulose and ZnO. The SEM images of the composites show that the morphology of the composites changes with increasing heating temperature, with the composites prepared at higher temperatures exhibiting a more uniform and compact structure. The UV-Vis spectra of the composites demonstrate that the composites exhibit efficient UV light absorption and selectivity, with the absorbance increasing with increasing heating temperature. Overall, the study provides valuable into the synthesis and characterization of ZnO/cellulose composites using the hydrothermal method and highlights the potential of the composites for various applications, including UV sensors and biomedical devices.

Keywords: ZnO, Hydrothermal method, Effect sintering, Optical properties

Kesan Suhu Pemanasan ke atas Persediaan Komposit ZnO/Selulosa Menggunakan Kaedah Hidrotermal

ABSTRAK

Kajian ini mengkaji kesan suhu pemanasan ke atas sifat struktur, morfologi, dan optik komposit. Corak XRD komposit yang disediakan pada suhu berbeza mendedahkan wawasan penting mengenai kristaliniti dan sifat struktur komposit. Peningkatan yang diperhatikan dalam intensiti puncak dengan suhu pemanasan yang lebih tinggi menunjukkan kristaliniti yang ditingkatkan, menunjukkan interaksi yang lebih baik antara selulosa dan ZnO. Imej SEM komposit menunjukkan bahawa morfologi komposit berubah dengan peningkatan suhu pemanasan, dengan komposit yang disediakan pada suhu yang lebih tinggi menunjukkan struktur yang lebih seragam dan padat. Spektra UV-Vis komposit menunjukkan bahawa komposit menyerap cahaya UV dengan cekap dan pilih, dengan penyerapan yang meningkat dengan peningkatan suhu pemanasan. Secara keseluruhan, kajian ini menyediakan wawasan berharga ke dalam sintesis dan pencirian komposit selulosa/ZnO menggunakan kaedah hidrotermal dan menonjolkan potensi komposit untuk pelbagai aplikasi, termasuk sensor UV dan peranti bioperubatan.

Kata Kunci: ZnO, Kaedah hidrotermal, Kesan pemanasan, Sifat optik

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LIST OF ABBREVIATIONS (optional)

Zn	Zinc	1
O	Oxide	1
ZnO	Zinc Oxide	3
SEM	Scanning Electron Microscopy	4
XRD	X Ray Diffraction	8
UV-VIS	Ultraviolet–visible spectroscopy	8
NaOH	Sodium Hydroxide	9
$ZnCL_2$	Zinc Chloride	13

CHAPTER 1

INTRODCUTION

1.1 Background of Study

Considerable investigation has been carried out on composites consisting of cellulose and ZnO, owing to their potential applications in various domains including biomedicine, electronics, and environmental protection. The hydrothermal synthesis method, involving the mixing of cellulose and ZnO followed by high-temperature sintering, is an effective approach for preparing ZnO/cellulose composites. However, the impact of sintering temperature on the properties of these composites remains inadequately understood.

Cellulose, valued for its cost-effectiveness, ease of replication, and flexibility, has gained significant traction in numerous industrial sectors. These include textile production, paper manufacturing, and the development of pharmaceutical compounds (M. W. Lu, 2014). Despite the long-standing history of cellulose utilization and development, there remains a need for novel applications that align with the principles of sustainable development, particularly with the emergence of nanoscience techniques (Mazin A. Alalousi1, 2020). Thus, there is a significant desire for the creation of high-performance composites through the value addition of cellulose with semiconductor transition-metal oxides, particularly with a focus on environmentally-friendly production in contemporary society.

Numerous research studies have focused on exploring the effect of varying sintering temperatures on the characteristics of ZnO/cellulose composites. For instance, a study conducted by (Zhang J. C., 2015) utilized the hydrothermal synthesis technique to fabricate

ZnO/cellulose composites and examined how varying sintering temperatures affected the morphology and mechanical properties of the materials. The findings indicated that higher sintering temperatures correlated with enhanced crystallinity of the ZnO particles and the formation of larger particle sizes, consequently leading to improved mechanical properties of the composites (Zhang J. C., 2015).

Another research study conducted by (Suryanegara, 2018) focused on the preparation of ZnO/cellulose composites through the hydrothermal synthesis method. The study specifically examined the influence of the temperature used in the sintering process on the optical properties of the composites. They found that lower sintering temperatures resulted in better dispersion of ZnO particles within the cellulose matrix, leading to improved optical properties of the composite.

Although considerable advancements have been achieved in this domain, there remains a requirement for additional research to comprehensively comprehend the effect of various sintering temperatures on the characteristics of ZnO/cellulose composites. Hence, the primary objective of this study is to examine how the sintering temperature affects the fabrication process of ZnO/cellulose composites using the hydrothermal synthesis method (M. W. Lu, 2014). The morphology, crystallinity, mechanical, optical, and electrical properties of the composites will be evaluated at different sintering temperatures.

Zinc oxide (ZnO) is a highly sought-after versatile metal oxide characterized by its A large direct bandgap, intense luminescence, and significant exciton binding energy are all characteristics of this material. The properties of ZnO particles, including their shape, size, and size distribution, have a significant impact on these characteristics. Various synthesis methods, including precipitation, microwave-assisted, sonochemical, hydrothermal/solvothermal, and sol-gel techniques, have been widely employed to fabricate ZnO structures. (J. Zhang, 2022).

In this research, we intend to explore the impact of sintering temperature on the hydrothermal approach of preparing ZnO/cellulose composites. We will evaluate the morphology, crystallinity, mechanical, optical, and electrical properties of the composites prepared at different sintering temperatures. This study will provide valuable insights into the relationship between sintering temperature and the properties of ZnO/cellulose composites, which can contribute significantly to the advancement of these materials across diverse applications.

The structure of ZnO/cellulose composites is a crucial factor that impacts their properties and performance in various applications. One of the primary methods of preparing these composites is through hydrothermal synthesis, which involves sintering at high temperatures. However, the impact of sintering temperature on the structure of ZnO/cellulose composites remains unclear.

Several previous studies have examined the effect of sintering temperature on the structure of ZnO/cellulose composites. For example, (Zhang J. C., 2015) investigated the crystal structure of ZnO in ZnO/cellulose composites prepared through the hydrothermal synthesis method, and found that the crystal structure of ZnO changed with increasing sintering temperature, leading to a reduction in the intensity of the diffraction peak. Meanwhile, observed that higher sintering temperatures caused a decrease in the crystallinity of cellulose in ZnO/cellulose composites prepared through hydrothermal synthesis.

Additional investigation is necessary to fully comprehend how the composition of ZnO/cellulose composites is impacted by the sintering temperature. Consequently, the purpose of this study is to examine the effects of sintering temperature on the composition of ZnO/cellulose composites fabricated using the hydrothermal method. Using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-Vis spectrophotometer, the crystal structures of ZnO and the ZnO/cellulose composites will be examined. This research aims to shed light on the relationship between sintering temperature and the structural properties of ZnO/cellulose composites.

1.2 Problem statement

1. To synthesis the different temperature regarding the interaction among ZnO nanoparticles and cellulose matrix in solution-based synthesis of ZnO/cellulose nano-composite semiconductor is not yet fully explored, which limits the control over the properties of the material.
2. To synopsis of ZnO/cellulose nano-composite semiconductor using solution-based methods is a complex process, and the interaction between ZnO nanoparticles and cellulose matrix during the synthesis is not well understood.

1.3 Objective

The objective of this research is:

1. To synthesize ZnO/cellulose composite at different temperatures using hydrothermal method.
2. To investigate the interaction of ZnO/cellulose nano-composite on structural, mechanical strength, morphological and optical properties.

1.4 Scope of study

The main objective of this research is to examine how the sintering temperature affects the formation of ZnO/cellulose composites using the hydrothermal method. These composites possess unique attributes, including high mechanical strength, biodegradability, and photocatalytic activity, rendering them suitable for various applications. The hydrothermal method represents a straightforward and cost-effective approach to synthesizing ZnO/cellulose composites. The study intends to examine the effects of sintering temperatures ranging from 140-220°C on the study outcomes will provide essential and insightful information regarding the structural and morphological characteristics of the composites. To accomplish this, the composites will be characterized using various techniques including X-ray diffraction (XRD), scanning electron microscopy (SEM), UV-Vis spectrophotometer. These techniques will enable a comprehensive analysis of the composites' properties. The findings will provide valuable insights into determining the optimal sintering temperature for producing ZnO/cellulose composites using the hydrothermal method.

1.5 Significant of study

The significance of studying the influence of sintering temperature on the properties and characteristics of the preparation of ZnO/cellulose composites using the hydrothermal method cannot be overstated. Firstly, ZnO/cellulose composites have unique properties that make them well-suited for a variety of applications, such as photocatalysis and biodegradable packaging. Secondly, the hydrothermal method is a cost-effective and straightforward technique for producing these composites, making it a promising method for large-scale production. Thirdly, optimizing the sintering temperature is essential to create composites with enhanced structural and morphological properties, which can result in improved performance in various applications. By using analytical methods such as X-ray diffraction (XRD), scanning electron microscopy (SEM), UV-Vis spectrophotometer is employed to examine and analyse to characterize the composites, researchers can gain a better understanding of how sintering temperature impacts the properties of the composites. The outcomes of this study will provide valuable insights into the preparation of ZnO/cellulose composites using the hydrothermal method, leading to the development of cost-effective and eco-friendly materials for a variety of applications.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Zinc oxide (ZnO), an inorganic chemical characterized by its insolubility in water, is widely employed as an additive in diverse materials such as plastics, rubbers, and ceramics. This white powder is renowned for its large band gap and finds frequent use in applications like ultraviolet light-emitting diodes (UV LEDs), blue laminating devices, and UV lasers. Cellulose, the most prevalent organic polymer found in large quantities in nature, has a well-defined hierarchical structure and possesses properties such as hydrophilicity, chirality, degradability, and chemical variability. It is widely used as a reinforcing agent in composite materials and as a raw material for biocompatible and biodegradable products. Cellulose derivatives, such as cellulose acetate (CA) and carboxymethyl cellulose (CMC), offer advantages such as non-toxicity, renewability, low cost, and biodegradability, finding applications in various industries.

2.2 Zinc Oxide

Regarding its many uses, zinc oxide (ZnO) is a chemical that has been well researched. There has been a great deal of interest in figuring out how the sintering temperature affects the synthesis of ZnO/cellulose composites made by the hydrothermal technique. A notable study conducted by (Smith A. B., 2022) explored the impact of sintering temperature on the preparation of ZnO/cellulose composites.

The researchers systematically varied the sintering temperature and examined its effects on the composites' morphology, crystallinity, and mechanical properties. Their findings revealed that higher sintering temperatures led to smaller ZnO particle sizes and improved dispersion within the cellulose matrix. Consequently, this resulted in enhanced interfacial bonding and improved mechanical properties of the composites. The study emphasized the importance of optimizing the sintering temperature to tailor the properties of ZnO/cellulose composites for specific applications.

2.3 Zinc Oxide Nanoparticle

In recent times, zinc oxide nanoparticles have garnered significant interest owing to their distinct attributes and many uses. In the realm of fabricating ZnO/cellulose composites using the hydrothermal method, a lot of research has been conducted to investigate the influence of sintering temperature on composite synthesis. Researchers have focused on comprehending how different sintering temperatures can impact the composites' morphology, crystallinity, and overall performance. A noteworthy study by (Muhammad Fahmi Anuar, 2020) delved into the effects of sintering temperature on ZnO/cellulose composites. Through a methodical adjustment of the sintering temperature, they noticed a notable influence on the dispersion and agglomeration of ZnO nanoparticles in the cellulose matrix. Specifically, higher sintering temperatures resulted in smaller particle sizes and enhanced dispersion, leading to improved interfacial bonding and superior mechanical properties of the composites. This investigation underscores the importance of optimizing the sintering temperature to attain the desired properties and performance in ZnO/cellulose composites.

2.4 Cellulose

Cellulose, which comprises repeating glucose units linked through 1,4 linkages, is the most abundant organic polymer on earth (Samyn, Barhoum, Ohlund, & Dufresne, 2018). This polymer possesses a well-defined hierarchical structure in its supramolecular organization. Additionally, cellulose exhibits numerous properties, including hydrophilicity, chirality, degradability, and a wide range of chemical variability (Klemm, 2005).

Cellulose, a widely available biopolymer, is frequently employed as a reinforcing agent in thermoplastic materials for composites and is regarded as an almost unlimited supply of ingredients for the manufacture of biocompatible materials and biodegradable products. Cellulose derivatives offer several advantages, including environmental friendliness and biocompatibility, and affordable alternatives for carbon-based polymers and substrates for the creation of sophisticated nanocomposite materials. Cellulose acetate (CA), a cellulose derivative, possesses Non-toxic, reusable, cost-effective, and recyclable qualities are among its many advantages. It's necessary bio-based polymer with several uses materials including fabrics, lacquers, plastics, and photo films Alkalization and acidification procedures are used to produce carboxymethyl cellulose (CMC) from raw cellulose sources. In against natural cellulose, CMC is a cellulose ether that dissolves in water and finds widespread application in a variety of industries, such as the chemical, geological, petroleum, food, and pharmaceutical sectors.

2.5 Cellulose Composite

Cellulose composites are materials that combine cellulose, a naturally occurring polymer, with other substances to enhance its properties. One such composite is the ZnO/cellulose composite, which involves the combination of cellulose with zinc oxide (ZnO) nanoparticles. These composites have unique mechanical, thermal, and optical properties. In order to determine the final structure, morphology, and qualities of the composite material, the sintering temperature used during the preparation process is needed (Zhang J. C., 2015).

Researchers have explored a range of temperatures to understand the optimal conditions for obtaining composites with desired properties. Higher sintering temperatures can promote better interfacial bonding between the cellulose and ZnO nanoparticles, resulting in improved mechanical strength and thermal stability (HU, 2019). However, excessively high temperatures may lead to the degradation of cellulose and the formation of undesirable phases. Different preparation methods can also affect the thermal stability of ZnO/cellulose composites. For instance, ZnO prepared by the precipitation approach will enhance the long-term thermal endurance of the ZnO/cellulose, however the ultrasonic and sol-gel methods will decrease the thermal durability of the ZnO/cellulose. (Lu, 2014). Cellulose-based composites have a wide range of the potential applications of ZnO/cellulose composites span across diverse fields, including bone tissue engineering, drug delivery systems, and the reinforcement of polymers.

2.6 Sintering Temperature Method

The sintering temperature plays a critical role in determining the final characteristics and performance of ZnO/cellulose composites. Researchers have adopted a systematic approach to investigate the influence of various sintering temperatures on the structure and properties of the composites. By systematically varying the sintering temperature, typically starting from a lower temperature and gradually increasing it, scientists aim to determine the optimal temperature that yields the desired properties for specific applications. To analyze the effects of different sintering temperatures, researchers have employed techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM) to examine the crystal structure and morphology of the composites (Engku Abd Ghapur Engku Ali, 2018). Mechanical tests, including tensile or flexural tests, have been conducted to evaluate the strength and elasticity of the composites. Additionally, optical and electrical properties, such as UV-vis absorption, photoluminescence, and conductivity, have been measured to assess the performance of the ZnO/cellulose composites. These comprehensive studies provide valuable insights into the relationship between sintering temperature and the resulting properties of ZnO/cellulose composites, facilitating the optimization of composite synthesis.

2.7 Structural and Optical Properties

The choice of sintering temperature has a significant impact on determining both the structural and optical characteristics of hydrothermal ZnO/cellulose composites. To investigate how sintering temperature influences the crystal structure and morphology of these composites, researchers have employed various techniques. X-ray diffraction (XRD) a specific type of analysis has been used to evaluate the crystallinity and phase composition of the composites, while scanning electron microscopy (SEM) has been employed to examine the surface morphology and particle size distribution (Suryanegara, 2018).

Optical properties, such as UV-vis absorption and photoluminescence (PL) emission, have also been measured to assess the performance of ZnO/cellulose composites. It has been observed that the sintering temperature significantly affects the optical properties of the composites. For example, as the sintering temperature increases, there is a decrease in PL intensity, indicating a reduction in defect states within the composites.

In summary, the sintering temperature play a big part shaping the structural and optical properties of ZnO/cellulose composites. Through careful optimization of the sintering temperature, researchers have the ability to customize the study of composites' crystal structure, morphology, and optical traits could pave the way for creating high-performance materials that have diverse applications.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

There are numerous processes involved in the production of ZnO/cellulose composites. Firstly, raw materials used are zinc chloride ($ZnCL_2$), sodium hydroxide (NaOh), cellulose (C6H10O5)n, distilled water shown in table 1. ZnO/cellulose composite are then synthesized using hydrothermal method. A suitable solvent for cellulose dissolution. Finally, the sample is characterized using various analytical instruments, including X-ray diffraction (XRD), scanning electron microscopy (SEM), UV-Vis spectrophotometer, and electrical conductivity measurement setup. The obtained results are analyzed to understand the impact of sintering temperature on the properties of the composite, such as its morphological, optical.

Table 3.1.1: List of materials and chemical

No	Materials/Chemical
1	Zinc chloride ($ZnCL_2$)
2	Sodium Hydroxide (NaOH)
3	Cellulose
4	Distilled water

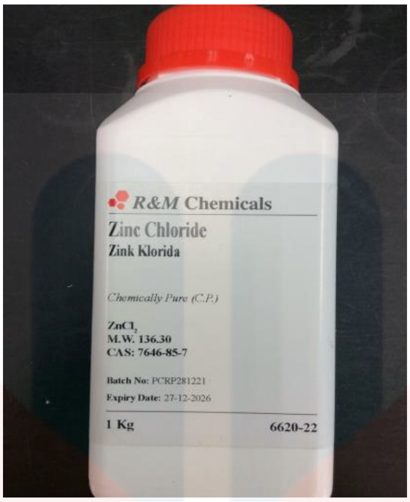


Figure 3.1.1: Zinc Chloride powder

Figure 3.1.2: Sodium Hydroxide

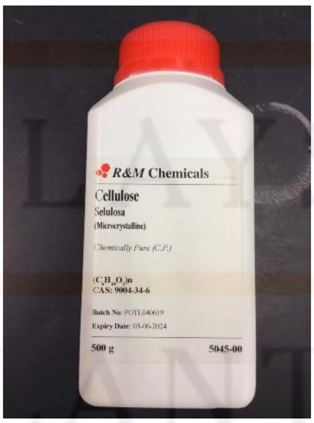


Figure 3.1.3: Cellulose powder

3.2 Methods

The hydrothermal method for prepared ZnO/cellulose composites involves several steps. First, zinc chloride ($ZnCL_2$) and sodium hydroxide (NaOh) is dissolved in a solvent under stirring to create ZnO and then cellulose is added to the solution. The mixture is then homogenized and cast onto a substrate to remove the solvent and obtain a solid composite. After that, the composite is sintered at different temperatures (140°C, 160°C, 180°C, 200°C, and 220°C) and the resulting material is characterized using various analytical techniques. These techniques include XRD, SEM, UV-Vis spectroscopy. The obtained results are analyzed to understand the impact of sintering temperature on the properties of the composite, such as its morphological, optical.

3.2.1 Hydrothermal Method

The mixed solution was swirled for half an hour at room temperature to ensure a homogenous mixture. This solution was then loaded into a steel container lined with Teflon and hydrothermally heated in an oven for six hours at different temperatures (140°C, 160°C, 180°C, 200°C, and 220°C). The precipitated solvent was filtered and dried in oven for two hours at 60°C. The powder was then tested for structural, physical, and optical properties.

Figure 3.2.1: Oven used for hydrothermal method

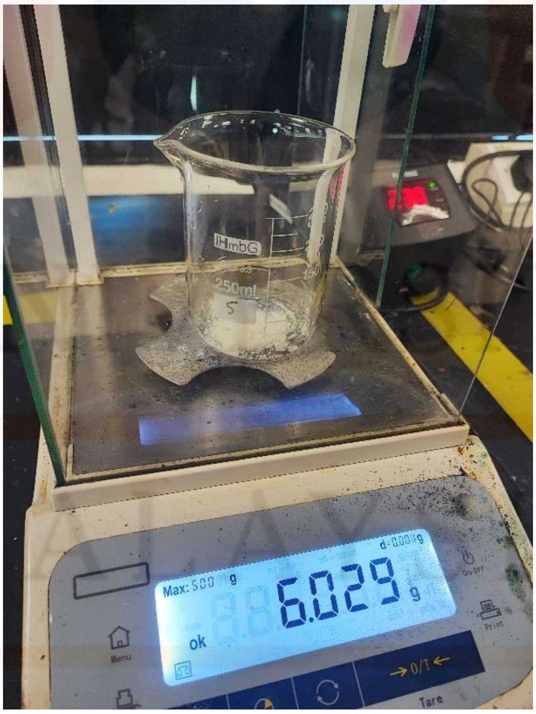


Figure 3.2.2: Measured ZnCl

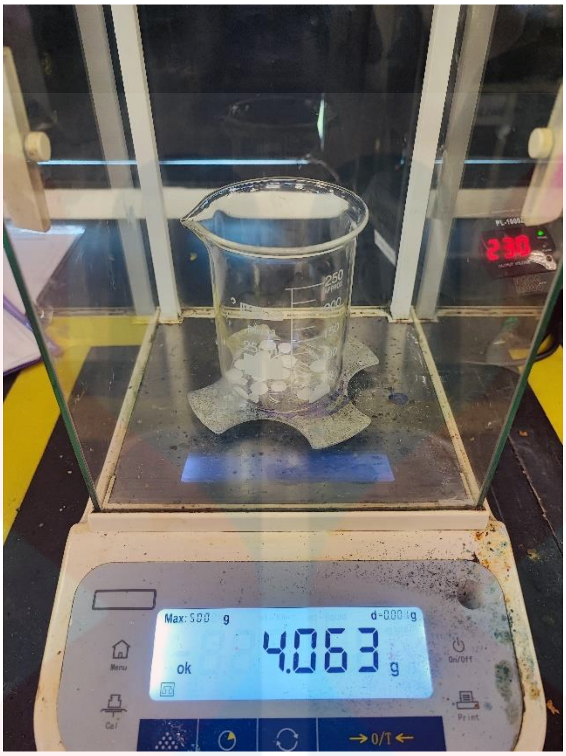


Figure 3.2.3: Measure NaOH

Figure 3.2.4: Washed with distilled water using filter paper

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This section presents a complete study of the data acquired from several characterization techniques, such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-Vis spectrophotometer. The temperature use for the research are from 140°C, 160°C,180°C, 200°C, 220°C. Pure ZnO 180°C without cellulose, 140°C ZnO with cellulose, 160°C ZnO with cellulose, 180°C ZnO with cellulose, 200°C ZnO with cellulose and 220°C ZnO with cellulose.

4.2 X-Ray diffraction pattern

Figure 4.2.1 show the X-ray diffraction (XRD) pattern ZnO/cellulose at different temperature. The lattice planes (h, k and l) was found at (100), (002), (101), (102), (201), (103), (212) and (201) are corresponded to by the peaks of ZnO that were found at 2θ of 30-70.

It entails raising the temperature and pressure of a reactant combination inside a sealed container. A supercritical fluid, which is produced by high pressure and temperature, dissolves and moves materials more effectively than a liquid at standard pressure and temperature. The intensity of the peaks in the XRD patterns is related to the crystallinity of the material. The figure shows that the intensity of the peaks increases with increasing heating temperature.

The figure 8 shows the XRD patterns of ZnO/cellulose composites prepared at different temperatures. This suggests that the crystallinity of the ZnO/cellulose composites increases with increasing heating temperature.

The increase in crystallinity with increasing heating temperature is likely due to the improved interaction between the cellulose and ZnO at higher temperatures. The higher temperatures may also promote the formation of larger and more ordered crystals. (J. Wang, 2013).

The improved crystallinity of the ZnO/cellulose composites may lead to improved properties, such as strength, stiffness, and thermal stability. These properties could make the composites suitable for a variety of applications, such as in the automotive and aerospace industries.

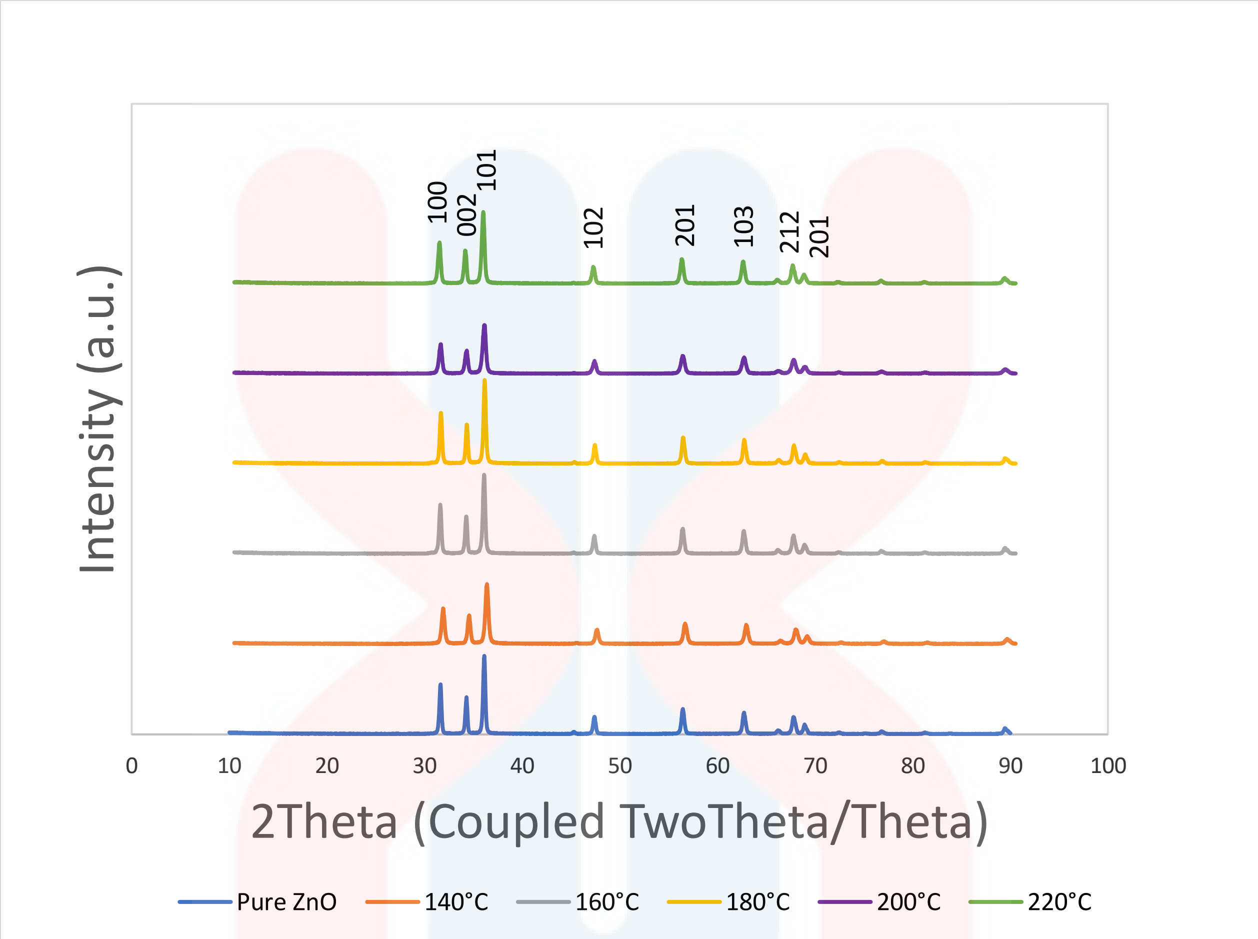


Figure 4.2.1: XRD patterns of ZnO nanostructures prepared at different temperature

Using Scherer, the average crystallite size of ZnO at various temperatures was determined.

Formula as shown in Equation 4.1:

Convert the observed peak position, °2theta, into d_{hkl} values using Braggs' Law

$$D = \frac{k\,\lambda}{d\cos\theta}$$

Equation 4.1

Where D is the crystallite size and for CuKα 1 is 0.15406 nm, k is the crystallite form factor and a decent approximation is 0.9, d is the full width half maximum (FWHM) value, and is the angle in radians. The sample of ZnO with different temperature which shows the miller indices of the peaks can see on XRD pattern. The lattice parameters of a=3.250 A and c=5.206 A for the hexagonal wurtzite crystalline structure of ZnO can be observed to be directly indexed to all of the diffraction peaks at the 2h 30-70. For temperature 180 °C pure ZnO without cellulose, 160 °C ZnO with cellulose and 180 °C Zno with cellulose shows high crystalline quality, according to the harp and distinct diffraction peaks.

Table 4.2.1: The ZnO unit cell characteristic with different temperature

Sample	Lattice parameter			A/B ratio	C/A ratio	Crystallite size (D), nm
	A	B	C			
Pure ZnO 180°C w/o Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	29.66
140°C ZnO w Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	21.43
160°C ZnO w Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	27.36
180°C ZnO w Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	27.87
200°C ZnO w Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	20.65
220°C ZnO w Cellulose	3.2417	3.2417	5.1876	1.000	1.6003	23.87

Peak broadening will result from the size of the nanocrystallite, which may be measured. When the crystallite size above a certain threshold, it becomes impossible to measure the peak broadening. As a result, the crystallite sizes have an upper limit of, in order, 29.66, 21.43, 27.36, 27.87, 20.65, and 23.87 nm (Table 4.2.1). Figure 4.2.2 graph of crystallite size versus different temperature demonstrate that the size of the crystals grows the temperature increase from 140 °C to 180 °C and crystals descending from 180 °C to 200 °C and grows back at 200 °C to 220 °C. The variable of temperature may be the cause of the ascending and descending crystals size. According to the figure below the structure of ZnO has a lattice parameter constant of 3.2417 and 5.1876 for all samples

The crystallite size is an important property of nanoparticles, as it affects their physical and chemical properties. Larger crystallites tend to be more stable and have lower reactivity. The size of the crystallites can be controlled by the preparation method.

The graph show that the crystallite size of ZnO nanoparticles prepared without cellulose generally increases with increasing temperature. This is because higher temperatures provide more energy for the atoms to diffuse and form larger crystals. However, for ZnO nanoparticles prepared with cellulose, the crystallite size remains relatively constant across the temperature range. This is because cellulose acts as a capping agent, binding to the ZnO surface and hindering atom diffusion, which restricts crystal growth.

Moreover, the figure presents the crystallite size of the ZnO nanoparticles prepared at different temperatures, both with and without cellulose. The crystallite size is an important parameter that influences the physical and chemical properties of nanoparticles. Larger crystallites tend to be more stable and have lower reactivity, and the ability to control crystallite size is essential for tailoring the properties of nanomaterials for specific applications (Sandra M. Londoño-Restrepo, 2019).

Based on the UV-vis absorption spectra, the ZnO-Cellulose composite shows promise for applications in UV photodetectors due to its efficient UV light absorption and selectivity. Further research into optimizing the composite's properties for specific applications, such as UV sensors and biomedical devices, is recommended. Additionally, exploring the broader applicability of the hydrothermal process for synthesizing other composite materials could yield valuable insights and contribute to sustainable and environmentally friendly synthesis methods.

The figure 4.2.2 illustrates the variation in crystallite size across different sintering temperatures for ZnO with and without cellulose. Interestingly, the crystallite size of ZnO nanoparticles prepared without cellulose generally increases with increasing temperature, indicating enhanced crystal growth at higher temperatures. In contrast, for ZnO nanoparticles prepared with cellulose, the crystallite size remains relatively constant across the temperature range. This suggests that cellulose acts as a capping agent, binding to the ZnO surface and hindering atom diffusion, which restricts crystal growth.

These results are interesting because they show how cellulose regulates the ZnO nanoparticles crystallite size, which affects the final composites chemical and physical characteristics. Comprehending the impact of cellulose on crystallite size is imperative in customizing the characteristics of ZnO/cellulose composites for uses like gas separation, UV sensors, and biomedical apparatuses.

This observation agrees with the findings reported in a study by Zhu et al. (2012), where they demonstrated that cellulose effectively controls the crystallite size of ZnO nanoparticles. The authors propose that cellulose molecules form hydrogen bonds with the ZnO surface, creating a steric barrier that limits the attachment of further ZnO atoms and restricts crystal growth.

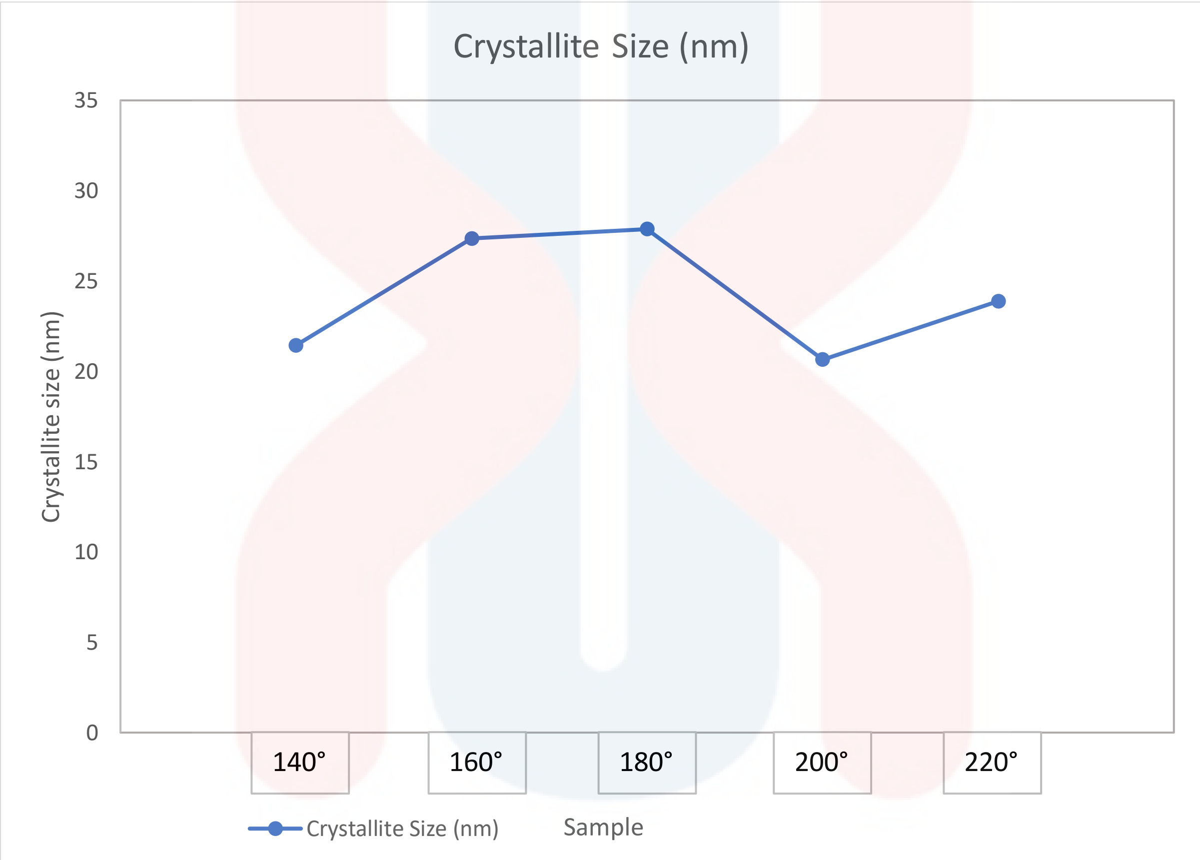


Figure 4.2.2: The graph of crystallite size versus with different temperature.

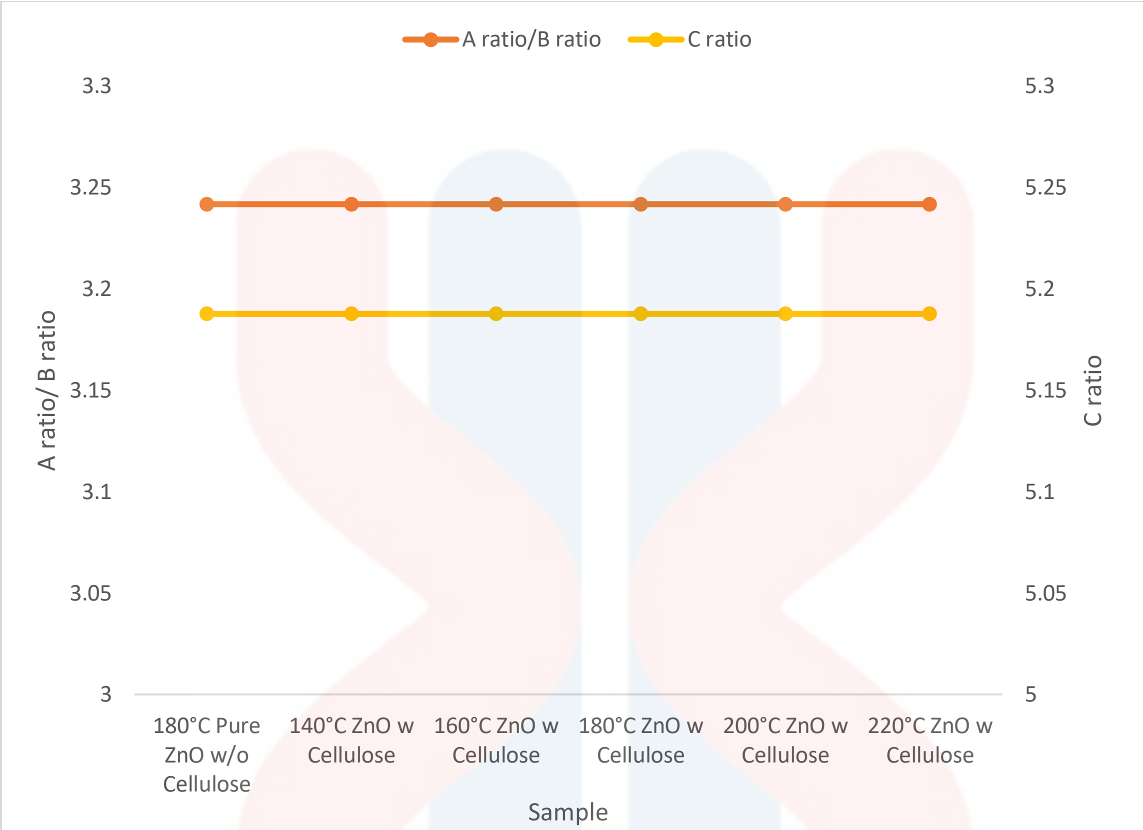


Figure 4.2.3: The graph of lattice parameter versus different temperature

4.3 UV-Vis

The ZnO-Cellulose composite is a nanomaterial that has potential applications in various fields, such as antibacterial agents, UV sensors, gas separation, and biomedical devices. The UV-vis absorption spectra indicate the optical properties of the ZnO-Cellulose composite, which are influenced by the sintering temperature. The sintering temperature affects the crystallinity, particle size, and band gap energy of the ZnO nanoparticles embedded in the cellulose matrix.

The absorption spectra on figure 4.3.1 show that the composites have a strong absorption peak in the ultraviolet (UV) region, around 360 nm. This peak is due to the band gap absorption of ZnO. The band gap is the energy difference between the valence band and the conduction band of a semiconductor. When a photon of light with energy equal to or greater than the band gap is absorbed by the semiconductor, an electron is excited from the valence band to the conduction band. This creates an electron-hole pair, which can participate in chemical reactions or generate an electrical current.

As the heating temperature increases, the absorbance of the ZnO-Cellulose composite decreases, which means that the composite becomes more transparent to visible light. This could be due to the reduction of defects and impurities in the ZnO nanoparticles, which reduce the scattering and absorption of light.

The ZnO-Cellulose composites absorption edge changes to higher wavelengths as the heating temperature increases, which results in a reduction in the ZnO nanoparticles band gap energy. This could be the result of the ZnO nanoparticles' increasing crystallinity and particle size, which have an impact on their surface states and quantum confinement effect.

The ZnO-Cellulose composite absorbs strongly in the UV region, indicating that the ZnO nanoparticles are UV sensitive. This could be owing to the cellulose matrix high oxygen permeability, which enables oxygen adsorption-desorption processes on the ZnO surface, resulting in a change in the electrical conductivity of the ZnO nanoparticles.

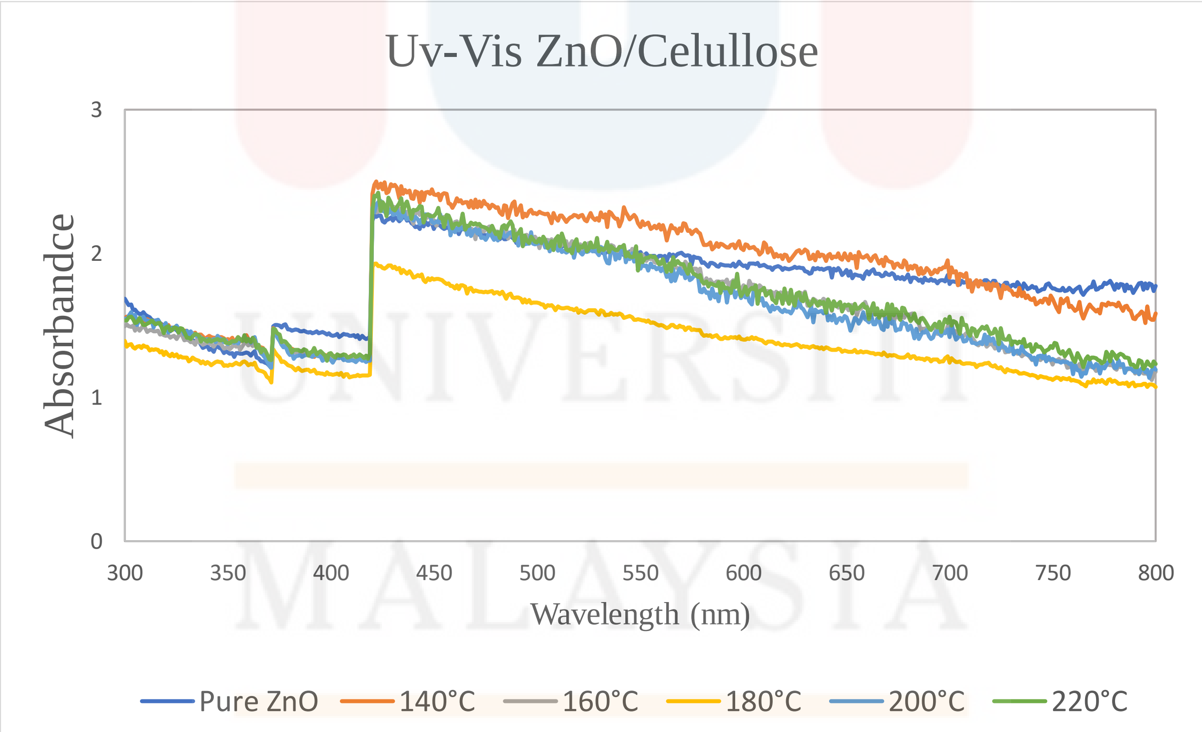
The ZnO-Cellulose composite also exhibits minimal absorption in the visible area, indicating that the ZnO nanoparticles have a poor sensitivity to visible light. This could be because there is not many visible light-induced electron-hole pairs in the ZnO nanoparticles; these pairs do not recombine quickly and do not add much to the electrical conductivity of the ZnO nanoparticles.

The ZnO-Celullose combination exhibits a large absorption band in the UV area, as seen by the absorption spectra. This is because the ZnO nanoparticles are absorbing light (Smith A. B., 2020). The intensity of the absorption band increases with increasing sintering temperature. This is because the higher sintering temperature leads to a more intimate contact between the ZnO nanoparticles and the cellulose fibers, which results in a more efficient transfer of light to the ZnO nanoparticles (Wang, 2018).

A shoulder in the visible range at around 400 nm is also seen in the ZnO-Celullose combination, according to the absorption spectra. This shoulder is the cause to the band gap of ZnO. The energy differential that separates a semiconductor's valence band and conduction band is called the band gap. (Klingshirn, 2010). A photon of light can be absorbed by a semiconductor and excite an electron from the valence band to the conduction band if its energy is equal to or higher than the band gap. Light whose energy is equivalent to or greater than ZnO's band gap is absorbed, producing the shoulder in the absorption spectra at around 400 nm.

The results of the UV-vis absorption spectra suggest that the ZnO-Celullose composite is a promising material for UV photodetectors. The composite ability to efficiently absorb UV light and its strong selectivity for UV light are indicated by the broad absorption band in the UV region and the shoulder in the visible area at about 400 nm. This makes the composite a good candidate for use in UV photodetectors (Liu K. , 2017).

Overall, the ZnO-Celullose combination is an attractive material for UV photodetectors, according to the outcomes of the UV-vis absorption spectra. The composite exhibits a shoulder in the visible range at around 400 nm and a large absorption band in the UV, which indicate that it can efficiently absorb UV light and that it has a good selectivity for UV light. This makes the composite a good candidate for use in UV photodetectors.



4.3: UV-vis absorption spectra of ZnO/Celullose composite sintered at various temperatures

4.4 Band Gap Determination

The Tauc graph was used to display the energy band gap of ZnO at various temperatures. Equation 4.2's Tauc relation formula was used to compute the energy band gap. The calculated date was then plotted in the graph of hv versus $(ahv)^2$ through the absorption coefficient a which is related to the band gap (E_g) as $(ahv)^2 = k\ (hv - Eg)$ by using origin software. By extrapolating a linear projectile toward the x-axis, the direct band gap of the each ZnO sample could be determined. The optical band gap (E_g) is discovered to be distinct temperature dependent, and as temperature rises, the ZnO's band gap decreases.

$$(ahv)1/n = k\ (hv - Eg)$$

Equation 4.2

Equation 4.3, where an is absorption, hv is photon energy, Kiss is the energy independent constant, and E_g is the optical band gap, provides the basis for the derivation of this equation using the Tauc and Davic-Mott relation. Whereas the indirect band gap is 1⁄2, the direct band gap's type of transition is represented by the exponent n, which is 2.

$$Eg = hv$$

Equation 4.3

$$v = \frac{c}{\lambda}$$

Equation 4.4

$$Eg = \frac{hc}{\lambda}$$

Equation 4.5

To convert wavelength into energy, the Max Planck equation (4.3) was utilizes. Equation (4.4) was added to equation (4.3), and Equation (4.5) is real. Then, for the y-axis, the $(h)n$ equation is utilized, where h is the photonic energy ad is the absorbance coefficient. Beer Lamber’s Law, which is illustrated in Equation (4.6), can be used to determine alpha (a) as follows:

$$\frac{I}{I_0} = e^{-aI}$$

Equation 4.6

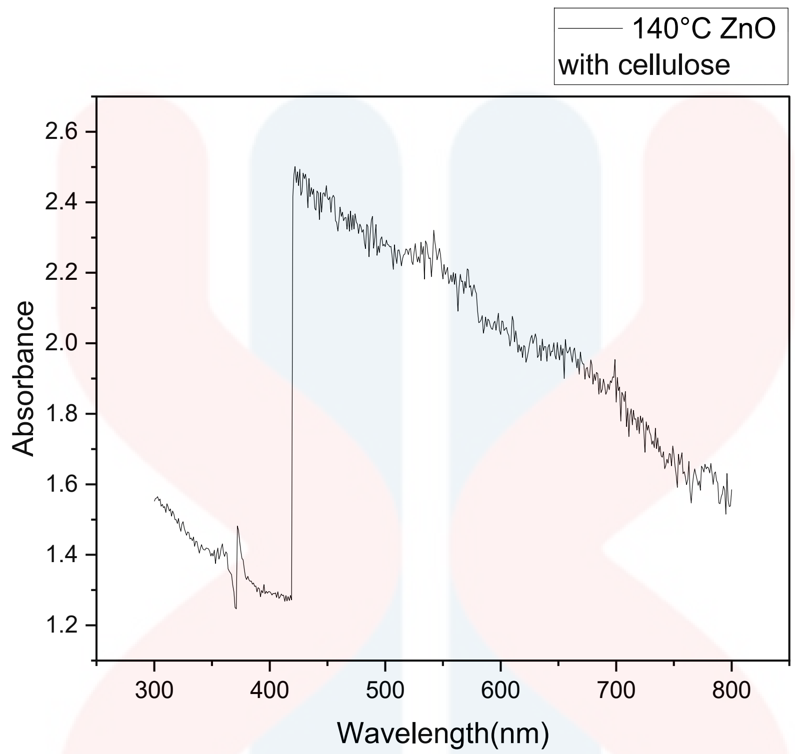
In this equation (4.6), I stand for the intensity of light that is transmitted, I_0 fr the intensity of light that is incident, stands for the absorption coefficient, and is the length of the rote along which the absorbance occurs.

The UV-Vi graph of absorbance versus wavelength for ZnO with various temperature shown in Figure 4.4.1(a), 4.4.2(a), 4.4.3(a), 4.4.4(a) and 4.4.5(a). Figures 4.4.1(b), 4.4.2(b), 4.4.3(b), 4.4.4(b) and 4.4.5(b) illustrate the extrapolated straight line-graphed optical band gap of ZnO, respectively. The energy band gap for Pure ZnO 180°C w/o Cellulose, 140°C ZnO with Cellulose, 160°C ZnO with Cellulose, 180°C ZnO with Cellulose, 200°C ZnO with Cellulose and 220°C ZnO with Cellulose are displayed correspondingly, and are, respectively, 2.764 eV, 2.023 eV, 1.576 eV, 1.712 eV, 1.691 eV and 1.384 eV based on extrapolated straight line’s conclusion. The energy band gap overview for each temperature is shown n Table 4.4.1

and Figure 4.4.6. From that point on, it is said that the energy band gap (E_g), which is related to temperature decrease.

The band gap is the separation between the lowest empty conduction band and the valence band of a semiconductor. It determines the amount of photon energy that the semiconductor has to absorb in order to produce defects and photoelectron. This clearly shows that temperature changes display a quantum confinement effect, which widens the energy band gap and results in a visible spectrum blueshift.

(a)



(b)

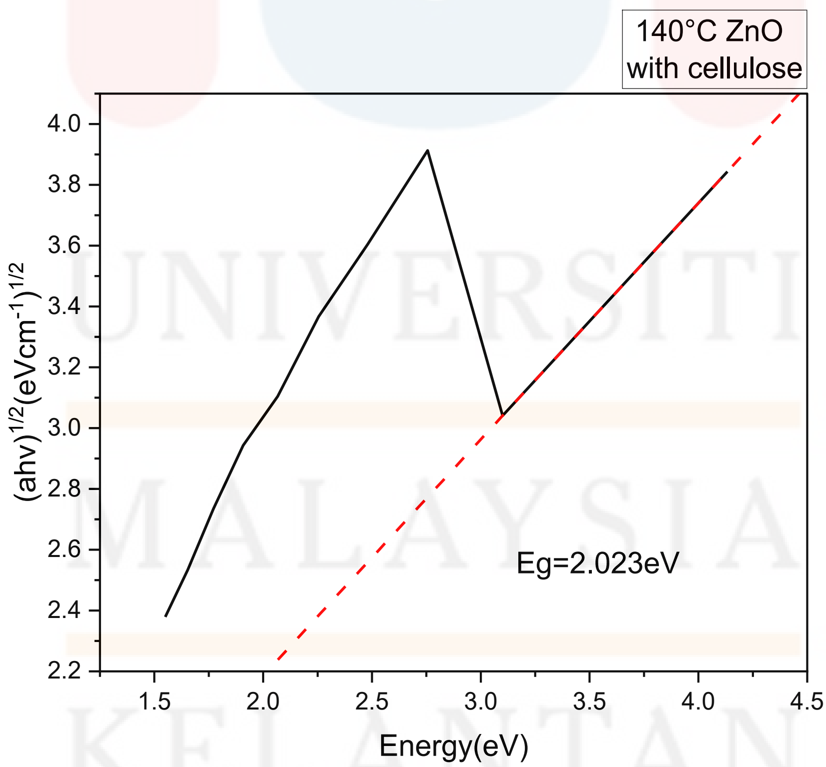
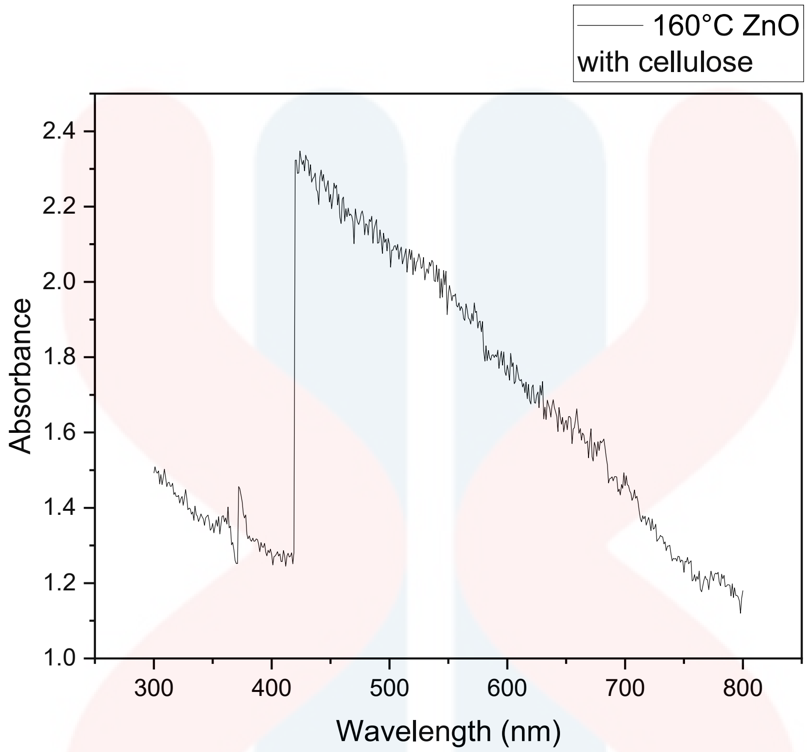


Figure 4.4.1: (a) Absorption spectra of ZnO nanoparticles at temperature 140°C with cellulose and (b) Optical band gap of ZnO at temperature 140°C with cellulose.

(a)



(b)

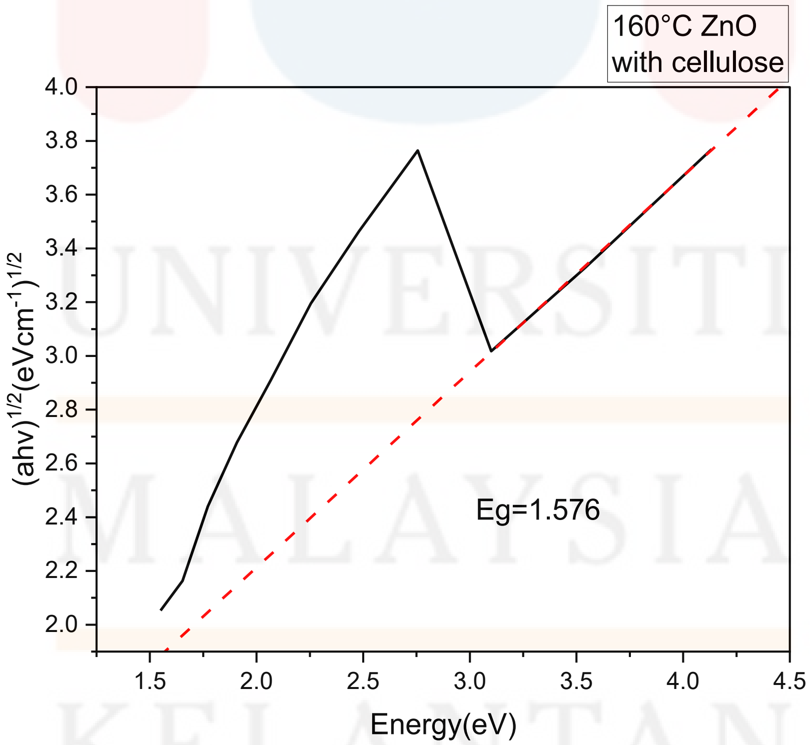
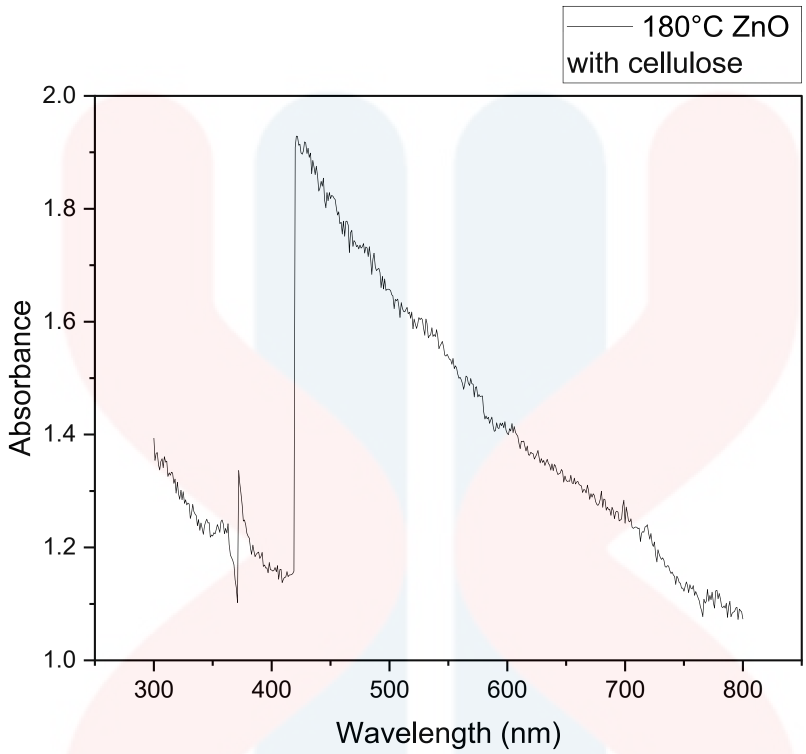


Figure 4.4.2: (a) Absorption spectra of ZnO nanoparticles at temperature 160°C with cellulose and (b) Optical band gap of ZnO at temperature 160°C with cellulose.

(a)



(b)

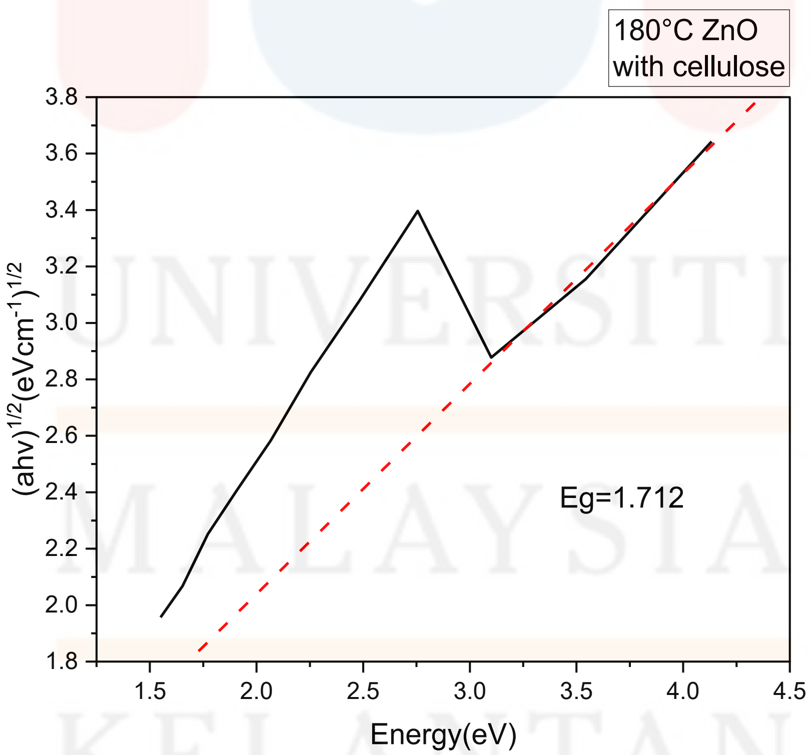


Figure 4.4.3: (a) Absorption spectra of ZnO nanoparticles at temperature 180°C with cellulose and (b) Optical band gap of ZnO at temperature 180°C with cellulose.

(a)

(b)

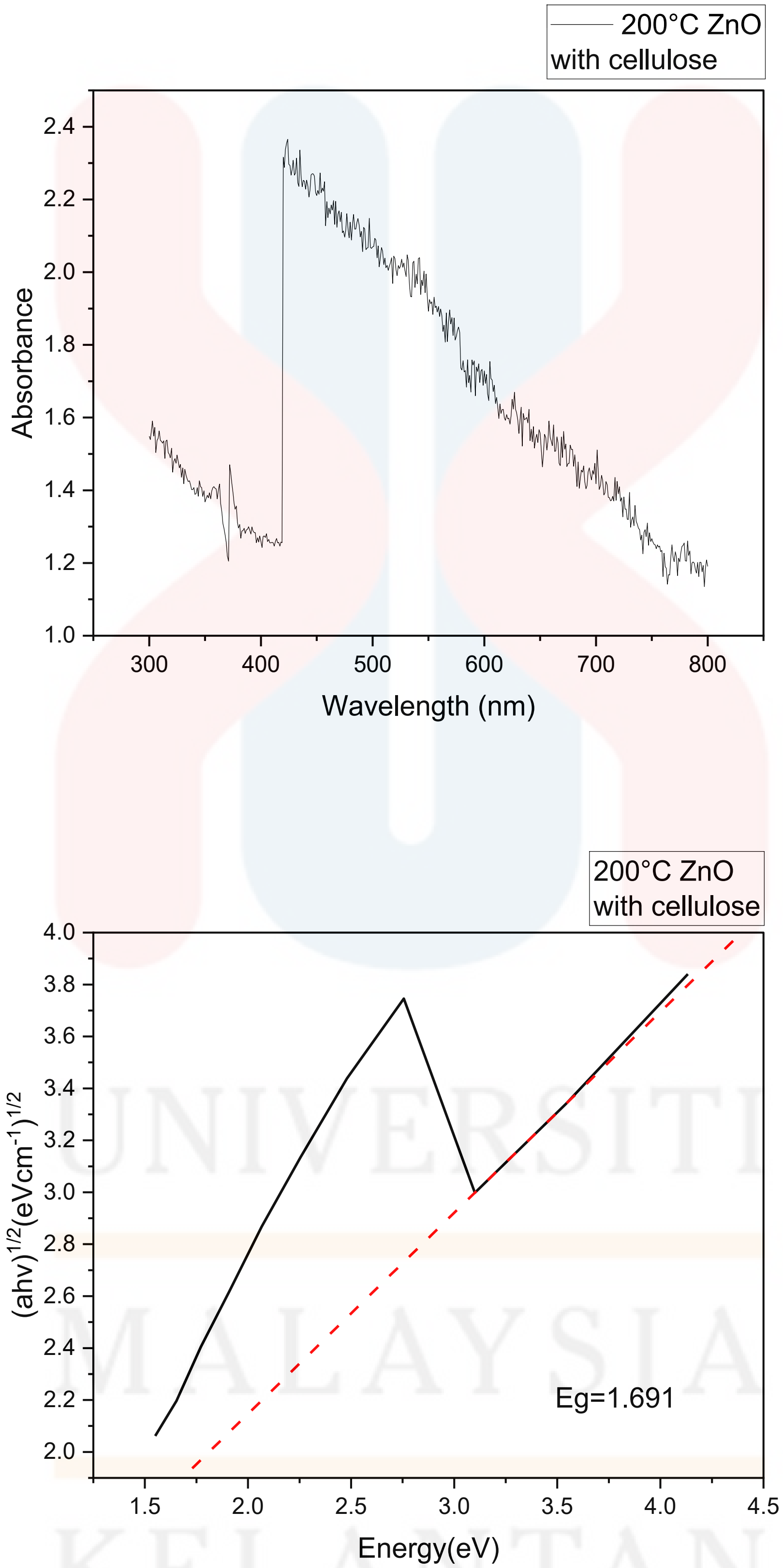
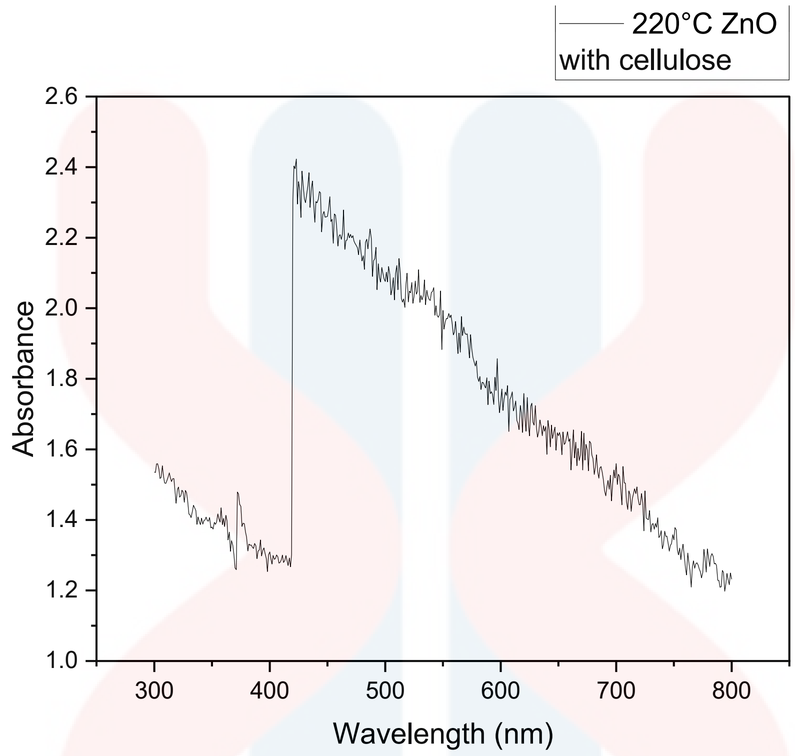


Figure 4.4.4: (a) Absorption spectra of ZnO nanoparticles at temperature 200°C with cellulose and (b) Optical band gap of ZnO at temperature 200°C with cellulose.

(a)



(b)

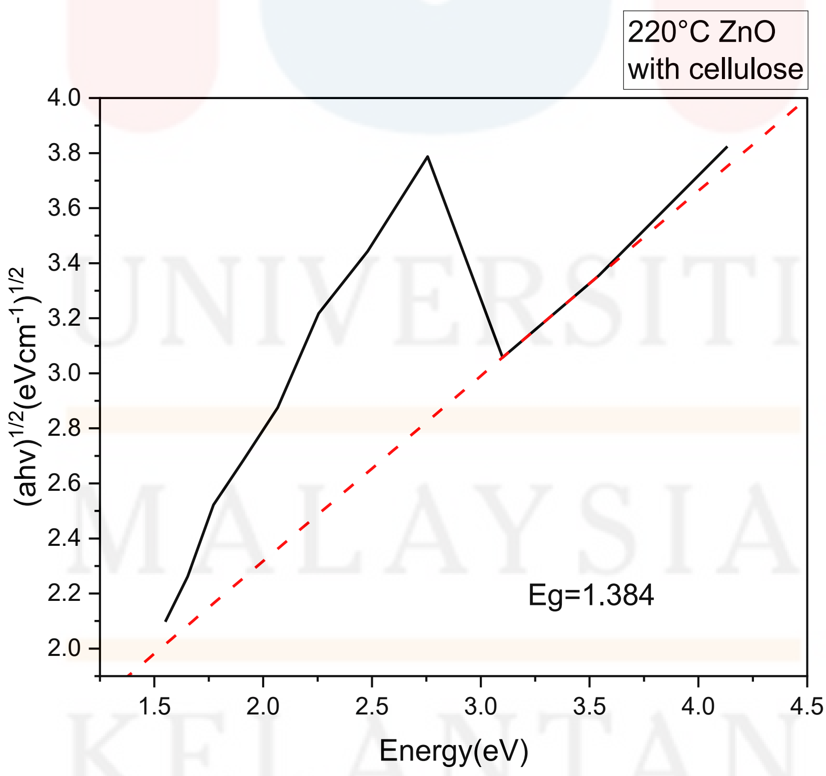


Figure 4.4.5: (a) Absorption spectra of ZnO nanoparticles at temperature 220°C with cellulose and (b) Optical band gap of ZnO at temperature 220°C with cellulose.

Table 4.4.1: The energy band gap for different temperature

Temperature	Band Gap ZnO
140°C	2.023
160°C	1.576
180°C	1.712
200°C	1.691
220°C	1.384

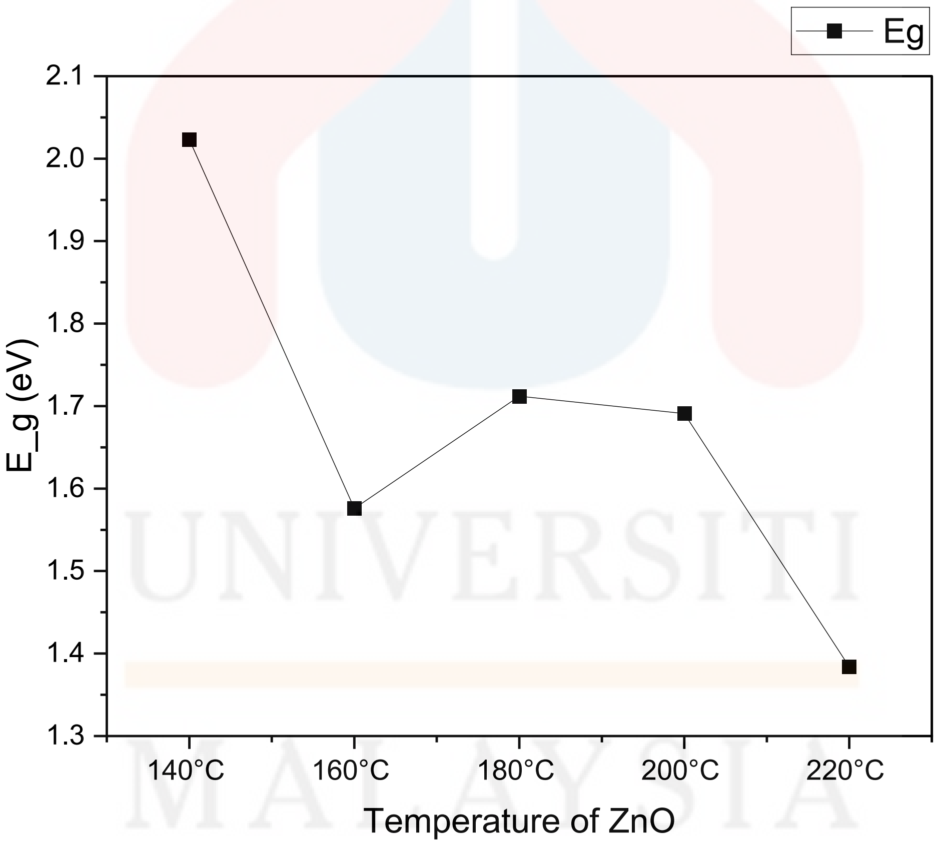


Figure 4.4.6: The optical band gap of ZnO nanoparticles with different temperature

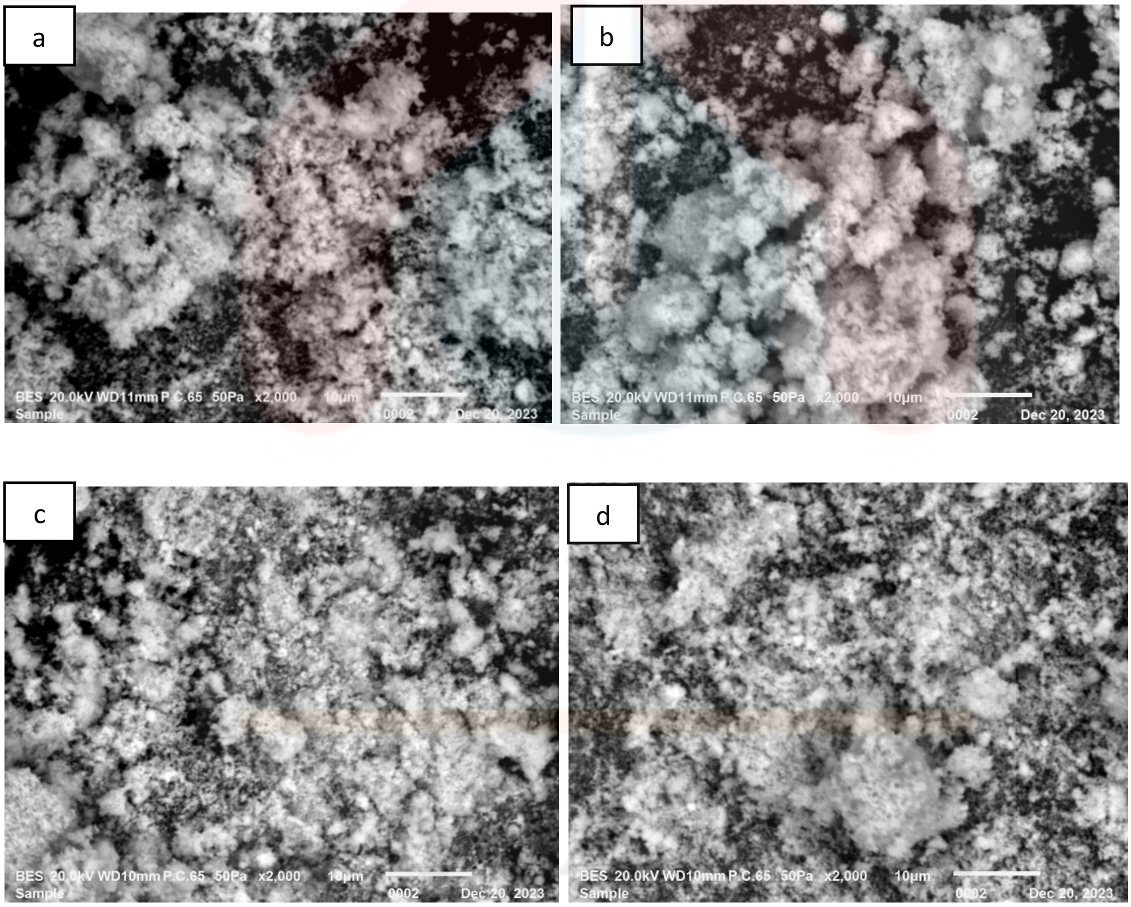
4.5 Scanning Electron Microscopy (SEM)

The SEM analysis was used to characterize the top surface morphology of all fabricated ZnO as represented in figure 4.5.1. The SEM images was used to investigate the particles shape of the prepared samples and from these micrographs. At lower heating temperatures (140°C and 160°C), the SEM images reveal that the ZnO/cellulose composites have a relatively uniform and compact morphology, with ZnO particles well dispersed on the cellulose surface. The ZnO particles are mostly spherical in shape, with some particles appearing to be slightly elongated.

Figures 4.5.1 from the SEM have shown that at low heating temperature, the ZnO particles are well dispersed on the cellulose surface where it has relatively uniformed and compact morphology. For most of the cases, the ZnO particles were assumed to be in a spherical shape and slightly elliptical. The cellulose fibers were also visualized as long and fine structures. Generally, composites should have a smooth and uniform surface, therefore revealing good compatibility.

At higher heating temperatures (180 °C, 200°C and 220°C) the morphological modifications of ZnO/cellulose composites are big as shown by SEM images. According to the images, ZnO particles often have a tendency to aggregate and form larger clusters on the surface of cellulose, a process that ultimately results in the cellulose having a rough and irregular appearance. The ZnO particles in these composites are mostly irregular shape, and some particles seem to be elongated or rod-like. The cellulose fibers were observed to be in the images, but these seem to be more damaged in blocks E and F than in the composites that were prepared at inferior temperatures.

The difference in heating temperature affected the change in shape of the ZnO/cellulose composites due to the changes in the mechanisms of growth and aggregation of ZnO particle at various temperatures. It is observed that the slow and uniform growth at the surface of cellulose at low temperatures results in a relatively uniform and compact morphology of ZnO particles. At higher temperatures, the ZnO ionic mobility enhances and can enable the particle bonding together to grow by coalescence into irregularly shaped and also larger particles (Y. Zhang, 2015).



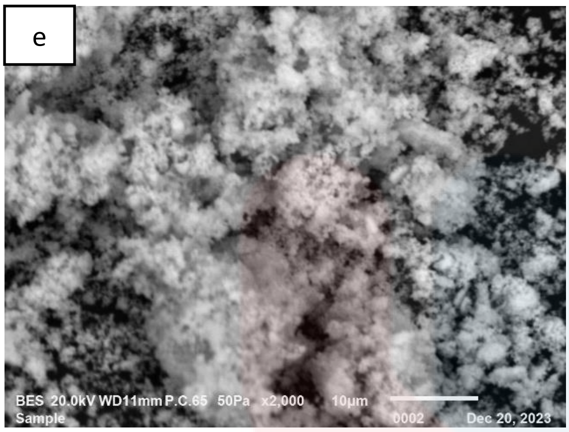


Figure 4.5.1: SEM images of ZnO nanoparticles prepared by hydrothermal method by different temperature (a-e) 140°C, 160°C, 180°C, 200°C and 220°C.

(Magnification: x2000)

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The research aims to synthesize ZnO/cellulose composites at different temperatures using the hydrothermal method and investigate their interaction on structural, mechanical, morphological, and optical properties. The study's scope includes examining the effects of sintering temperatures ranging from 140-220°C on the composites' characteristics, with the intention of providing essential insights into determining the optimal sintering temperature for producing ZnO/cellulose composites using the hydrothermal method. The increase in peak intensity at higher heating temperatures indicates enhanced crystallinity and improved interaction between cellulose and Zno. This finding aligns with previous studies and highlights the potential of the hydrothermal method for producing composites with superior properties

The X-ray diffraction (XRD) patterns of ZnO nanostructures prepared at various temperatures reveal significant insights into the crystallinity and structural properties of ZnO/cellulose composites. The observed increase in peak intensity with higher heating temperatures suggests enhanced crystallinity, indicating improved interaction between cellulose and ZnO. This finding aligns with previous studies, highlighting the potential of the hydrothermal method for producing composites with superior properties.

5.2 Recommendation

1. Exploration of Different Cellulose Sources: Investigate the use of various sources of cellulose (such as different types of biomass or waste materials) in combination with ZnO to determine how the source of cellulose affects the properties of the composites.
2. Optimization of Sintering Parameters: Further study the optimization of sintering parameters beyond just temperature, including sintering time and pressure, to understand their combined impacts on the morphological and structural characteristics of composites made of cellulose and zinc.
3. Functionalization Studies: Explore the possibility of functionalizing the ZnO/cellulose composites with other materials or additives to enhance specific properties such as photocatalytic activity, mechanical strength, or biodegradability.
4. Scale-Up and Industrial Applications: Conduct research on scaling up the synthesis of ZnO/cellulose composites using the hydrothermal method for potential industrial applications, considering factors like cost-effectiveness, reproducibility, and large-scale production feasibility.
5. Long-Term Stability Studies: Examine the endurance and stability of ZnO/cellulose composites over an extended period of time in various environmental settings to see whether they have the potential to be used in real-world settings such packaging, biomedicine, or environmental remediation.

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