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Preparation and characterization of ZnO at different amounts of cellulose composite using hydrothermal method

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DECLARATION

I declare that this thesis entitled "Preparation and characterization of ZnO at different amounts of cellulose composite using hydrothermal method" is the results of my own research except as cited in the references.

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Student's Name : Nor Irsalina Binti Mohamad Nazeru

Date : _____

Verified by:

Signature : _____

Supervisor's Name : Dr. Arlina Binti Ali

Stamp : _____

Date : _____

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Penyediaan dan pencirian ZnO pada jumlah komposit selulosa yang berbeza menggunakan kaedah hidrotermal

ABSTRAK

Dalam kajian ini, zink oksida berjaya disintesis melalui kaedah hidrotermal mudah dengan mengawal jumlah selulosa yang berbeza. Zink klorida digunakan sebagai prekursor logam peralihan untuk mensintesis ZnO dengan kehadiran natrium hidroksida. Campuran dipanaskan pada suhu 180 gram C selama 6 jam untuk semua sampel. Jumlah selulosa yang berbeza dari (1, 2, 3, 4 dan 5) g telah ditambah dalam campuran. Variasi morfologi, jurang pita optik zink oksida berbeza dengan jumlah selulosa yang berbeza telah dikaji. Sampel-sampel itu dicirikan oleh difraksi sinar-x (XRD), spektroskopi UV-vis (UV-vis) dan mikroskop elektron pengimbas (SEM). Corak XRD menunjukkan bahawa struktur ZnO / selulosa menunjukkan struktur wurtzite heksagonal dengan saiz kristallit purata dikira. Spektroskopi UV-Vis menunjukkan puncak penyerapan atau pantulan zink oksida adalah sekitar 300 hingga 400 nm menunjukkan kehadiran pergeseran biru. Jurang pita tenaga didapati berkurangan dengan jumlah selulosa yang berbeza. Oleh itu, jumlah selulosa yang berbeza memainkan peranan penting dalam kawalan struktur dan perubahan jurang pita. Imej morfologi oleh SEM mendedahkan struktur zarah yang terkumpul untuk semua sampel.

Kata kunci: Zink Oksida, Selulosa, Kaedah Hidrotermal, Wurtzite Heksagonal, XRD, SEM, UV-Vis.

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Preparation and characterization of ZnO at different amounts of cellulose composite using hydrothermal method

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ABSTRACT

In this paper, zinc oxide were successfully synthesized via simple hydrothermal method by controlling the different amounts of cellulose. The zinc chloride was use as a transition metal precursor to synthesize the ZnO with the presence of sodium hydroxide. The mixture were heated at 180 °C for 6 hours for all samples. The different amount of cellulose from (1, 2, 3, 4 and 5) g were added in mixture. The morphogical variation, the optical band gap of zinc oxide varied by the different amount of cellulose were studied. The samples were characterized by X-ray diffraction (XRD), UV-vis spectroscopy (UV-vis) and Scanning electron microscope (SEM). XRD pattern shows that the ZnO/Cellulose structured exhibit the hexagonal wurtzite structure with the average crystallite size is calculated. UV-Vis spectroscopy shows the absorption or reflectance peaks of zinc oxide was around 300 to 400 nm indicates the presence of blueshift. The energy band gap was found to be decrease with the different amount of the cellulose. Thus, different amount of cellulose plays a crucial role to the structure control and turning of band gap. The morphology images by SEM revealed the agglomerated particles structures for all samples.

Keywords: Zinc Oxide, Cellulose, Hydrothermal method, Hexagonal wurtize, XRD, SEM, UV-Vis spectroscopy.

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TABLE OF CONTENT

FYP FBKT
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FYP FBKT
UNIVERSITI MALAYSIA KELANTAN

LIST OF TABLES

FYP FBKT

UNIVERSITI
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LIST OF FIGURES

FYP FBKT
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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Inorganic nanoparticle synthesis in nanofibular materials has recently received a lot of attention because of the wide range of potential uses for inorganic nanoparticles in the fields of catalysts, nanocomposites, sensors, optoelectronics, and electronic nanodevices (Liu et al. 2010; Yin et al. 2010). (Patel et al. 2007) Both the host polymer and the embedded composite particles have physical sizes that are in the submicrometer or nanometer range, keeping the typical enormous surface area of nanofiber materials and nanoparticles. Furthermore, it has been demonstrated that these nanocomposites combine the benefits of inorganic particles and polymers, among these include specialized functionality, high strength, and heat stability (Liu et al. 2010; Chronakis 2005; Wang et al. 2005). This is because they are both polymers and inorganic particles, and both have advantages such as being flexible, moldable, and lightweight. To improve the chemical and physical properties of matrices, cellulose has been used as a reinforcing phase in the majority of studies. Recently, metallic nanoparticles have been created using cellulose nanocrystals as a substrate (Cai et al. 2009). For instance, cellulose nanocrystals were used to synthesize Au nanoparticles with a limited size distribution (Khaled et al. 2009). In other experiments, metal-containing composites have been produced using carboxylated cellulose nanocrystals. (Liu et al. 2010) For instance, Ag-Pd alloy particles separately synthesized tiny Ag and were successfully dispersed in carboxylated cellulose

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nanocrystals. Most metallic particles can adsorb to the surface of the cellulose due to the dispersion of cellulose nanocrystals and metallic salts caused by electrostatic interactions between oxygen atoms of polar hydroxyl and metallic particles. (He et al. 2003). This effect prevents particle aggregation, which in turn regulates the sizes of metallic nanoparticles.

1.2 Problem Statement

Having a high exciton binding energy, a direct semiconductor with a broadband gap is ZnO. Due to their enormous surface area and high surface energy, zinc oxide nanoparticles tend to agglomerate. Nanoscale cellulose has exceptional characteristics such as a high capacity for absorbing metallic particles, a large aspect ratio, good water dissolvability, strong mechanical capabilities, and little thermal degradation behavior. So, that's why cellulose will be added to zinc oxide to improve the agglomeration. The hydrothermal technique is a viable alternative synthetic method because of the low process temperature and the simplicity with which the particle size can be adjusted. In comparison to other growth methods, the hydrothermal process has several benefits, including the use of simple tools, catalyst-free development, low cost, uniform production over a large area, environmental friendliness, and less risk. The low reaction temperatures make this method appealing for microelectronics and plastic electronics.

When zinc oxide and cellulose are mixed, their mechanical properties will be changed. So, the structural, absorption band, and optical properties at different amounts of cellulose also give different results.

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1.3 Objectives

The objectives of this research are:

1. To prepare the different amount of cellulose (1, 2, 3, 4 and 5)g with ZnO composites using hydrothermal method.
2. To characterize the structural, morphology, and optical properties of cellulose/ZnO composites.

1.4 Scope of Study

To prepare consistent dispersion morphology of cellulose/ZnO nanoparticles hydrothermal technique in the current study, ZnO nanoparticles were synthesized within cellulose. The structural, absorption band and optical properties at different amounts of cellulose with ZnO were studied.

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1.5 Significances of Study

Most of the research has used cellulose as a reinforcing phase to enhance the chemical and physical characteristics of matrices. Recently, metallic nanoparticles have been created using cellulose as a substrate. For instance, in cellulose nanocrystals, narrowly scaled Au nanoparticles were created. In other experiments, metal-containing composites have been produced using carboxylated cellulose nanocrystals. For instance, small-sized Ag and Ag-Pd alloy particles were independently produced and effectively distributed in carboxylated cellulose nanocrystals. Due to electrostatic interactions between metallic particles and oxygen atoms in polar hydroxyl and cellulose nanocrystals, most of the metallic particles in a suspension of cellulose nanocrystals and metallic salts can adsorb on the surface of CNC. By avoiding particle aggregation, this action regulates the sizes of metallic nanoparticles.

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CHAPTER 2

LITERATURE REVIEW

2.1 Zinc chloride

Zinc chloride ($ZnCl_2$) is a chemical compound composed of zinc and chlorine. It is a white crystalline solid that is highly soluble in water (Tina Wu, 2022). Zinc chloride can exist in various forms, including anhydrous (without water) and hydrated (containing water) forms. Zinc chloride is highly soluble in water, and its aqueous solution conducts electricity, indicating the presence of ions (Tina Wu, 2022). Anhydrous zinc chloride is hygroscopic, meaning it readily absorbs moisture from the air. Zinc chloride has a relatively high melting point and boiling point for a molecular compound. Zinc chloride is known as a Lewis acid, meaning it can accept a pair of electrons from another substance in a chemical reaction. It is used as a catalyst in certain chemical reactions, such as the Friedel-Crafts acylation reaction in organic synthesis. Zinc chloride is involved in the galvanizing process, which is a method used to coat steel or iron with a layer of zinc to protect it from corrosion. In soldering applications, zinc chloride is sometimes used as a flux, aiding in the removal of oxide layers from metal surfaces and promoting better solder adhesion. Zinc chloride is used in the preservation of wood, providing protection against decay and insects. It has been used in certain types of batteries as an electrolyte. Anhydrous zinc chloride is a strong dehydrating agent, and it is used in some chemical processes to remove water from reactions.

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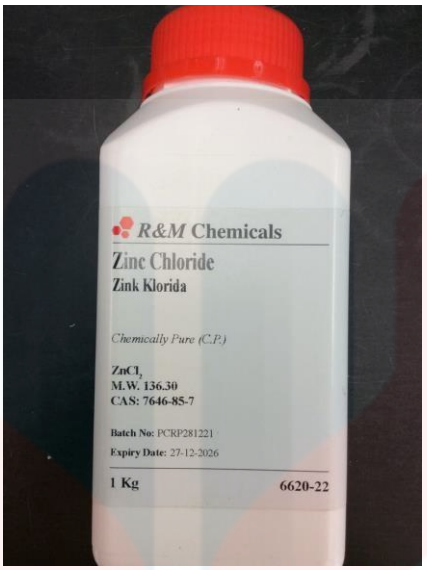

Figure 2.1: Zinc Chloride powder

2.2 Cellulose

Cellulose is tasteless, odourless, and biodegradable. It is a carbohydrate polymer with a straight chain. Like the other carbs, it is an organic component. It consists of a linear chain of many glucose residues (between 300 and 1000 units, for example) joined together by a glycosidic bond. The hydroxyl groups on glucose from one chain link with the oxygen atoms on glucose from another or the same chain through hydrogen bonding. There are no glycosidic connections between the chains. Hydrogen bonds hold the chains side by side to one another. As a result, cellulose is visible as a microfibril. It acts as the "cytoskeleton" of the plant, giving the cell wall tensile strength. The size of the chain or degree of polymerization affects the other properties of cellulose. In addition to plants, several types of creatures also naturally create cellulose. It has been discovered that some bacteria, protists, algae, and animals (such as tunicates) generate it. The earliest known organism to make cellulose is thought to have been a cyanobacterium. In higher plants, cellulose is mostly made in the extracellular matrix or cell wall, which is located outside

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the cell. The proteinaceous rosette terminal complex, which is a floating structure at the plasma membrane, produces it. Cellulose synthases, which are involved in the synthesis of the cellulose chain, are present in the complex. The cell walls of plants must include cellulose. The cell wall is stabilized, stiffened, and strengthened by it. It's probable that this carbohydrate provides energy to animals that can digest cellulose. Humans are unable to digest cellulose because they lack the necessary enzymes. However, as a dietary fibre, cellulose can still be ingested in moderation. Pumpkin seeds, avocado, cherries, apples, lentils, and cabbage. In the food industry, it may also be chemically processed for use as a creaming agent or thickening for parmesan cheese, ice cream, and other processed foods. These foods' cellulose acts as an insoluble fibre when taken, absorbing water to increase stool volume. Normal human large intestine microbiota can digest cellulose and create short-chain fatty acids and gases. The body absorbs and processes the short-chain fatty acids. There are numerous industrial uses for cellulose as well. For instance, the cotton plant creates cotton fibres with a cellulose content of more than 90%. They can be harvested to make building materials, clothing, paper, rayon, and cellophane. Energy crop cellulosic material has also been used to create biofuels, such as cellulosic ethanol.

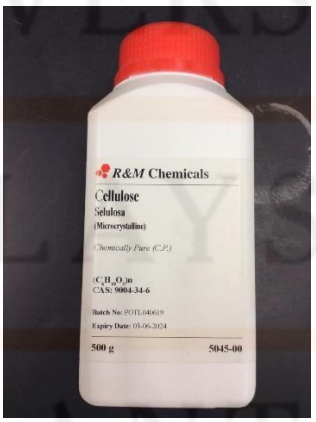

Figure 2.2: Cellulose powder

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2.3 Sodium Hydroxide

Sodium hydroxide (NaOH) is a chemical compound called caustic soda or lye. It is an inorganic alkali and a highly caustic metallic base. Sodium hydroxide is composed of sodium ions (Na^+) and hydroxide ions (OH^-). It is a white, crystalline solid at room temperature and is highly soluble in water, producing an alkaline solution. Sodium hydroxide is highly caustic and can cause severe chemical burns. It reacts strongly with proteins and lipids in living tissues, leading to its use in various industrial processes and applications (James Thompsom, 2003). Sodium hydroxide is a strong base, and its aqueous solution is highly alkaline. It can be used to neutralize acids and adjust the pH of solutions. Sodium hydroxide participates in a variety of chemical reactions, including neutralization reactions with acids to form water and salts. Sodium hydroxide has numerous industrial applications. It is used in the production of paper, textiles, soaps, detergents, and various chemicals. It is also employed in petroleum refining, aluminium production, and in the synthesis of various organic compounds. Due to its ability to dissolve grease and proteins, sodium hydroxide is a common ingredient in some drain cleaners. In the food industry, sodium hydroxide is used in the processing of certain foods, such as in the preparation of pretzels. Sodium hydroxide is used in water treatment processes to adjust the pH of water and to neutralize acidic substances. It is used in laboratories for various purposes, including titrations, and as a strong base in chemical reactions (S. Ramachandra Rao, 2006). When handling sodium hydroxide, it is important to follow proper safety precautions, as it can be corrosive and cause burns. Protective equipment, such as gloves and goggles, should be used when working with this substance. Additionally, it should be stored and handled with care to prevent accidental exposure.

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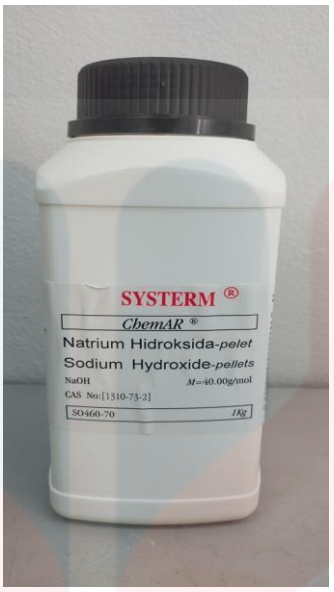

Figure 2.3: Sodium Hydroxide

2.4 Hydrothermal Method

A procedure known as the "hydrothermal method" is described, which involves using an aqueous solution as a reaction system in a particular closed reaction vessel to create a high-pressure reaction environment and high temperature. Heat and pressure are applied to the reaction system in order to accomplish this. Under normal circumstances, an insoluble or barely soluble substance dissolves and recrystallizes. There are four stages to the hydrothermal method: synthesis, hydrothermal crystal growth, reaction, and treatment. These stages are utilised to make different kinds of single crystals, practical ceramic powders, conduct some organic reactions, and manage some hazardous organic wastes for the environment. Additionally, certain ceramic materials can be sintered using it at comparatively low temperatures (Vanja Kokol, 2019).

A low-temperature hydrothermal method and a supercritical hydrothermal method can be identified based on the reaction temperature. A standard hydrothermal method and

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a specialised hydrothermal method are the two categories into which it can be separated based on the variations in the equipment. Recent years have seen a significant amount of research conducted on the hydrothermal preparation technique. The goal of the hydrothermal process is to replicate how ores naturally form. It belongs to the group of soft chemical synthesis. In addition, it can be used to handle some organic waste that is detrimental to the environment in which people live, produce a variety of single crystals, make ultrafine or less agglomerated crystallised ceramic powders, and sinter some ceramic materials at low temperatures (Xu et al., 2018).

The widespread application and increasing importance of the hydrothermal method have led to a continuous process of improvement. For example, there has been much interest in applying electric fields, mechanical mixing, and microwaves to enhance the reaction kinetics of the hydrothermal method. These procedures have reduced the amount of time needed for experiments, making this technology more practical and affordable. Among them, the microwave hydrothermal technique has garnered significant attention in recent times for research purposes. As an advancement of the hydrothermal process, it has gained popularity in the preparation of ceramics. The temperature differential caused by the hydrothermal process is effectively compensated for by the microwave temperature. The size, morphology, and aggregation of ceramic oxide crystals can also be affected by varying the temperature, the pH of the reaction system, the starting material ratio, and the reaction time.

With so many advantages, the hydrothermal process holds great potential for the production of materials such as garnets, vanadates, thin films, bioceramics, and ceramic oxides, among others. In general, materials for hydrothermal preparation are still the subject of active research and development. Non-aqueous solvent systems are the subject of very few hydrothermal studies. Most materials made using the hydrothermal process

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are oxides and oxygenated salts. It is crucial to conduct research on the physicochemical properties of hydrothermal devices, solvents, and mineralizers as well as the mechanisms underlying chemical reactions during hydrothermal processes in order to promote the preparation of materials using these methods. As modern materials science and engineering research advances so too will the range of applications and basic theory of hydrothermal methods. The hydrothermal method will become more widely used at the same time as the intersection of disciplines as a result of the emerging trend of combining it with other approaches.

2.5 Structural and optical properties

Researchers have employed a variety of methodologies to examine the effects of varying the amount of cellulose used in the fabrication and characterization of the ZnO/Cellulose composites on their shape and crystal structure. The phase composition and crystallinity of the composites have been examined using X-ray diffraction (XRD) analysis, and an infrared spectrum of the absorption or emission of a solid, liquid, or gas has been obtained using Fourier-transform infrared spectroscopy (FTIR). The performance of the ZnO/Cellulose composites has also been assessed using measurements of optical characteristics such as UV-vis absorption. According to reports, the different cellulose amounts significantly affect the composites' optical characteristics. In conclusion, if the amount of cellulose increases, the structural and optical properties of the sample will improve the preparation and characterization of ZnO/Cellulose.

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CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

In this work, $ZnCl_2$, cellulose, and NaOH were used as materials. R&M is the brand name of the company that makes zinc chloride ($ZnCl_2$). Environmental toxicity is the category of risk, and the chemical name is $ZnCl_2$. The faculty is the owner of it. This substance's charge number is 7646-85-7, and the content is 1000 g. Cellulose is the following material. Cellulose is the product's chemical name, while R&M is the brand name. The faculty is the owner of it. This substance's charge number is 9004-34-6, and the weight is 500 g. This substance's molecular structure is $(C_6H_{10}O_5)n$. Sodium hydroxide is the last material. the chemical name is sodium hydroxide and R&M is the brand name. The owner of the substances is the faculty. This substance's charge number is 1310-73-2 and the weight is 1000 g. This substance's chemical formula is NaOH.

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3.2 Methods

To prepare the ZnO solution. First, we have to combine $ZnCl_2$ and NaOH in one beaker. Then, the solution was mixed with 100 ml distilled water for 30 minutes. After 30 minutes, the different amounts of cellulose were added to the solution (1, 2, 3, 4 and 5)g. The solutions were mixed in 10 minutes. After that, the solutions were poured into the Teflon. Then, the Teflon was put into the oven for 6 hours to dry at 180°C. After drying for 6 hours, the samples were cleaned using distilled water several times. Then, after drying for the first time, the samples were put again in the oven for one hour at 80°C to make sure the samples were 100% dried. Then, after the samples were dried, the samples were grind into the smooth powder. The purpose of grinding is to produce smaller particles from larger ones, and this size reduction may then assist other processes.

3.3 Characterization

Characterization methods are used in research and development to discover qualities like structure and functionality. For the greatest success, several materials and techniques were used in various industries and applications. Physical and chemical characteristics can be used to categorize this description.

3.3.1 X-ray Diffraction (XRD)

One effective analytical method for determining a crystalline material's structure is X-ray diffraction, or XRD. It is widely used to ascertain the arrangement of atoms in a crystal lattice in a variety of scientific and industrial domains. The foundation of X-ray diffraction by crystals, first put forth by Max von Laue and later refined by

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William and Lawrence Bragg, who shared the 1915 Nobel Prize in Physics for this discovery, forms the basis of X-ray diffraction (XRD). X-ray diffraction analysis (XRD), a non-destructive technique, provides exact information about the crystallographic structure, chemical composition, and physical properties of a material. It is predicated on the constructive interference of monochromatic X-rays and crystalline samples. X-ray a non-destructive method for learning about the atomic and molecular configurations of a variety of materials is diffraction. It has significant practical applications in numerous scientific and industrial domains and has advanced our understanding of the structure of matter.

3.3.2 UV-Vis Spectroscopy

UV-Vis Spectroscopy (also known as Spectrophotometry) is a quantitative method for calculating how much light an individual molecule absorbs. By contrasting the amount of light that travels through a sample with that which passes through a reference sample or a blank, this is achieved.

3.3.3 Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) is a potent and useful imaging tool in the modern day. It uses a vacuum system and a variety of lenses to produce an electron beam, and depending on the hardware, it can scan the surface at magnifications of 1 µm to 1 nm. To take advantage of elemental analysis at the specimen surface, an energy-dispersive spectrometer is integrated with a scanning electron microscope. The scanning potential has significantly increased thanks to two new SEM imaging features: secondary electrons and backscattered electrons. Along with other parts, the electron gun forms the

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core of the SEM. This chapter discusses the various kinds of electron guns and their characteristics. Different magnetic lenses and a vacuum system distinguish SEM as a special kind of imaging (Annika Singh, 2021).

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CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

Appended of the objectives of this research, the different amount of cellulose from 1 g, 2 g, 3 g, 4 g and 5 g of ZnO nanostructures by hydrothermal method. All samples of ZnO/Cellulose were characterized by X-ray Diffraction (XRD), UV-Vis Spectroscopy (UV-Vis) and Scanning Electron Microscopy (SEM). These characterization tests were carried out to study the structural, absorbance band and optical properties of ZnO/Cellulose nanostructures. Such as, X-ray Diffraction (XRD) studied the phase identification of a crystalline materials. For UV-Vis Spectroscopy, studied the visible regions of the electromagnetic spectrum and identified the band gap. Scanning Electron Microscopy (SEM) studied on the surface/morphology characteristics in three-dimensional structure images.

4.2 X-ray Diffraction (XRD)

Figure 4.1 shows the X-ray Diffraction (XRD) pattern of ZnO/Cellulose nanostructures with different amounts of cellulose 1 g, 2 g, 3 g, 4 g and 5 g. The (h,k,l) peaks of ZnO/Cellulose were detected at 2θ values of 31.6°, 34.1°, 36.1°, 47.2°, 56.4°,

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62.4°, 66.1°, 67.6°, 68.8°, 71.9°, 76.6° are corresponded to the lattice planes (100), (002), (101), (102), (210), (103), (200), (212), (201), (004), and (202) respectively. These peaks that appeared at different 2θ values indicate a hexagonal wurtzite structure of ZnO nanoparticles.

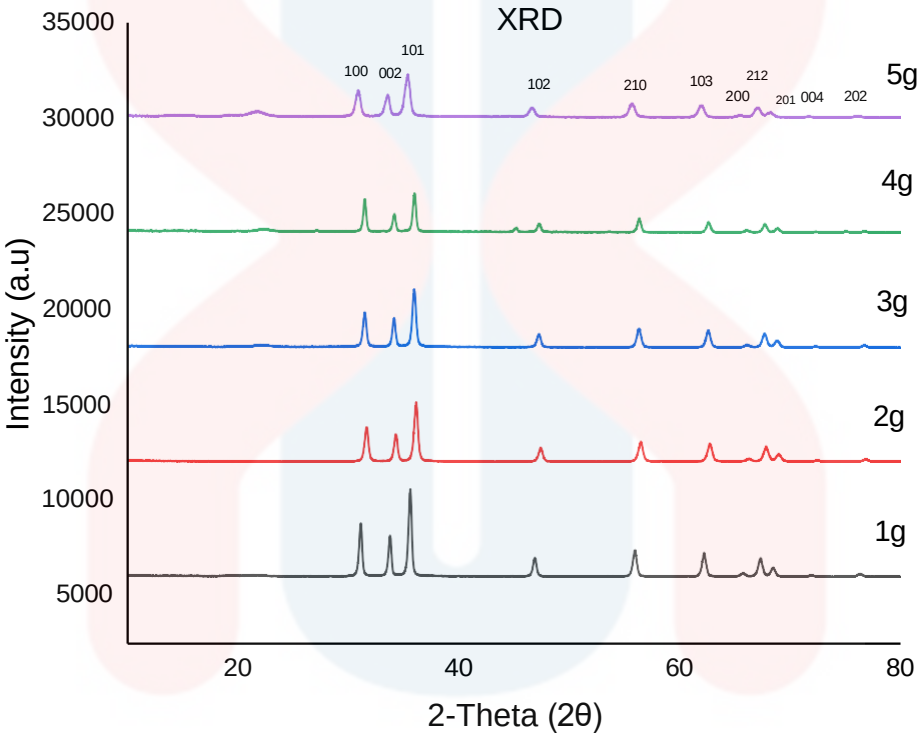

Figure 4.1 : XRD patterns of ZnO structures prepared at different amount of cellulose by hydrothermal method.

From the result in Figure 4.1, the intensity of the graph decreases with the increasing amount of cellulose. The sharp peak of (101) plane in XRD graph indicated a high crystalline of the samples. In fact, intensity of (101) plane is found to be maximum for all 5 samples and the intensity of the peak decreases as the amount of cellulose increases, 1 g < 2 g < 3 g < 4 g < 5 g. The peak of (101) was prominent and absence of the other peaks in the XRD graph indicate purity of the samples (Mohite & Kothawale,

2015). In the meantime, the (101) plane which is the strongest peak can be used to calculate the mean crystalline size of the prepared samples at different amount of cellulose by using Scherrer equation. The hexagonal wurtzite structure of ZnO has a lattice parameter constant of a = 3.2538 Å and c = 5.2044 Å. a and b is constant while c is not constant. When a and b are equal, but c is different in the lattice parameter for X-ray diffraction (XRD), it indicates a crystal lattice with a specific type of symmetry. When a and b are equal, it means that the crystal lattice has a square or rectangular base in the a-b plane. This is often referred to as basal symmetry. The crystal system exhibiting this behaviour is likely to be orthorhombic. In an orthorhombic crystal system, the lattice parameters a, b, and c are all different, and the angles between the axes are 90 degrees. The equal values of a and b imply that the crystal lattice is symmetrical with respect to rotations in the a-b plane. This symmetry leads to equivalent lattice parameters along the a and b directions. The fact that c is different indicates that there is a difference in the spacing along the c axis, perpendicular to the a-b plane. This could be due to various factors, such as the presence of different types of atoms or arrangements along the c axis. In an orthorhombic crystal system with a=b, the crystallographic axes a and b are essentially interchangeable in terms of crystal symmetry. The c axis, however, has a different length and represents a distinct crystallographic direction.

The a/b ratio and c/a ratio in X-ray diffraction (XRD) results are parameters derived from the lattice parameters of a crystal structure. These ratios provide information about the shape and symmetry of the crystal lattice. The a/b ratio is the ratio of the lengths of the crystallographic axes a and b. It provides information about the shape of the crystal lattice in the plane perpendicular to the c axis. The c/a ratio is the ratio of the length of the crystallographic axis c to the length of either a or b. It provides information about the elongation or compression of the crystal lattice along the c axis. In a tetragonal system,

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where a=b but c may be different, the c/a ratio helps describe the elongation or compression along the c axis. Table 4.1 were shown in above.

Table 4.1 : Unit cell parameters of ZnO with different amounts of cellulose

ZnO with different amounts of cellulose	Lattice parameter (Å)			a/b ratio	c/a ratio	Crystallite size (D), nm
	a	b	c			
1 g	3.2538	3.2538	5.2044	1.000	1.5994	25.8
2 g	3.2538	3.2538	5.2044	1.000	1.5994	25.5
3 g	3.2538	3.2538	5.2044	1.000	1.5994	25.6
4 g	3.2538	3.2538	5.2044	1.000	1.5994	25.7
5 g	3.2538	3.2538	5.2044	1.000	1.5994	26.7

The average crystallite size of ZnO with different amounts of cellulose was calculated using Scherrer formula as shown in Equation 4.1:

$$D=K\lambda / \beta\cos\theta$$

Where D is crystallite size, λ for wavelength of the X-ray beam used (1.54060Å), k is the crystallite shape factor, a good proximation is 0.9, where d is the value of full width half maximum (FWHM) and the θ is the angle in radians. Table 4.1 shows the different crystallite size for ZnO with different amount of cellulose 1 g, 2 g, 3 g, 4 g, and 5 g at 25.8, 25.5, 25.6, 25.7 and 26.7 nm respectively. From the table, the average crystallite size for ZnO nanostructures is calculated as 25.86 nm.

Figure 4.2, the graph of crystallite size versus with different amount of cellulose shows the decreases, increases when the different amount of cellulose increases. The increase in crystalline size could be due to the increase in concentration of OH^- during the hydrothermal process.

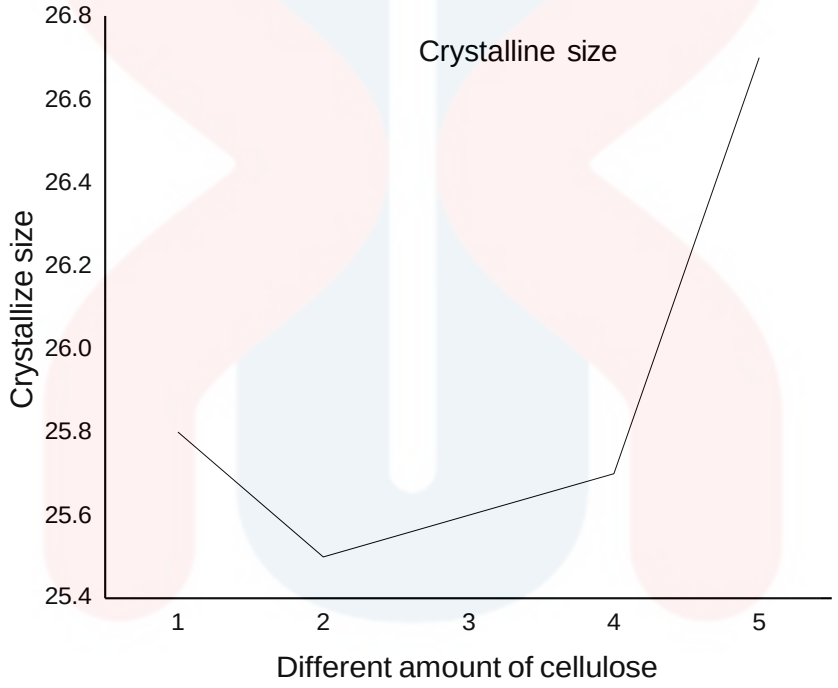

Figure 4.2 : Crystallite size versus different amount of cellulose

The hexagonal wurtzite structure of ZnO has a lattice parameter constant of a = 3.2538 Å and c = 5.2044 Å for all samples as displayed in figure 4.3. The results show constant because it provide valuable information about the shape, symmetry anisotropy of the crystal lattice.

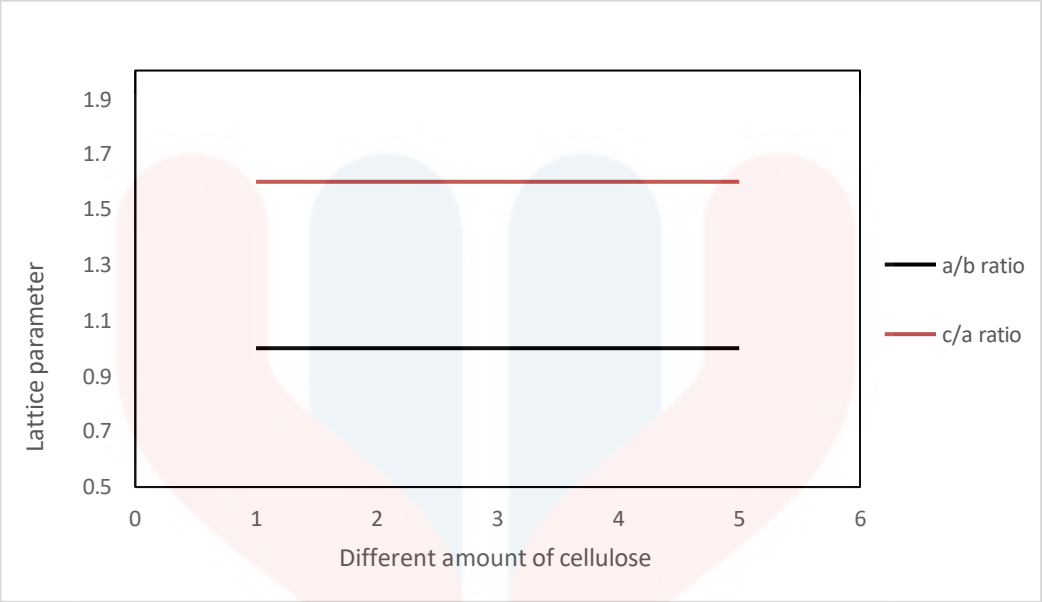

Figure 4.3 : Lattice parameter versus different amount of cellulose

4.3 UV-Vis Spectroscopy (UV-Vis)

Figure 4.4 shows the UV-Vis Spectroscopy graph of ZnO nanoparticles with different amounts of cellulose from 1 g, 2 g, 3 g, 4 g, and 5 g. The range of absorption spectrum of ZnO/Cellulose nanostructures was measured from 200 nm until 800 nm. From the graph, the intensity of the absorbance is increasing with increasing the value of the cellulose. The results occurred where different of cellulose plays the main role in the formation of nanostructured that affecting the absorbance in the electromagnetic spectrum.

In the graph, the absorbance peak of ZnO/Cellulose is around 300 to 430 nm. The broad peak indicates that ZnO/Cellulose nanoparticles absorbs ultraviolet (UV) region and visible light region. The UV region covers the range of wavelength from 100-400 nm and divided into three bands. First band is Ultraviolet A (UVA) has 315 – 400 nm. Second band is Ultraviolet B (UVB) has 280 – 315 nm. And third band is Ultraviolet C (UVC)

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100 – 280 nm (WHO). While the visible region covers 380 to 423 nm wavelengths. As mentioned, ZnO/Cellulose nanoparticles that absorbs 300 to 400 nm lies between UVA and blue region in the visible spectrum. This information describes the presence of blueshift is observed and it is important to determine the direct band gap of ZnO/Cellulose.

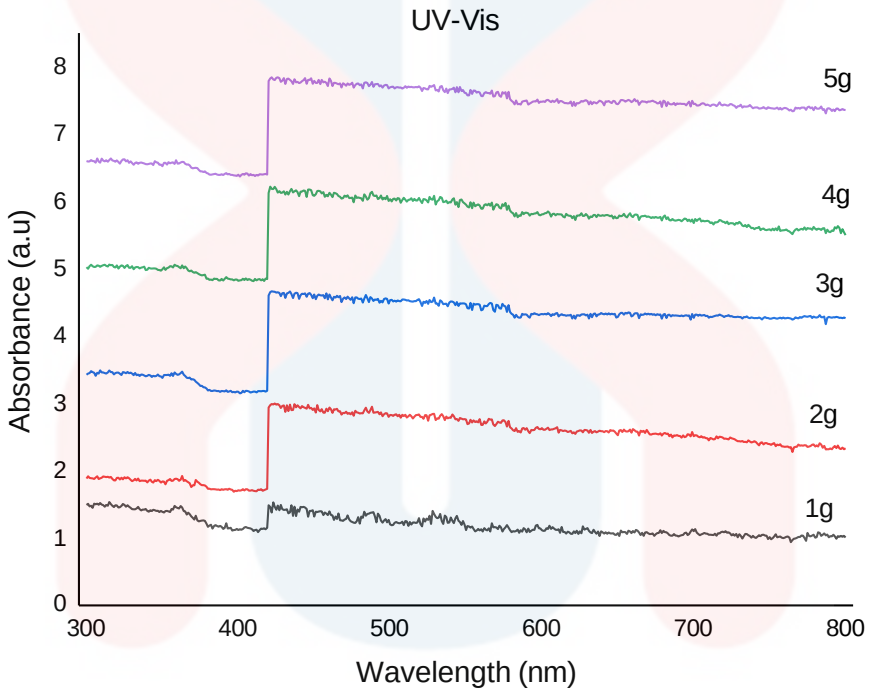

Figure 4.4 : The UV-Vis graph spectra of ZnO/Cellulose with different amounts of cellulose via hydrothermal method.

4.3.1 Band Gap Determination

The energy band gap of ZnO with different amounts of cellulose value was plotted using the Tauc graph. The energy band gap was calculated from the Tauc relation formula as stated in the Equation 4.2. The calculated data was then plotted in the graph of hv

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versus $(\alpha hv)^2$ through the absorption coefficient α which is related to the band gap Eg as $(\alpha hv)^2 = k\ (hv - Eg)$. By extrapolating a linear projectile towards the x-axis, the direct band gap of the each ZnO/Cellulose sample could be determined. The optical band gap (Eg) were found decrease in the band gap of ZnO/Cellulose with an increase in different amounts of cellulose.

$$(\alpha hv) \wedge 1/n = k\ (hv\text{-}Eg) \qquad \text{Equation 4.2}$$

This equation is derived from Tauc and David-Mott relation refer to Equation 4.2, where α is absorption, hv is photon energy, k is the energy independent constant and Eg is the optical band gap. The exponent n is the nature of transition for direct band gap is 2 whereas for indirect band gap is ½.

$$Eg = hv \qquad \text{Equation 4.3}$$

$$v = c/\lambda \qquad \text{Equation 4.4}$$

$$Eg = hc/\lambda \qquad \text{Equation 4.5}$$

Max Planck equation (4.3) was used for conversion of wavelength to energy. Equation (4.4) inserted into equation (4.3) and equation (4.5) exists. Next, $(\alpha hv)n$ equation is used for y-axis which α is for absorbance coefficient and hv is used for photonic energy. Alpha (α) can be calculated by Beer Lambert's law that showed in Equation 4.6 below.

$$I / Io = e^{-\alpha l}$$

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I stand for intensity transmitted light. Io stand for intensity of incident light. A stand for the absorption coefficient and l stand for the path length of light in which absorbance take place.

Figure 4.5 (a), 4.6 (a), 4.7 (a), 4.8 (a) and 4.9 (a) shows the UV – Vis graph absorbance versus wavelength for ZnO with different amounts of cellulose. Meanwhile, Figure 4.5 (b), 4.6 (b), 4.7 (b), 4.8 (b) and 4.9 (b) shows the graph of optical band gap of ZnO/Cellulose with extrapolated straight line. Based on the results, the extrapolated straight line indicates the energy band gap for 1 g, 2 g, 3 g, 4 g, and 5 g different amount of cellulose are shown accordingly, which is 2.38 eV.

The direct band gap of ZnO/Cellulose can be estimated from the graph of hv versus $(ahv)_2$ in Figure 4.5, 4.6, 4.7, 4.8, and 4.9 for different amount of cellulose (1, 2, 3, 4, and 5) g were shown above.

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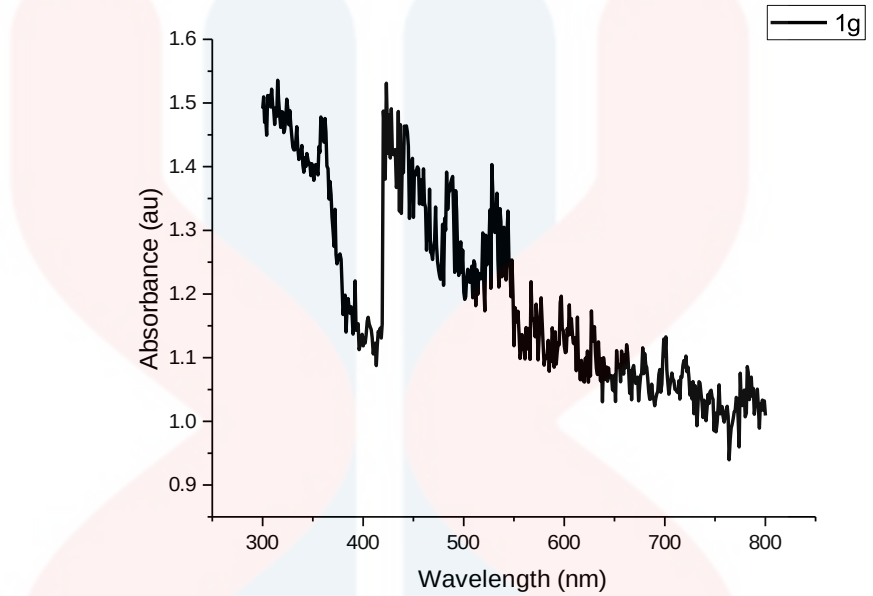
(a)



(b)

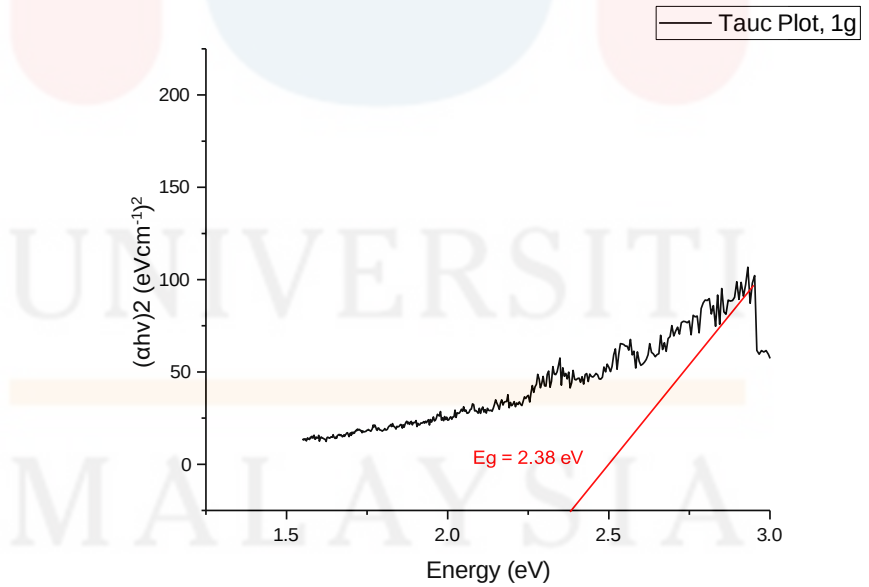


Figure 4.5 : (a) Absorption spectra of ZnO/Cellulose at 1 g of cellulose and (b) Optical band gap of ZnO/Cellulose at 1 g cellulose.

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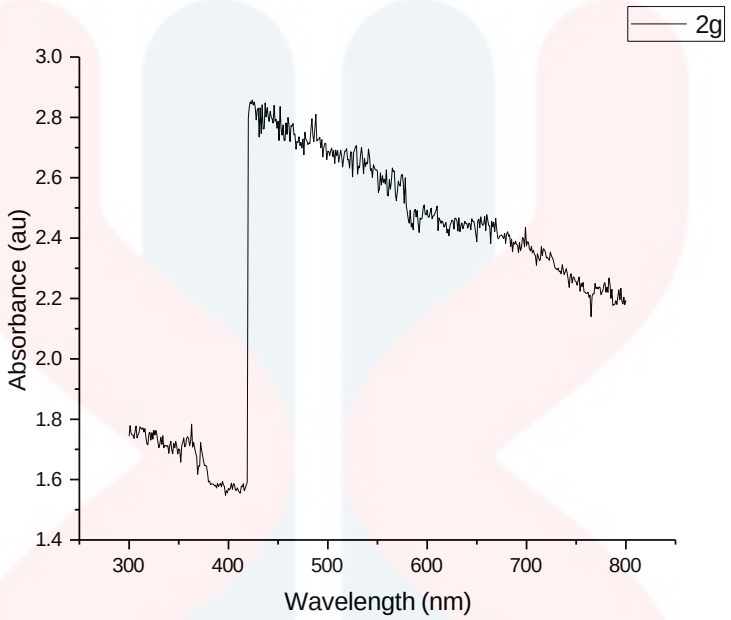
(a)



b)

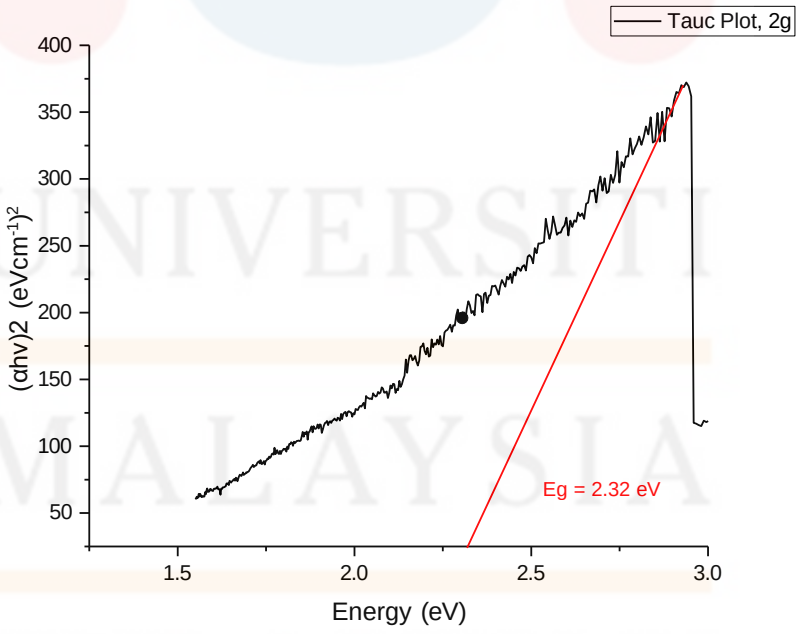


Figure 4.6 : (a) Absorption spectra of ZnO/Cellulose at 2 g of cellulose and (b) Optical band gap of ZnO/Cellulose at 2 g cellulose.

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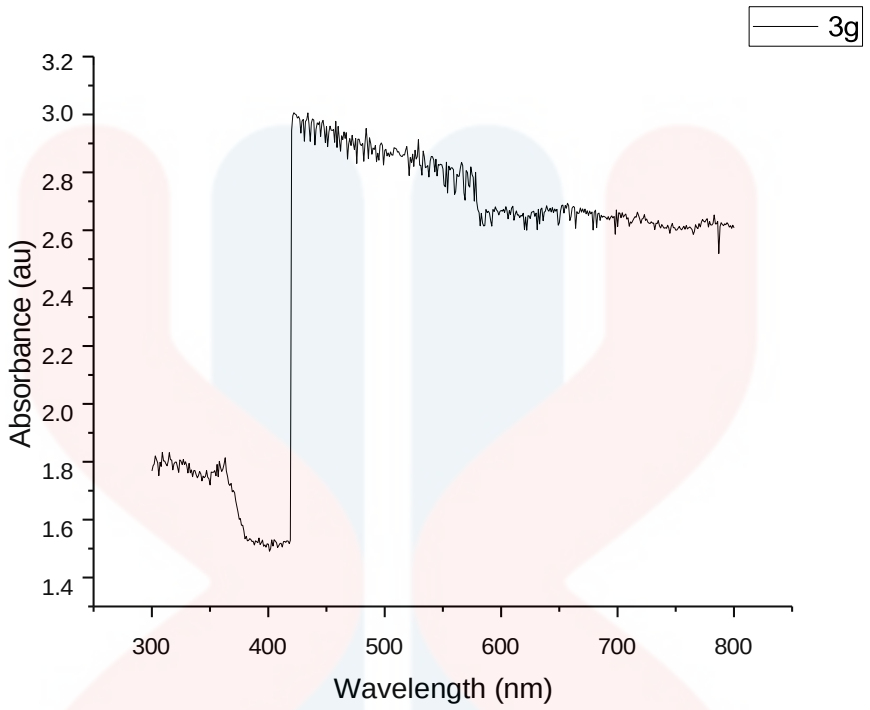
a)



b)

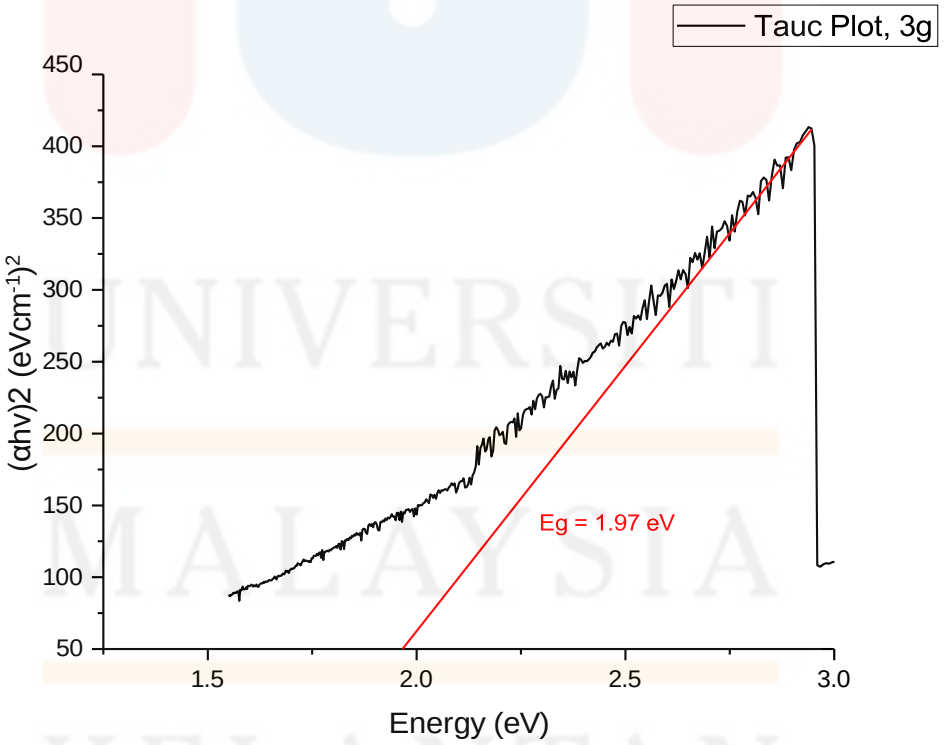


Figure 4.7 : (a) Absorption spectra of ZnO/Cellulose at 3 g of cellulose and (b) Optical band gap of ZnO/Cellulose at 3 g cellulose.

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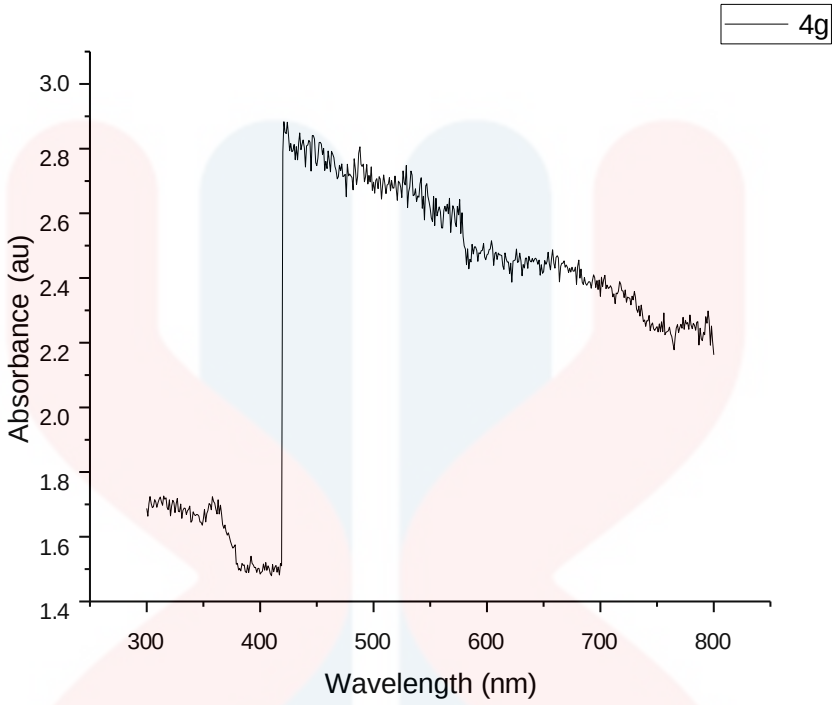
a)



b)

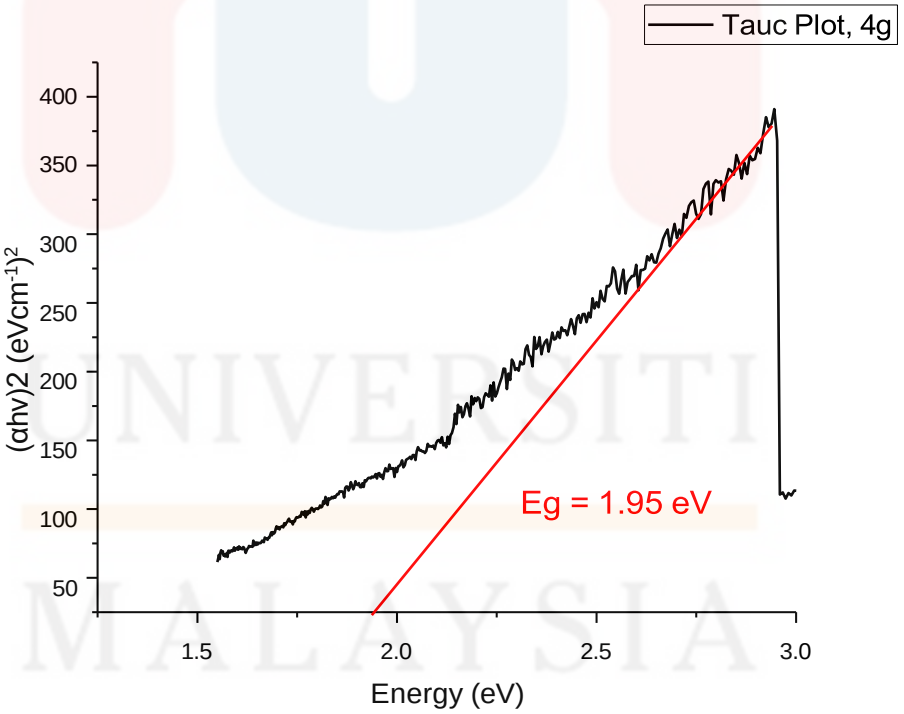


Figure 4.8 : (a) Absorption spectra of ZnO/Cellulose at 4 g of cellulose and (b) Optical band gap of ZnO/Cellulose at 4 g cellulose.

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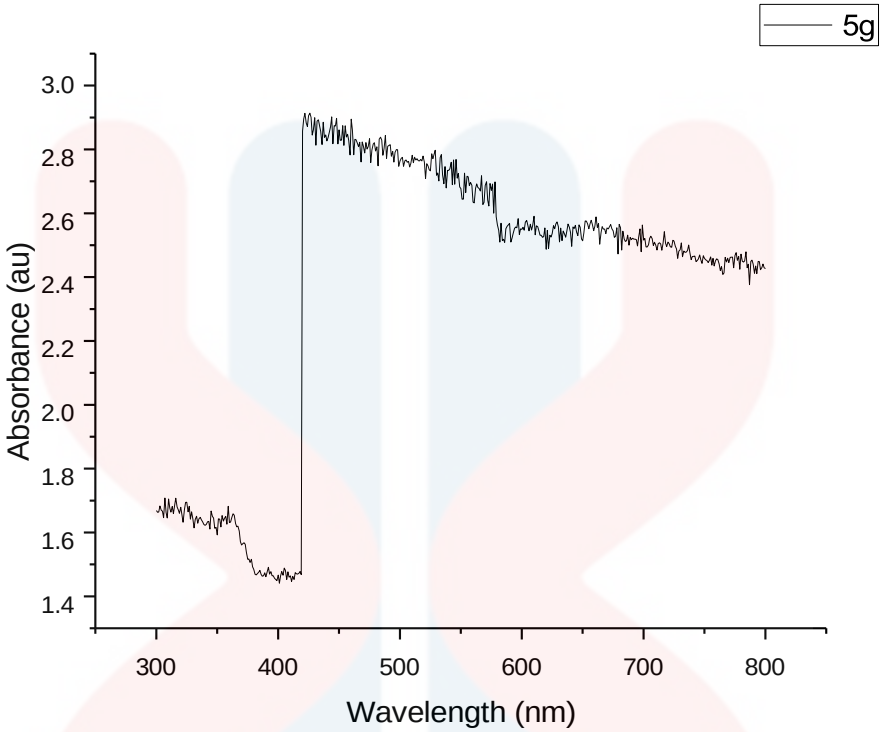
a)



b)

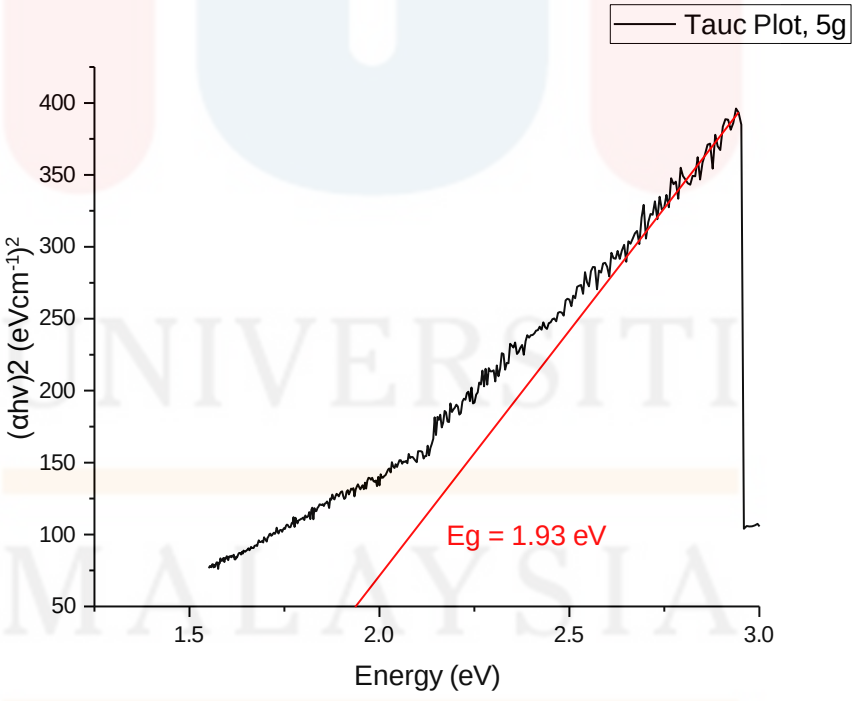


Figure 4.9 : (a) Absorption spectra of ZnO/Cellulose at 5 g of cellulose and (b) Optical band gap of ZnO/Cellulose at 5 g cellulose.

In the table 4.2, the energy band gap for different amounts of cellulose shown. The energy band gaps were decrease because of the different amounts of cellulose.

Table 4.2: The energy band gap for different amounts of cellulose

Different amount of cellulose (g)	Eg (eV)
1	2.38
2	2.32
3	1.97
4	1.95
5	1.93

In the Figure 4.10, the optical band gap ZnO/Cellulose with different amounts of cellulose were shown. The graph are decrease because the different amounts of cellulose were added.

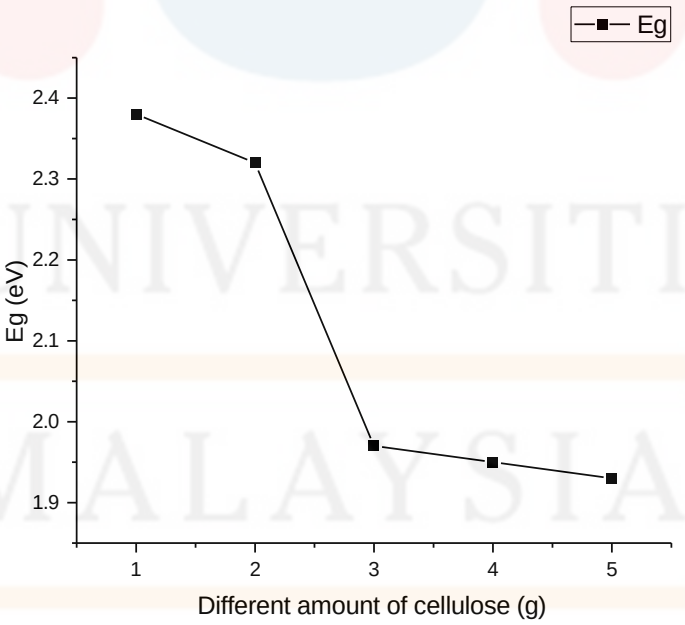


Figure 4.10 : The optical band gap of ZnO/Cellulose with different amounts of cellulose.

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4.4 Scanning Electron Microscopy (SEM)

Scanning Electron Microscope Image of the ZnO/Cellulose nanostructured synthesis at the different amounts of cellulose (1,2,3,4 and 5) g are presented in Figure 4.11. The morphology for 1 g is agglomerated particles. The shapes are irregular because it does not have the specific geometry. For the morphology in sample 2 g is agglomerated particles. The shape is irregular because it has unclear morphology. For the sample 3 g, the particles are agglomerated particles. It also has irregular shape but this morphology is clearer than sample 1 g and 2 g. For the sample 4 g, the particles also agglomerated particles. The shape is irregular because not specific shape is found. For the sample 5 g, the particles also have agglomerated particles. It also have irregular shape because not specific shape is found. From the 5 samples, it can see that the morphology were increased when the amount of the cellulose were increased.

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Figure 4.11 show the SEM image for ZnO/Cellulose. The particles in the images were changed and increase because of the increase amount of cellulose.

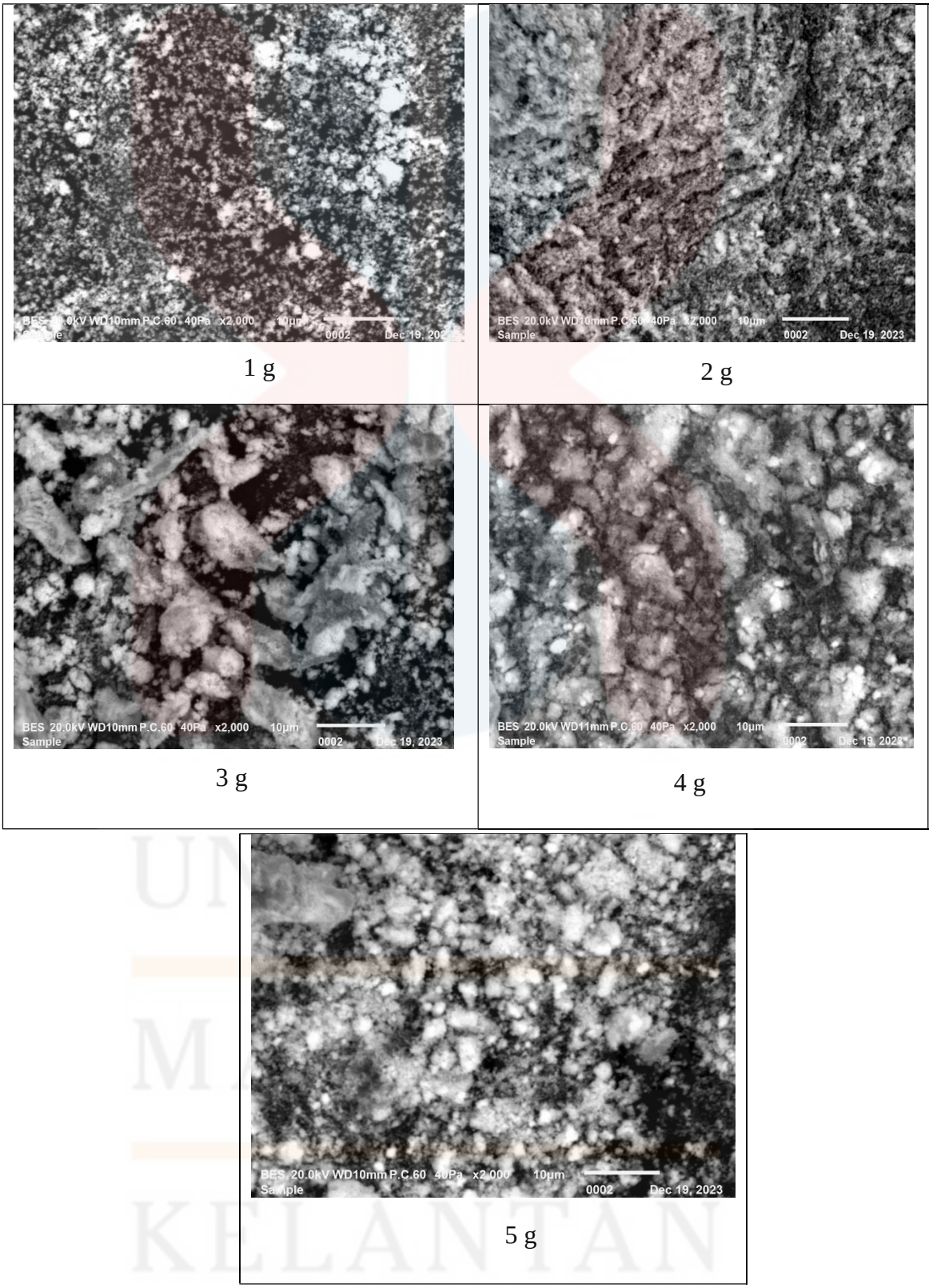

Figure 4.11: SEM image for (1,2,3,4 and 5) g sample

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

This research was conducted to study the morphological structure and the optical structure at different amount of cellulose. In this work, ZnO/Cellulose particles were successfully synthesized by using hydrothermal method and characterized by XRD, UV-Vis and SEM. It can be concluded that the increase amount of cellulose influences the optical and structural properties of ZnO/Cellulose particles.

In conclusion, the systematic preparation and detailed characterization of ZnO particles embedded within a cellulose matrix, employing the hydrothermal synthesis method at various cellulose concentrations, have provided a nuanced understanding of the resulting composite material. The investigation not only underscores the versatility and controllability of the hydrothermal approach but also elucidates the intricate interplay between cellulose content and the ensuing properties of the ZnO/cellulose composite.
The optimization of cellulose content emerges as a pivotal aspect of this study, unveiling its significant impact on the structural, morphological, and mechanical characteristics of the composite. The observed variations in crystalline structure and particle morphology underline the influence of cellulose in shaping the final architecture of the composite. The nuanced adjustments in cellulose concentration present a fine-tuning opportunity for tailoring the material to meet specific application requirements.

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The morphological characterization through advanced microscopy techniques has unveiled the uniform dispersion of ZnO particles within the cellulose matrix. This homogeneity is crucial for ensuring consistent material properties and is particularly promising for applications demanding a high degree of structural integrity. The discerned relationships between cellulose loading and mechanical properties, such as tensile strength and flexibility, provide valuable insights for designing materials with optimized mechanical performance. Furthermore, the thermal stability analysis sheds light on the composite's behavior under varying temperature conditions. This information is paramount for applications subjected to thermal stress, contributing to the broader understanding of the material's suitability in diverse environmental settings.

The potential applications of the ZnO/cellulose composite are diverse, spanning from flexible electronics to biomedical devices. The material's unique combination of properties, influenced by cellulose concentration, positions it as a versatile candidate for innovations in nanotechnology and materials science. The environmentally conscious consideration of cellulose, a renewable and abundant resource, aligns with the global push towards sustainable materials and processes. As a stepping stone in the exploration of ZnO/cellulose composites, this study lays the groundwork for future investigations. Further research could delve into specific applications, exploring functionalities such as electrical conductivity, optical properties, and biocompatibility in greater detail. Additionally, targeted modifications to either the ZnO particles or cellulose matrix could unlock new possibilities for enhanced performance and novel applications.

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5.2 Recommendations

For future research, the following experiments are suggested for future works:

1. Verify that the grain is clearly visible, and that the magnification is appropriate if using SEM for the characterization.
2. Ensure that the hydrothermal process is completed, and the sample is weighed.
3. One characterization method that can be used to determine the ideal sample weight and temperature is TGA process.
4. Before adding the sample, verify that the surface of Teflon is clean to prevent samples from being damaged.

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