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Characterization of ZnO with different pH values incorporate with cellulose on physical and optical properties using Hydrothermal method

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DECLARATION

I declare that this thesis entitled **"Characterization of ZnO with different pH values incorporate with cellulose on physical and optical properties using Hydrothermal method"** is the result of my own research except as cited in the references.

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Characterization of ZnO with different pH values incorporate with cellulose using hydrothermal Method

ABSTRACT

In this research, a hydrothermal method synthesis of zinc oxide (ZnO) nanoparticles added with cellulose using a hydrothermal method of various morphology while regulating the pH parameter. The process involved dissolving zinc cloride and sodium hydroxide in distilled water. For all sample, the mixture was heated at 180°C for 24 hours. By adjusting the amount of sodium hydroxide (NaOH), the precursor's pH was changed from 8,9,10,11and 12. After that, added cellulose at the solution. X-ray Diffraction (XRD), Ultraviolet-visible spectroscopy and Scanning Electron Microscopy (SEM) were used to evaluate the zinc oxide/cellulose nanocomposite. XRD to calculate the various crystal structure properties, such as lattice strain and crystallite size. Optical properties UV-vis analysis results show that zinc oxide/cellulose nanocomposite reveal ZnO absorption or reflectance peak between 300 to 400 nm. The ZnO/cellulose powder with various pH value were observed at magnificent 500x, 1000x,2000x for SEM characterized of the supplied sample. the successful synthesis of zinc oxide nanoparticles added with cellulose, coupled with comprehensive characterization using various techniques, demonstrates the success of the project in achieving its objectives.

Keywords: ZnO/cellulose, Hydrothermal, Optical Properties

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Pencirian ZnO dengan nilai pH berbeza ditambah dengan selulosa menggunakan kaedah hidrothermal

ABSTRAK

Dalam penyelidikan ini, kaedah hidrotermal digunakan untuk mensintesis nanopartikel zink oksida (ZnO) yang ditambah dengan selulosa dengan pelbagai morfologi sambil mengawal parameter pH. Proses ini melibatkan larutan zink klorida dan natrium hidroksida dalam air suling. Bagi semua sampel, campuran dipanaskan pada suhu 180°C selama 24 jam. Dengan menyesuaikan jumlah natrium hidroksida (NaOH), pH pendahulu diubah dari 8, 9, 10, 11, dan 12. Selepas itu, selulosa ditambah ke dalam larutan. Penentuan X-ray (XRD), spektroskopi ultraviolet-terlihat, dan mikroskopi imbas elektron (SEM) digunakan untuk menilai nanokomposit zink oksida/selulosa. XRD digunakan untuk mengira pelbagai sifat struktur kristal, seperti regangan kisi dan saiz kristalit. Hasil analisis optik UV-vis menunjukkan bahawa nanokomposit ZnO/selulosa menunjukkan puncak serapan atau pantulan ZnO antara 300 hingga 400 nm. Serbuk ZnO/selulosa dengan nilai pH yang berbeza diperhatikan pada pembesaran 500x, 1000x, 2000x untuk pengekalan SEM sampel yang disediakan. Penghasilan berjaya nanopartikel zink oksida yang ditambah dengan selulosa, yang disertakan dengan pencirian komprehensif menggunakan pelbagai teknik, menunjukkan kejayaan projek dalam mencapai objektifnya.

Kata Kunci: ZnO/selulosa, Hidrotermal, Sifat Optik

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LIST OF ABBREVIATIONS (optional)

Zn	Zinc
O	Oxide
Zno	Zinc Oxide
pH	Potential Hydrogen
NaOH	Sodium Hydroxide
$ZnCL_2$	Zinc Chloride
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction
UV-Vis	Ultraviolet–visible spectroscopy

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CHAPTER 1

INTRODUCTION

1.1 Background of study

The atoms of zinc (Zn) and oxygen (O) combine to form the chemical zinc oxide (ZnO). It is a white, powdery chemical that naturally exists as the mineral zincite, but it may also be made synthetically using a number of different processes for a variety of uses. A complex carbohydrate called cellulose, also known as polysaccharide, is used as a structural element in the cell walls of several bacteria, algae, and plants. It is the most prevalent organic substance on Earth and a significant portion of plant biomass. The chemical structure of cellulose, which is composed of long chains of connected glucose molecules, offers it special qualities including great tensile strength, insolubility in water, and resistance to digestion by most mammals.

Short-wavelength ultraviolet (UV) photodetectors are significant components with numerous industrial, commercial, and military uses. Examples of applications for visible-blind UV photodetectors include ozone layer monitoring, flame detection, and space communications. Si-based optical photodetectors are still used for UV light detection at this time. These devices are sensitive to visible and infrared light, but their UV sensitivity is poor because to low 1.2 eV room temperature bandgap energy. High-performance solid-state photodetectors with UV region sensitivity may now be made thanks to the development of optoelectronic devices built

on broad direct bandgap materials. Another material with a broad direct bandgap and UV sensitivity is ZnO. ZnO is a potential photonic material for uses in light-emitting diodes (LEDs), laser diodes (LDs), and UV photodetectors due to its high exciton-binding energy of 60 meV and broad bandgap energy of 3.37 eV at ambient temperature. (Young et al., 2006)

By employing water as a solvent in a confined system at a certain temperature and pressure, the hydrothermal technique may replicate the development of crystals during the natural mineralization process. The characteristics of water, including its vapour pressure, density, surface tension, viscosity, and ionic product, will be significantly altered in a hydrothermal environment. The hydrothermal process was employed to create crystals as early as 1882. It is obvious that it can lower the temperature at which systems react as well as produce highly crystalline products with a restricted size distribution, high purity, as well as minimal aggregation. It is challenging to examine the growing process of materials because of the response in the closed system. (Meng et al., 2016)

ZnO is a typical oxide semiconductor that has undergone the most extensive and thorough research for fundamental optoelectronic devices like LEDs and TFTs. The effective stimulated emission7 and laser action8 of excitons in the UV region at room temperature upon optical pumping of ZnO thin films served as the impetus for the research activities focused on LEDs. The ability of certain oxide semiconductors, such as ZnO, to create working TFTs grown at low temperatures (300°C)9 or even at room temperature is advantageous for TFT operations.6,10 In terms of the kind of carriers that make these two devices operational, they differ. Given that TFTs are unipolar devices, it is simpler for n-type oxides to create high-mobility n-channel TFTs.

For the past ten years, producing stable, high-quality p-type ZnO has been a difficult task. The effective creation of p-type ZnO has been documented in a number of papers. However, it is

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believed that a large number of these claims are based on inaccurate Hall effect measurements. Conductivity inhomogeneity dis thin films frequently results in an unexpected current flow route, which can lead to a false Hall signature.2 Adding acceptor elements within the host ZnO often results in the production of such unknown semiconducting behaviour with positive Hall coefficients, although the ZnO was substantially n-type due to the abundance of donor-type defects (Kamiya & Kawasaki, 2008).

1.2 Problem statement

ZnO is a straight wide-band-gap semiconductor with a high exciton binding energy. Zinc oxide nanoparticles have a propensity to clump together due to their vast surface area and high surface energy. The unique properties of nanoscale cellulose include a high capacity for absorbing metallic particles, a high aspect ratio, good water dissolvability, high mechanical strength, and negligible heat degrading behaviour. Therefore, to enhance agglomeration, cellulose will be added to zinc oxide with different pH. Due to the low process temperature and ease with which the particle size can be changed, the hydrothermal approach is a feasible alternative synthetic method. The hydrothermal process provides a variety of advantages over other growth techniques, including the use of straightforward instruments, catalyst-free development, cheap cost, consistent production over a vast area, environmental friendliness, and reduced risk. This approach is well suited for plastic electronics and microelectronics because to the low reaction temperatures.

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

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1.3 Expected output

In this project, hydrothermal will be prepare of zinc oxide with different pH added cellulose using the same temperature by using hydrothermal method for 180° C. zinc oxide It would be expected that would significantly enhance the films' morphology, crystallinity, and optical properties using hydrothermal method.

1.4 Objective

1. To prepare ZnO/cellulose composite at different pH values using the hydrothermal method.
2. To investigate the effects of ZnO/cellulose composite on the morphology, crystallinity and optical properties using hydrothermal methods.

1.5 Scope of study

The research focuses on the Zinc oxide added with cellulose to synthesized using a hydrothermal technique by adjusting the different pH which is 8,9,10,11 and 12 pH. The zinc oxide will be prepared for the characterization process. Cellulose functions as an adsorbent and

substrate. This research will evaluate the effect on the morphology, crystallinity, and optical properties using hydrothermal methods such as X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and UV-VIS for sample preparation. This research also looked at the cellulose effect with different pH values. with the different value, we can study the suitable pH for zinc oxide added with cellulose.

1.6 Significant of study

The study focus on ZnO/cellulose composite on morphology, crystallinity, and optical properties using hydrothermal method. the purpose of this study is to find solution to find the hydrothermal technique for morphology, crystalinity, and optical characteristics. in this scenario, ZnO is a promising low-cost and readily available natural material that absorb UV radiation, less poisonous, has high resistance and has high electron mobility. Insights into the manufacture and modification of composite materials may be gained by examining the impact of adding cellulose to zinc oxide with NaOH. This study might aid in the creation of new materials with improved qualities including increased optical, electrical, or mechanical conductivity. Then, for ph value, must keep the pH within the ideal range of 6-9. to avoid all these negative consequences. That can minimise the adverse effects of hydrolysis by meticulously regulating the pH in this range. This means that inside the composite framework, the ZnO particles can maintain the desired characteristics and functions.

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CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Zinc oxide powder has traditionally been used as a white pigment and as an additive to rubber. While it has largely been displaced as a pigment in paints, its usage in rubber remains very important. However, the myriad of other practical uses of ZnO are sometimes overlooked, and reviews in the recent scientific literature tend to emphasize high technology applications that do not yet have any commercial reality. (Moezzi, A., McDonagh, A. M., & Cortie, M. B. (2012). FE-SEM, FT-IR, thermogravimetric analysis, XRD, and UV-visible spectroscopy were used to characterise the gelatin/ZnO NPs combination films. One of the most prevalent biopolymers, cellulose, has been utilised extensively as a reinforcing component in fiber-thermoplastic composite materials and is thought to provide a nearly limitless source of raw materials for the growing market for biodegradable and biocompatible goods. The linear chains of glucose units that make up cellulose's chemical structure are connected by -1,4-glycosidic linkages. A cellulose microfibril is a long, thread-like crystalline structure that is created when cellulose glucan chains consolidate through hydrogen bonding and van der Waals forces (Betancourt & Osorio-Aguilar, 2022)

It is crucial to employ a transparent electrode made of a thin layer of the transparent conducting oxide (TCO) semiconductor for the majority of optoelectronic devices, such as flat panel displays.

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For the majority of transparent electrode applications, tin-doped indium oxide (usually known as indiumtin-oxide, or ITO) thin films formed by magnetron sputtering (MSP) have been in use, however there are several reports on various TCO semiconductors as well as deposition techniques. Because indium, the primary component of ITO, is expensive and scarce, a steady supply of ITO for the lately rising market for optoelectronic devices may be challenging to accomplish. (Minami, 2005)

2.2 Zinc oxide

Due to their wide variety of applications, including transparent electrodes in displays and solar systems, transparent conducting oxide layers have been the subject of substantial research. Due to its promising features, ZnO thin films have recently generated a lot of attention as transparent conductors. ZnO is an n-type wide band-gap semiconductor (Eg=3.3 eV), and the zinc excess at the interstitial location is primarily responsible for its electrical conductivity. Through suitable doping techniques or thermal treatment with hydrogen, its electrical characteristics might be changed over a number of decades. Hydrogen plasma doesn't affect the stability of ZnO thin films. Many different thin film deposition processes, including pulsed laser deposition RF magnetron sputtering, chemical vapour deposition, and spray pyrolysis, have been used to create ZnO thin films. (Lee et al., 2003)

ZnO is a significant semiconductor that has found extensive use in photocatalysis, solar cells, and environmental cleanup. Its photocatalytic performance may be enhanced by doping

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and morphological control, among other strategies. In this study, a simple two-step calcination technique was created to create porous, carbon(C)-doped cubic ZnO from zeolite imidazolate frameworks (ZIF-8). In comparison to one-step pyrolysis, the two-step calcination method successfully inserts C doping into the ZnO lattice while also maintaining the cubic shape with linked ZnO nanoparticles and porous structure. With C doping, there is a high charge-separation efficiency and advantages in charge transfer, optical absorption, and mass transfer during the photoreaction. As a result of the fast charge transfer across the border, the ZnO particle connection is advantageous for photoperformance. Second, ZnO has a low efficiency of charge separation because to its straight band gap. In order to overcome this, nonmetal doping of ZnO is a successful technique to prevent charge-pair recombination and to achieve the visible-light response. (Pan et al., 2016)

On glass slide substrates, excellent quality zinc oxide (ZnO) nanostructures have been produced utilising the modified chemical bath deposition (M-CBD) approach at a low temperature. Air bubbles will be pumped into an aqueous growth solution using the M-CBD approach. The glass substrates were grown with a ZnO seed layer using the RF magnetron sputtering technique. Using field emission-scanning electron microscopy (FE-SEM), energy dispersive analysis (EDX), X-ray diffraction (XRD), and UV-Visible Spectrometer, researchers have examined the effects of various pH values of the aqueous growth solution on the morphology, elemental chemical composition, crystal structural, and optical properties of ZnO nanostructures. (Abdulrahman et al., 2021)

The tetrahedrally coordinated O 2+ and Zn2+ are alternately stacked along the c-axis to form a multitude of alternating planes that make up ZnO's structure. ZnO nanostructures can be produced from gaseous phase or in solution. The techniques for gas phase synthesis are pricy and challenging. Typically, the solution step of synthesis is carried out in water. Due to its ease of use and bearable growth conditions, the hydrothermal method of generating ZnO

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nanostructures has become quite popular. The growth temperatures are lower than the boiling point of water because synthesis takes place in aqueous solutions. Amongst the different methods of synthesis of ZnO nanostructures, the hydrothermal method is attractive for its simplicity and environment friendly conditions. This review summarizes the conditions leading to the growth of different ZnO nanostructures using hydrothermal technique. Doping of ZnO nanostructures through hydrothermal method are also highlighted. (Baruah & Dutta, 2009)

2.3 Cellulose

Cellulose is one of its most abundant renewable resources. A flexible raw material, cellulose may be used to create a wide range of products that are useful in many industries. But because of how hard it is to dissolve, its full potential is not realised. In order to produce value-added cellulosic products like regenerated fibres, filaments, and films, cellulose dissolution is a must. as well as creating materials based on functional cellulose in a homogeneous environment. Because of the complete availability of hydroxyl (OH) groups, the ability to control the distribution and degree of substitution (DS), minimal chain degradation, and high yield due to minimal reagent consumption by side reactions, functionalizing cellulose in the dissolved state is desirable. However, cellulose is a refractory polymer arranged in a hierarchical structure within plant cell walls, making it the load-bearing structure in nature that is resistant to breakdown. The inability of cellulose to dissolve is attributed to its rigid chains and dense packing caused by many hydrogen bonds. Robust hydrogen bonding both inside and between molecules, as well as negative interactions, influence the solubility of cellulose (Väisänen et al., 2021).

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For the solvent to influence both the crystalline and amorphous areas of cellulose during dissolution, it must permeate into the cellulose's structure. The solvent must break up the strong intermolecular contacts (hydrogen bonding and hydrophobic) between the crystallised cellulose molecules, and the cellulose chains within amorphous areas should be apart from one another. For a solvent to be successful with cellulose, it must thus possess high diffusivity, aggression towards destroying the crystalline network, and the capacity to untangle chains. Numerous solvents possess appropriated diffusivity levels and have the ability to permeate and swell cellulose, but not dissolve it (Ghasemi et al., 2017).

Figure 1: Cellulose powder

2.4 Sodium Hydroxide

Sodium hydroxide has a strong base and a very alkaline aqueous solution. It is useful for adjusting the pH of liquids and neutralising acids. Sodium hydroxide is involved in many different chemical processes, such as those that neutralise acids to produce salts and water. There are a lot of industrial uses for sodium hydroxide. It is employed in the manufacturing of diverse chemicals, paper, textiles, soaps, and detergents. Aluminium manufacture, petroleum

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refining, and the synthesis of different organic chemicals all use it. Sodium hydroxide is a prominent component in several drain cleaners because of its capacity to dissolve grease and proteins. Pretzel preparation is one of the food industries uses of sodium hydroxide.Sodium hydroxide finds application in water treatment procedures to modulate the pH of water and counteract acidic compounds. Its utility extends to laboratory settings where it serves diverse functions such as titrations and as a potent base in chemical reactions. (S. Ramachandra Rao, 2006).

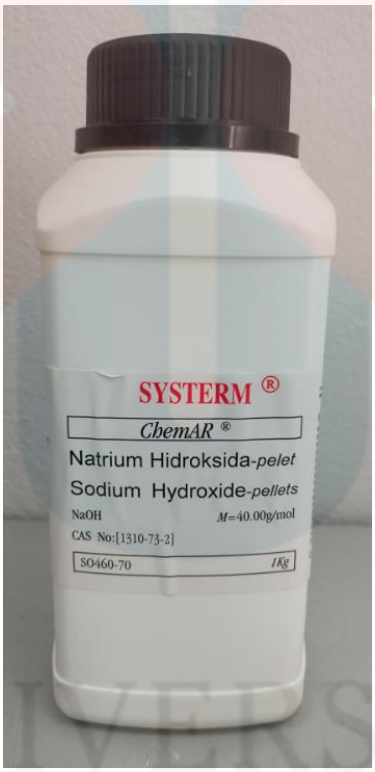

Figure 2: Sodium Hydroxide

2.5 ZnO/cellulose composite

ZnO is regarded as one of the most promising candidates for use in UV sensing devices due to its broad band gap of 3.37 eV and a high exciton binding power of 60 meV. Many authors have documented the physical and chemical production of nanostructured ZnO for use in UV sensors

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in recent years. Due to their ease of growth on the numerous conducting and non-conducting substrates necessary for the manufacture of sensing devices, one dimensional nanostructures of ZnO, such as nanorods and nanowires, have been the subject of substantial investigation. As one of the most prevalent polymers found in nature, cellulose is one of the biopolymers that has been widely employed in research because it is a renewable, affordable, biodegradable, and common biopolymer. It has previously been reported to create ZnO-cellulose nanocomposites, which are made of evenly coated cellulose fibres and ZnO nanorods with extremely narrow size dispersion.

The regulated development of nanostructured ZnO occurs on cellulose fibre micro templates, and the agglomeration events are significantly reduced. Due to cellulose's excellent binding abilities, ZnO-cellulose nanocomposite powder may be pressed and rolled into a variety of forms, including pellets and paper films without interfering with ZnO's shown optical or electrical capabilities. (Sahoo et al., 2017)

There are currently two different ways to make nano-ZnO/cellulose composite materials. The first technique involves dispersing ZnO nanomaterials in an aqueous solution of cellulose (or a derivative thereof) and then precipitating the result to create ZnO/regenerated cellulose composite materials. As a result, cellulose is extremely difficult to dissolve, and ZnO nanomaterials are often scattered throughout cellulose, which is unfavourable for light absorption and hence has low photocatalytic activity. The second approach involves soaking cellulose in a Zn2+-containing solution, followed by heat treatment to produce a composite of cellulose loaded with ZnO. Due to the high degree of crystallinity of cellulose, which prevents Zn2+ from penetrating into the crystalline regions, hydroxyl groups cannot be fully utilised, making it very challenging to assemble ZnO on the surface of the cellulose. This is because cellulose contains many hydroxyl groups, which can promote the formation of nano-ZnO. (X. Li et al., 2016)

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can be applied to support nano-ZnO, enhance catalytic activity, and enable nano-ZnO recovery. The impact of nano-ZnO/cellulose composites on the photocatalytic performance of natural pigments has been the subject of several studies in recent years, the majority of which have concentrated on the loading of nano-ZnO onto cellulose nanofibers. The amount of free hydroxyl on a cellulose fibre is proportional to its size; the bigger the specific surface area of the fibre, the more free hydroxyl is present on the surface. This promotes greater ZnO nucleation on cellulose by improving the interaction between zinc ions and hydroxyl groups. Furthermore, the diameters of the cellulose fibres had an impact on the mechanical characteristics and 3D network structure of the fibres. This leads to variations in ZnO dispersion and fixation on cellulose fibre, which further impacts the rate of recycling and photocatalysis. The impacts of fibre size on ZnO nanoparticle shape and photocatalytic activity will be examined in this work (Qiu et al., 2022).

2.6 Hydrothermal method

Hydrothermal synthesis is the process of creating chemicals in a contained, heated solution at room temperature and pressure. The word "hydrothermal" refers to a temperature and water pressure regime and was first used in earth science in the nineteenth century. The hydrothermal technique involves using an aqueous solution as a reaction system in a specialised closed reaction vessel to pressurise the reaction system (or the vapour pressure created by itself) and create a high-temperature, high-pressure reaction environment. Under normal circumstances, a chemical that is weakly soluble or insoluble is dissolved and then recrystallized by the process. In order to create oxide ceramics, hydrothermal synthesis relies on the forced hydrolysis of the reactants. By putting the reagents in a sealed container and heating the system

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to the reaction temperatures, this is accomplished at high pressures and moderate temperatures (200 °C). A metal hydroxide (for example, NaOH) is used as a mineralizer, while metal alkoxides or metal salts are used as the source of metal ions. The solvent is often water. Nucleation is followed by particle growth, much like in precipitation systems, to produce a powder with a certain distribution of particle sizes. (Xu et al., 2018b)

Generally speaking, the hydrothermal process develops in a closed system at a high autogeneous pressure. The benefit of the closed system under high pressure allows for a significant reduction in the temperature needed to prepare ZnO powder. one particles that have exceptional sinterability may be produced, and the amount of reactive species is reduced as a result of their increased reactivity. (Lu & Yeh, 2000)

2.7 pH value

The chemical parameter that is most frequently determined in our world is the pH value. The gold standard and by far the most used instrument is the glass pH electrode. Since pure oxide's charge and surface potential mostly depend on pH, it is occasionally possible to change the slurry's pH so that every particle has the same charge polarity. A stable suspension will form if the surface of the particular is high enough to prevent the formation of hard agglomerates. The presence of impurities like oxo ions or multivalent (and notably transition) metal ions can also affect a mineral's surface charge. Although there was a significant intergroup variance in polymorphonuclear leukocyte density for normal skin cells, despite the higher ZnO concentration. When developing materials through a chemical pathway, the pH value has a significant influence on the final product. (Steinegger et al., 2020).

The measurement of how basic or acidic aqueous or other liquid solutions are. The phrase,

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which is frequently used in chemistry, biology, and agronomy, converts the hydrogen ion concentration, which typically ranges between 1 and 1014 gram-equivalents per litre, into numbers between 0 and 14. The hydrogen ion concentration in pure water, which has a pH of 7, is 107 gram-equivalents per litre, making it neutral (neither acidic nor alkaline). A solution with a pH below 7 is referred to as acidic, and one with a pH over 7 is referred to as basic, or alkaline. (Britannica et al.,2020)

Additionally, the influence strength of the preferred orientation plane (002) as well as average transmittance spectrum were also impacted by higher pH levels. Meanwhile the quantum effect of size has caused the absorption band edge to move to a lower energy area. Additionally, it was discovered that the crystal size varied between 36.30 nm and 84.33 nm and that the pH ranged from 6.7 to 12. The highest findings for high aspect ratio, structural, and optical features were from ZnO synthesised at pH 6.7. The nanorod structure containing small diameters, a larger size, and a bigger energy band gap value were correspondingly shown by ZnO development at this pH level. (Abdulrahman et al., 2021)

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CHAPTER 3

MATERIAL AND METHOD

3.1 Materials

The chemicals solution use in this study is Zinc chloride ($ZnCL_2$), Sodium Hydroxide (NaOH) added cellulose from Sigma- Aldrich. Zinc chloride ($ZnCL_2$). The institution possesses it. This substance's identification number is 7646-85-7, and the quantity is 1000 g. Sodium Hydroxide (NaOH) Cellulose is the product's chemical denomination, while R&M stands as the brand designation, The academic institution holds ownership of it. This substance's identifier number is 9004-34-6, and the weight is 500 g.are both inert aqueous solution. Zinc hydroxide ($Zn(OH)_2$) is produced as a white precipitate when the two solution are combined. When the solution is combined, Distilled water was used throughout the experiment. Then, added the cellulose The chemical and reagent used in synthesis of ZnO with different pH added with cellulose is stated in details in Table 1.

Table 1: List of material and chemical of synthesis ZnO

No	Material chemical
1.	Zinc chloride ($ZnCL_2$)
2.	Sodium Hydroxide (NaOH)
3.	Cellulose
4.	Distilled water

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3.2 Method

3.2.1 Hydrothermal Method

To achieve homogeneity, the combined solution was agiated for 2 hour at room temperature. The solution was transferred to a Teflon- lined steel autoclave in an oven at 180°C for 6 hours using hydrothermal method and then cooled to room temperature. The precipitate is filtered for followed with drying for 2 hours. Accordingly, the powder is examined in term of their structural, physical and optical properties.

Figure 3: Hydrothermal metho

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3.2.2 Preparation and characterize the material

3.2.2.1 Zinc Cloride

Zinc chloride (ZnCL) and sodium hydroxide were dissolved in 50ml of distilled water and stirred using a magnet during the preparation of ZnO utilising the hydrothermal process (figure 2). The ZnO were produced using zinc chloride (ZnCL) and sodium hydroxide. 6.1g (1M) of zinc oxide (figure 2) was initially dissolved in 50ml of water. Then, 4.0g (2M) of sodium hydroxide (NaOH) (figure 3) was dissolved in 50 ml of water. NaOH was gradually added to each solution until a pH of 8, 9, 10, 11, or 12 is obtained (figure 5).

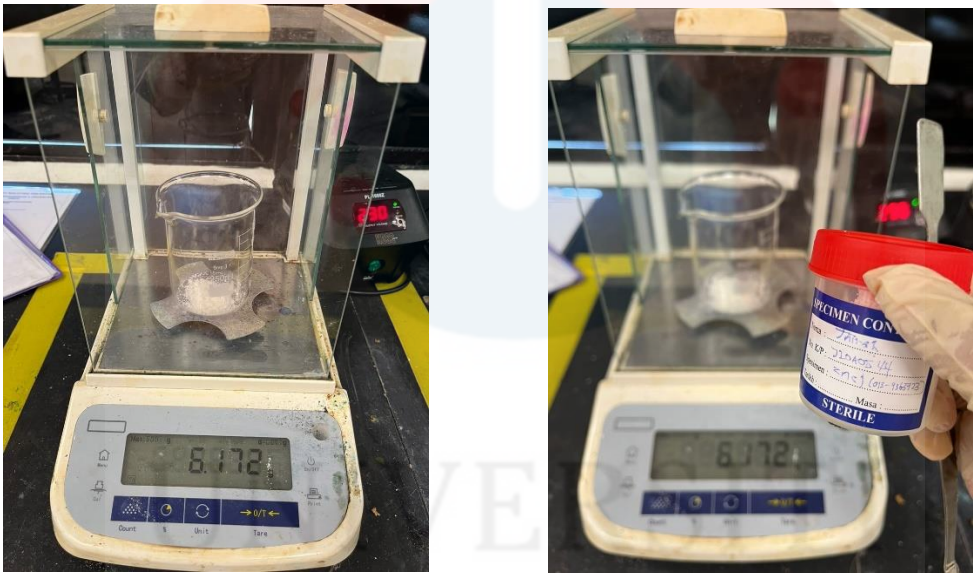

Figure 4 : Measured ZnCl

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Figure 5: Measure NaOH

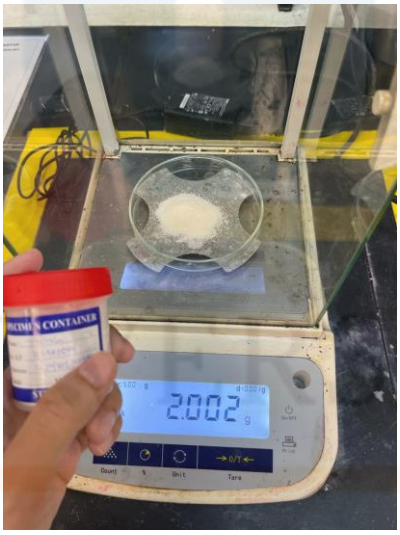

Figure 6: Measure cellulose

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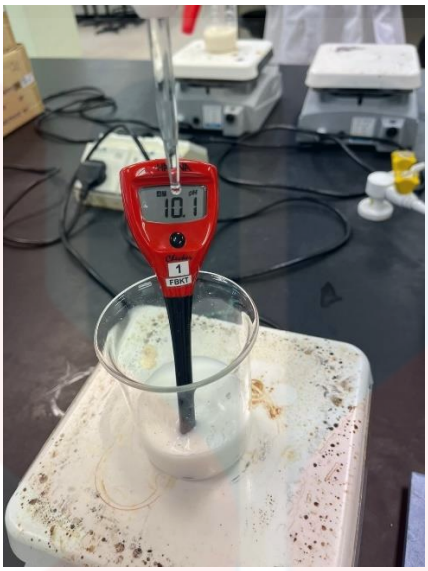
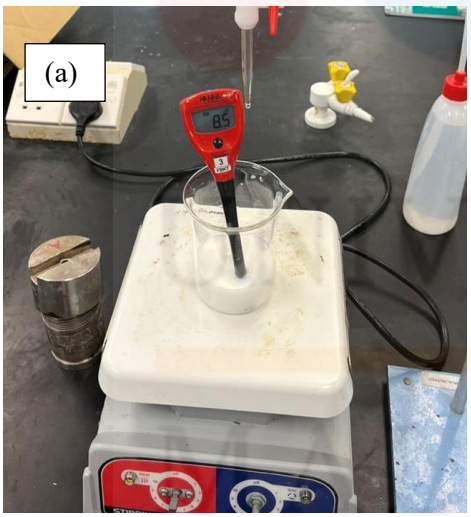
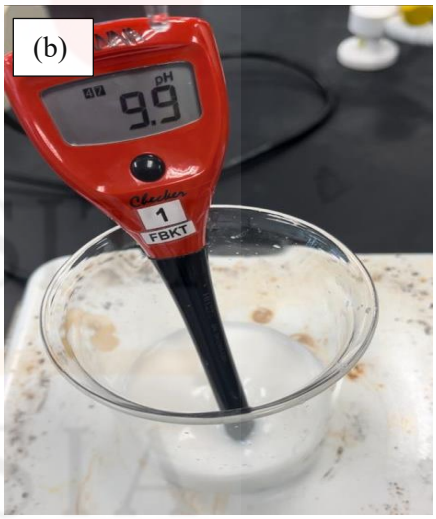


Figure 7: Dropping NaOH





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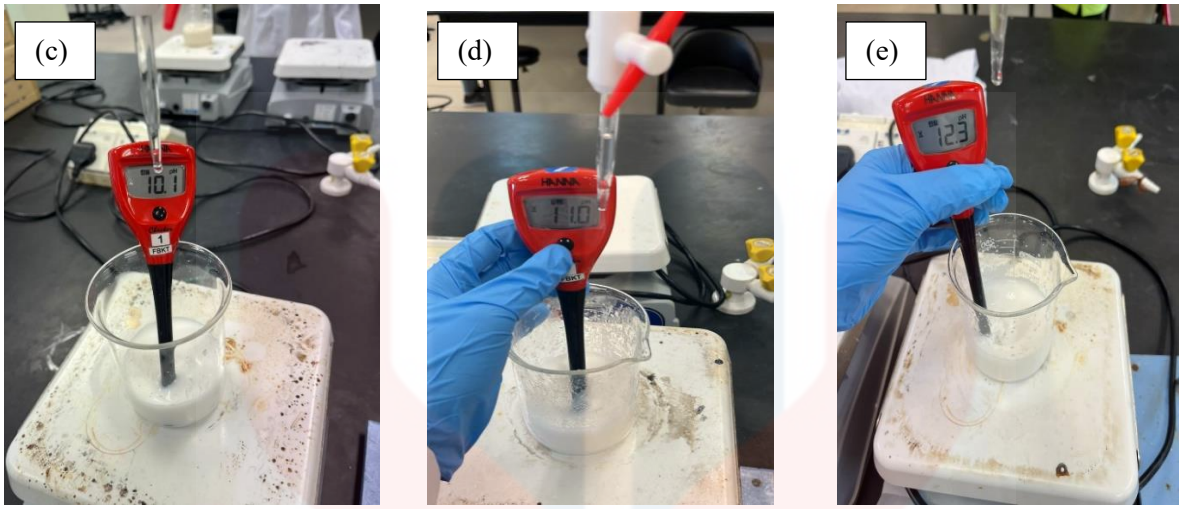

Figure 8: The value of (a) pH8, (b) pH9, (c) pH10, (d) pH11, (e) pH12 reached.

Figure 9: Washed with distilled water using filter paper

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3.2.2 Cellulose

The earlier combination received 2g of cellulose (figure 4), which was then stirred for 10 minutes at room temperature. ZnO particles are applied to the surface of the cellulose during this procedure. To achieve homogeneity, the combined solution for 15 minutes at room temperature. After washed several times, the precipitated is filtered for 2 hours showed in.

3.3 Characterization of zinc oxide/cellulose composite

Many methods may be used to characterise zinc oxide (ZnO) in order to learn everything there is to know about its chemical, physical, and optical characteristics. ZnO has a hexagonal wurtzite crystal structure, according to the XRD pattern from the synthetic ZnO NPs. The deposited films have polycrystalline ZnO phase, according to X-ray diffraction (XRD) investigations. In order to understand ZnO's lattice parameters and the existence of any impurities or secondary phases, X-ray diffraction (XRD) analysis is used to assess the crystal structure as well as phase purity of ZnO.

Then, next characterization which is SEM. Make sure you have a ZnO sample that is representative ready for SEM analysis. The sample can be in any form that is suitable for SEM measurements, including powder and thin films. The SEM instrument should be set up in accordance with the manufacturer's instructions. Set the equipment up for calibration and get any extras, such the sample holder, ready.

A representative sample is made and placed in the UV-Vis spectrophotometer to characterise ZnO. Setting up and calibrating the device ensures that the proper measurement parameters are used. To account for any background influences, a blank measurement using a reference material is carried out.

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CHAPTER 4

RESULT AND DISCUSSION

4.1 INTRODUCTION

The findings from the research that was done are presented in this chapter. The sample's components were added with Zinc Oxide and Cellulose used hydrothermal to got pH value of 8, 9, 10, 11 and 12. Used of by using X-Ray Diffaction (XRD), UV-VIS Spectroscopy and Scanning Electron Microscopy (SEM). These sample were create using a hydrothermal technique and each sample underwent at different pH. The procedure time for hydrothermal was altered to pH value 8,9,10,11 and 12. The characterize were done to investigate the physical and optical characteristics of ZnO nanostructure. X-ray diffraction (XRD) was used to determine the several of crystal structure crystal properties included crystallite size and lattice strain. UV-Vis spectroscopy was used to determine the sample's band gap, and scanning electron microscopy (SEM). It was capable of achieving nanoscale magnifications, which was beyond the capabilities of conventional light microscopes. SEM produced three-dimensional pictures that made it possible to examine the topography and structure of a sample in great detail. It was able to observe features that were not on the same focus plane as optical microscopes because of its significantly greater depth of field.

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4.2 X-Ray diffraction analysis (XRD) pattern

XRD analysis is important to identify the main crystalline phase in ZnO/Cellulose nanocomposite that stacking XRD that shows the difference in peak is observed. The XRD of the ZnO/cellulose composite at the 2θ of 30-70°. The figure 8 shows the ZnO/Cellulose collected XRD pattern at different pH value.

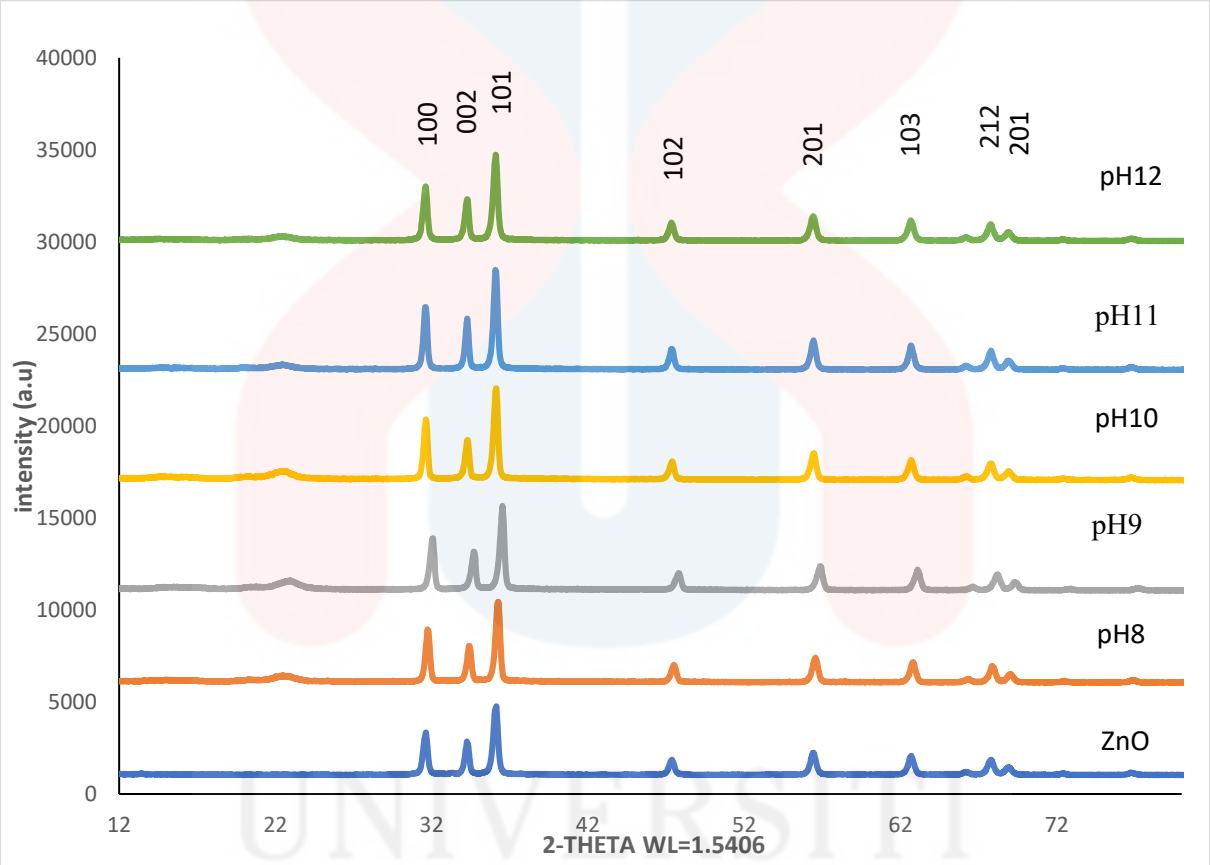

Figure 8: XRD pettern of ZnO/Cellulose nanocomposite prepared at different pH using hydrothermal method.

The many atomic planes inside the crystal lattice are represented by the peaks in the XRD pattern. The quantity of atoms in every layer determines the peak's intensity. The fact that the pattern of XRD results varies with pH suggests that ZnO's crystal structure is impacted by pH. The ZnO powder is in a very alkaline environment with a pH of 12. ZnO powder may dissolve in this environment and produce Zn(OH)42- ions. Following this, the Zn(OH)42- ions can re-

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precipitate as ZnO particles; however, the crystal structure of the particles differs from the initial ZnO particles. For this reason, ZnO powder at pH 12 exhibits a distinct XRD pattern than powder at pH 10 and pH 8. In this examination, there were no modifications seen in the cellulose structure. We may infer that this production procedure does not change the majority of cellulose particles. however, typical impure phase maxima pH value of 8,9,10,11 and 12 show were observed at 22° because presence of cellulose.

The synthesized ZnO nanoparticle the average was calculated using Debye-Scherrer formula:

$$D = \frac{k\lambda}{\beta \text{Cos}\theta}$$

Equation 4.1

The pure hexagonal wurtzite crystallite structure of ZnO/Cellulose with the lattice parameter of a a=3.250 A and c= 5.206 A. The synthetic zinc oxide's XRD pattern has wide peaks at 31.9, 34.5, 36.3, 56.7, and 62.9nm which correspond to typical zinc oxide structural values. The presence of a noticeable widening of the diffraction peaks indicates that the materials being synthesised are in the nanometer range. Scherrer's equation has been used to calculate the average particle size from the full width at half maximum (FWHM) of the diffraction peaks. This is expressed as t = 0.9 tλ/(B cos θ), where B is the full width at half maximum and λ is the x-ray wavelength. (Mohan & Renjanadevi, 2016).

In the realm of nanomaterials, the existence of nanocrystallites resulted in observable peak broadening. Nevertheless, precision in quantifying the associated peak broadening became challenging once the crystallite size exceeded a predetermined maximum limit. This situation posed a noteworthy constraint, imposing a ceiling on the calculable crystallite size within the analytical framework. The crystallite size for pure ZnO nanostructure is 22.66 nm. According to the pH value 8,10,11,12, shows ZnO/cellulose has crystallite size that sequentially 23.37, 24.52, 25.30 and 25.64 nm (Table 2).

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sample	Lattice parameter (Å)			a/b ratio	c/a ratio	Crystallite size (D), nm
	a	b	c			
Pure ZnO	3.2495	3.2495	5.2069	1.000	1.6024	22.66
pH 8	3.2495	3.2495	5.2069	1.000	1.6024	23.37
pH 9	3.2495	3.2495	5.2069	1.000	1.6024	23.67
pH 10	3.2495	3.2495	5.2069	1.000	1.6024	24.52
pH 11	3.2495	3.2495	5.2069	1.000	1.6024	25.30
pH 12	3.2495	3.2495	5.2069	1.000	1.6024	25.64

Table 2: the ZnO unit cell characteristics with different value of pH

However, variations in the pH of the ZnO/cellulose compositeThe amount of hydroxide ions (OH-) in the solution increased significantly when the pH was raised to 8,9,10, 11, and 12. As a result, the negative electrical charge on the outer layer of ZnO nanoparticles was amplified by this surge. The increased negative surface charge caused the conductive ZnO particles to repel each other more strongly, which affected the interparticle interactions in the composite. According the figure 10, the hexagonal wurtzite structure of ZnO has lattice constant of 3.249 and 5.206 for all sample.

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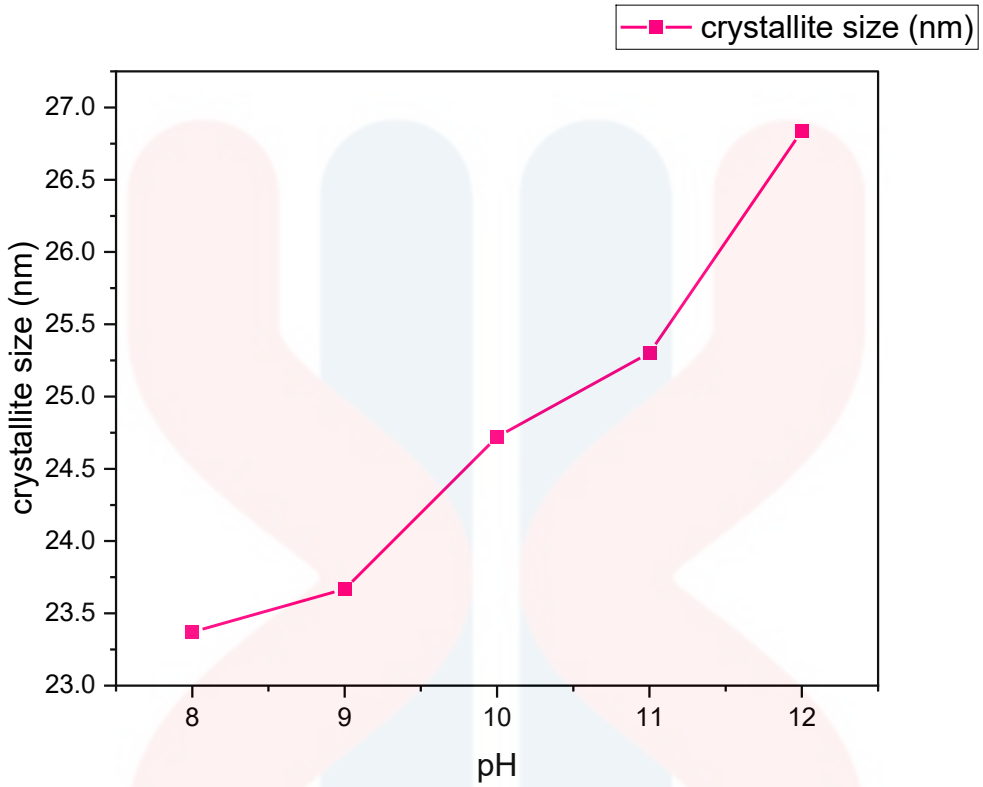

Figure 9: The XRD crystallite size versus with different pH.

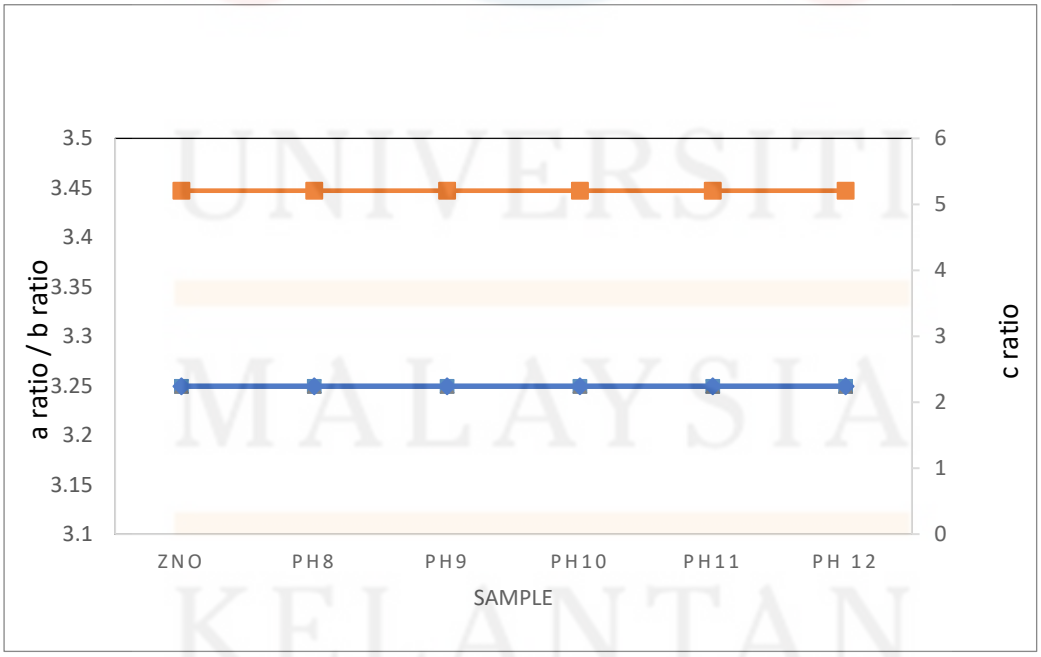

Figure 10: The XRD lattice parameter versus different pH

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4.2 UV-Vis spectroscopy analysis

Ultraviolet-visible spectroscopy give the value of absorbance regarding the electronic and energy band gap of the sample on different time of sythesis. Using a UV-Vis spectrograph, photoctalyst between 300 to 800 nm. It was utilised in this experiment to track the sythesis of ZnO/Cellulose nanocomposite. Figure 11 shows of ZnO nanostructure with pure ZnO and different pH value from 8,9,10,11 and 12. From the figure 16, the intensity of the absorbance is increase with different pH value. In the figure 12, the strong absorbance band peak at about around 400- 450nm. The broad peak indicates that ZnO nanoparticle absorb ultrabiolet (UV) region and visible light region.

The UV-Vis spectrum (Figure 11) reveals key insights into the ZnO/Cellulose nanocomposite's properties. The prominent peak at approximately 370 nm signifies light absorption by ZnO/Cellulose nanocomposite nanoparticles, characteristic of their band gap transition. This peak intensity hints at a moderate ZnO/Cellulose nanocomposite concentration within the composite. Additional, broader peaks around 420 nm likely stem from π-π* transitions in the cellulose matrix.and 500 nm likely stem from π-π* transitions in the cellulose matrix.

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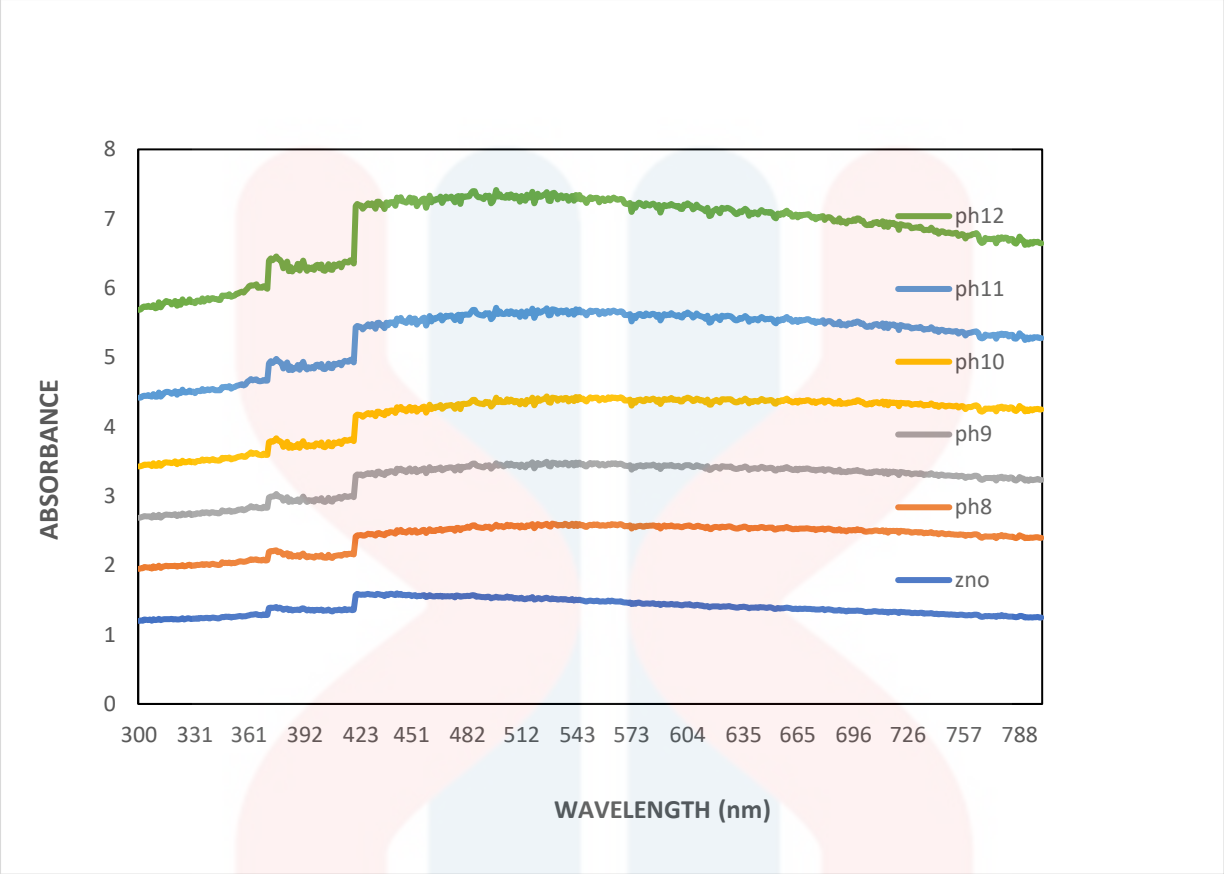

Figure 11: UV-Vis spectroscopy spectrum of ZnO/Cellulose nanocomposite

4.3 Band Gap Determination

The energy band gap of ZnO at various pH values was graphically represented using the Tauc plot. Calculation of the energy band gap was carried out in accordance with the Tauc relation formula, as expressed in Equation 4.2. The values derived from the Tauc relation formula were graphically depicted as hν versus $(\alpha h\nu)^2$. Through the absorption coffined α which is related to the band gap E_g as $\alpha h\nu = A(h\nu - Eg)n$ by using origin software. Through the extrapolation of a linear projection towards the x-axis, the direct band gap of each ZnO sample was determined.

It was observed that the optical band gap (E_g) is found to be pH dependent and there is an increase in the band ZnO an increase in pH value.

$$\alpha h\nu = A(h\nu - Eg)n$$

Equation 4.2

The equation was divided by Taus and Devic-Mott relation, as referred to in Equation 4.3. In this equation, *a* represented absorption, hν signified photon energy, A was an energy-independent constant, and Eg denoted the optical band gap. The variable *n* characterized the nature of the transition, taking the value of 2 for direct band gap materials and varying for indirect band gap materials.

Beer lambart's law, which is illustrated in equation (4.3), can be used to determined alpha (a) as follows:

$$A = \varepsilon Lc$$

Equation 4.3

The relationship between route length (l), concentration (c), molar extinction coefficient (ε), and absorbance (A) which is The molar extinction factor (ε), the concentration (c) of the solution, the path length (l) of light through the solution measured in centimetres, and the absorbance (A) were multiplied.

The UV- Vis graph of absorbance versus wavelength of pure ZnO and ZnO/Cellulose with different pH value is shown in figure 17 (a), 18 (a), 19 (a), 20(a), 21(a),22(a). In figure 17(b), 18(b), 19(b), 20(b), 21(b) and 22(b) illustrate the extrapolated straight line graph optical band gap of ZnO and ZnO/cellulose with different pH value. The energy band gap for ZnO is 3.30 eV while ZnO/cellulose for pH value of 8,9,10,11 and 12 are display corresponding which is

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2.11 eV, 2.26 eV, 2.34 eV, 2.79 eV, 2.90 eV based on the extraploted straight line's conclusion. This is because of the cellulose in the nanocomposite forming states within the band gap where electrons may be stimulated, the cellulose works to decrease the ZnO's band gap.

As a result, surface atoms exhibited a lower coordination number and reduced atomic interaction, leading to an elevation in the energy level of the highest valence band and a simultaneous reduction in the energy level of the lowest unoccupied conduction band. This phenomenon ultimately led to an augmentation of the band gap (Chand et al., 2012).

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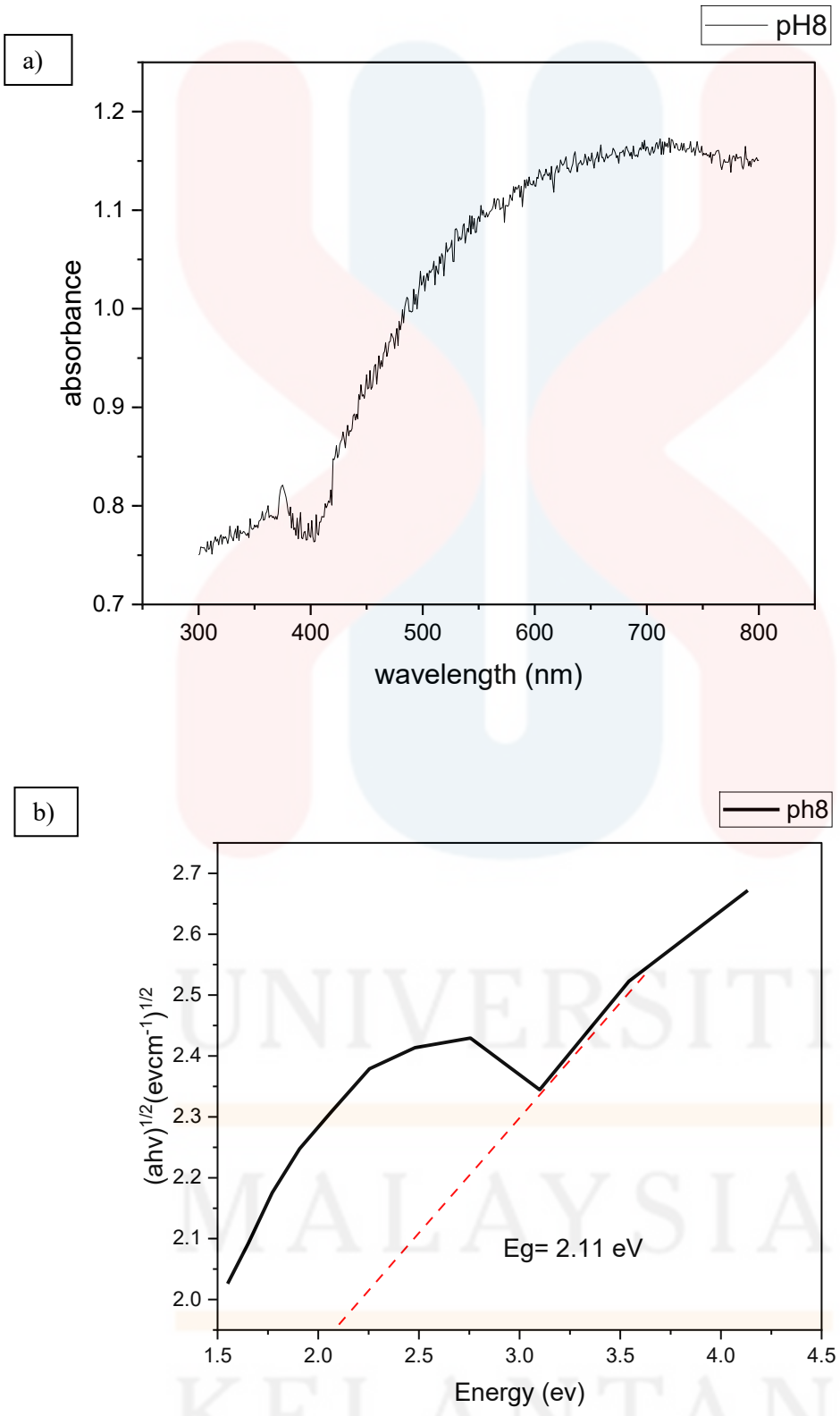

Figure 13: (a) Absorption spectra of ZnO/Cellulose nanocomposite at pH 8 (b) optical band gap of ZnO/cellulose at pH 8

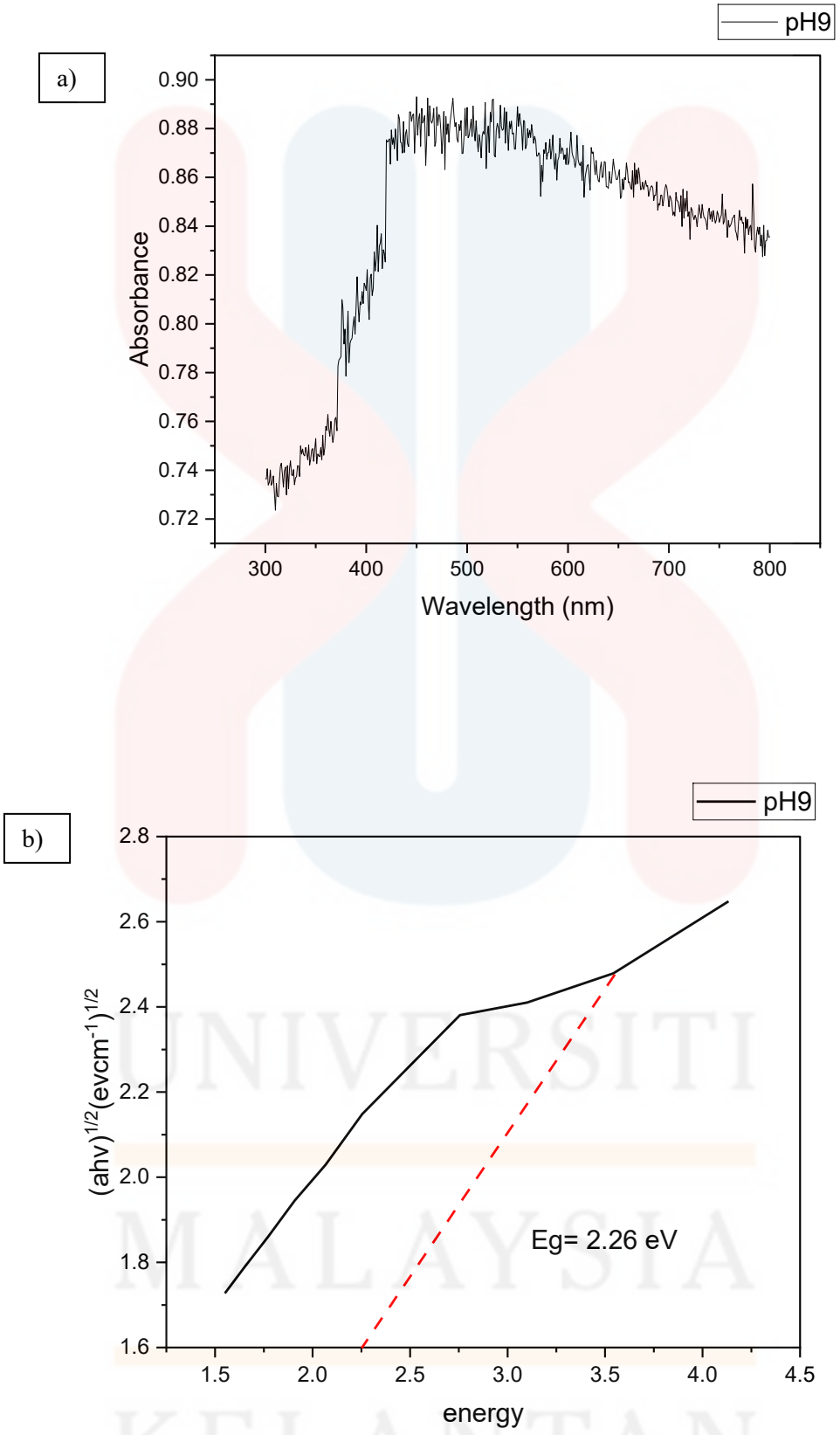

Figure 14: (a) Absorption spectra of ZnO/Cellulose nanocomposite at pH 9 (b) optical band gap of ZnO/cellulose at pH 9

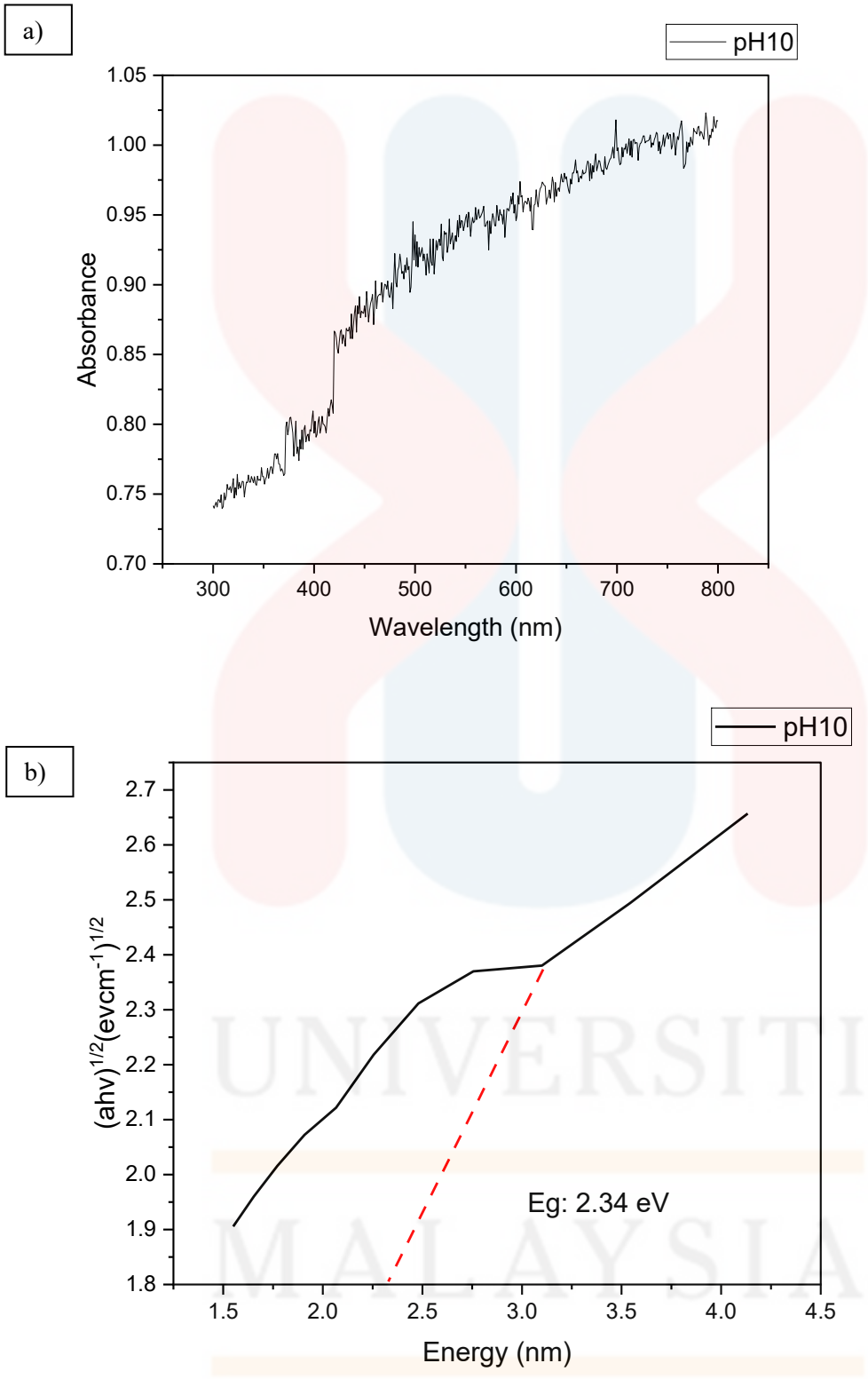

Figure 15: (a) Absorption spectra of ZnO/Cellulose nanocomposite at pH 10 (b) optical band gap of ZnO/cellulose at pH 10

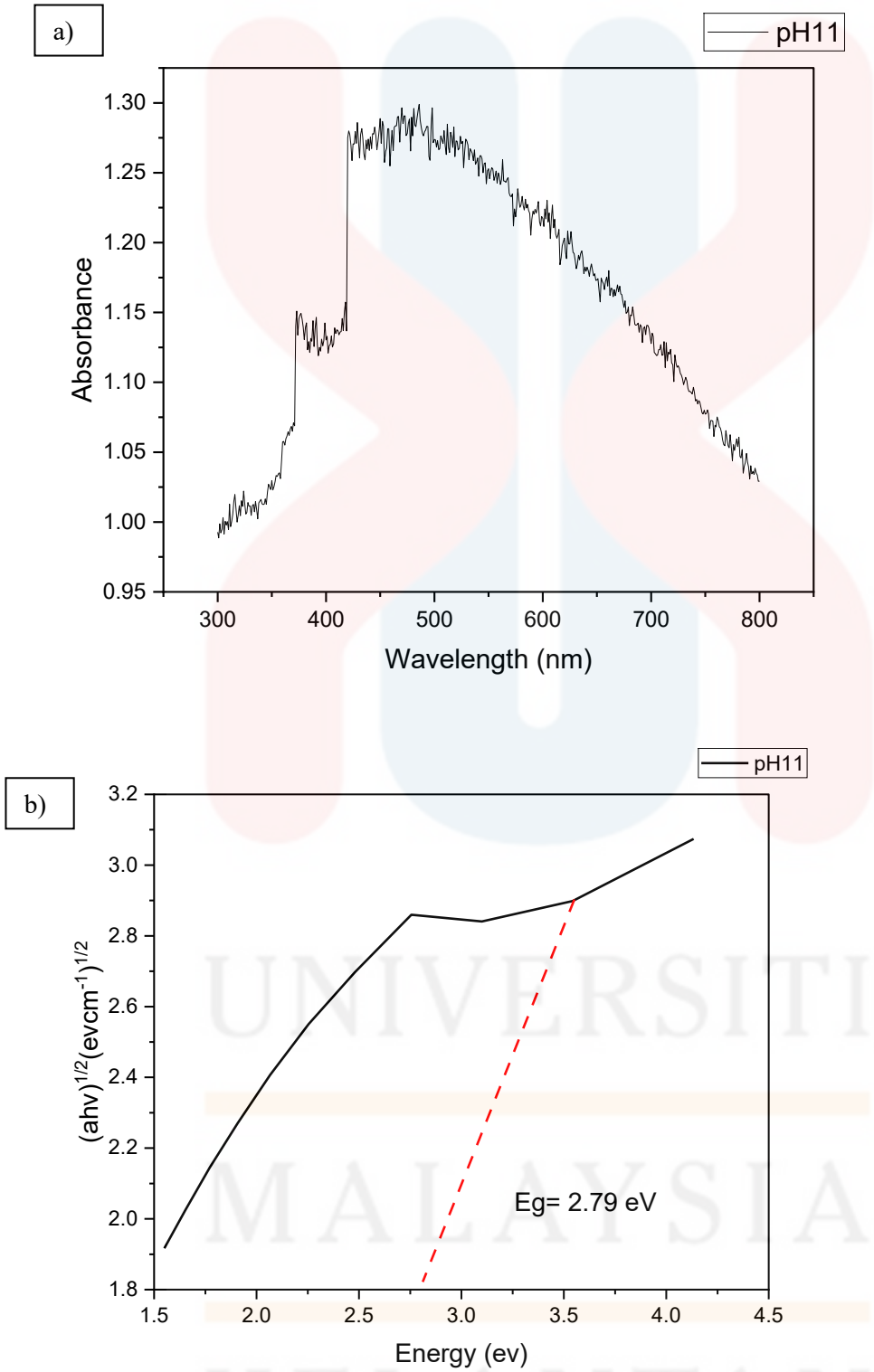

Figure 16: (a) Absorption spectra of ZnO/Cellulose nanocomposite at pH 11 (b) optical band gap of ZnO/cellulose at pH 11

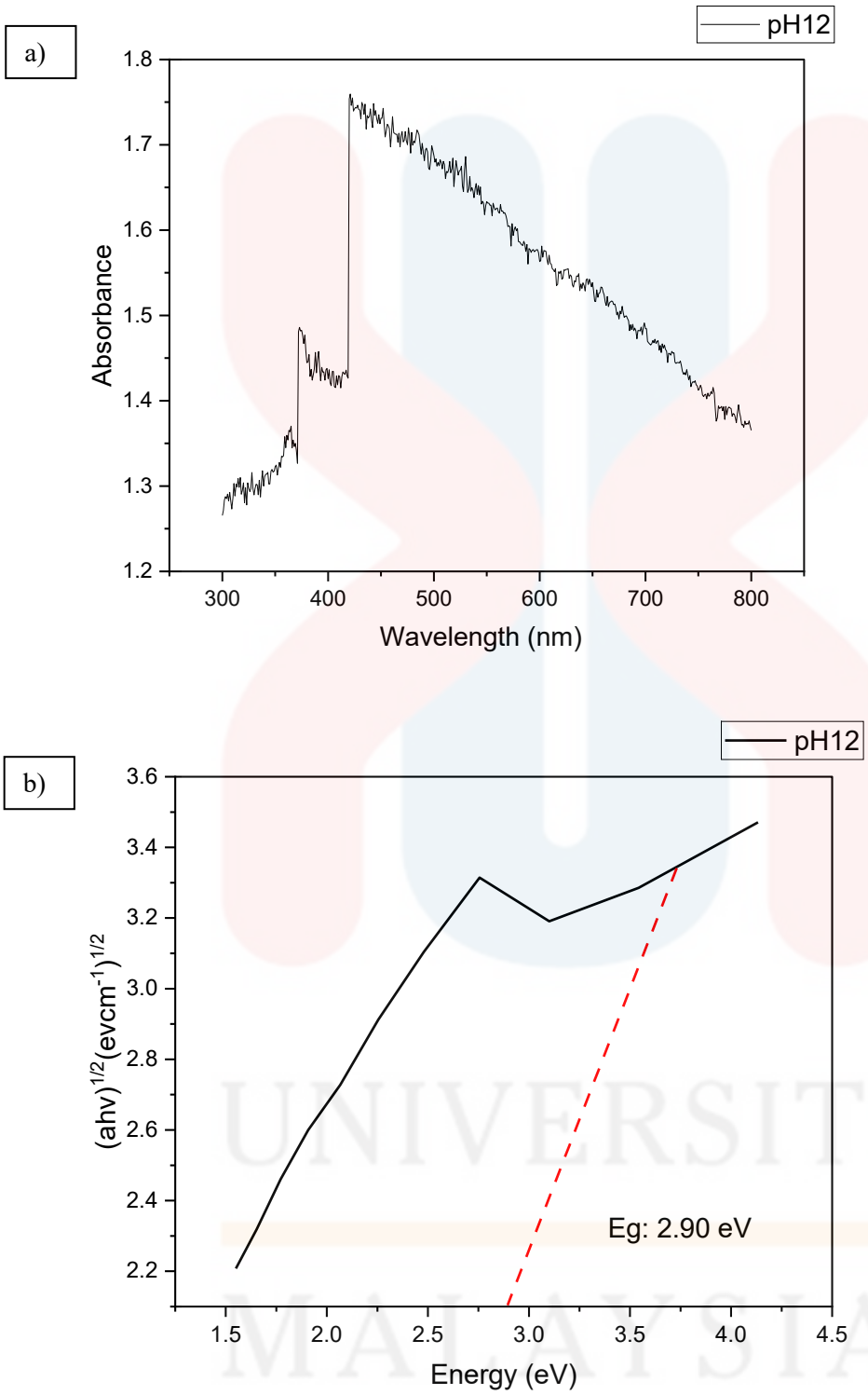

Figure 17: (a) Absorption spectra of ZnO/Cellulose nanocomposite at pH 12 (b) optical band gap of ZnO/cellulose at pH 12

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Ph of ZnO	E_g (eV)
Pure ZnO	3.30 eV
8	2.11 eV
9	2.26 eV
10	2.34 eV
11	2.79 eV
12	2.90 eV

Table 3: The uv-vis energy band gap for pure ZnO and different pH ZnO/Cellulose nanocomposite

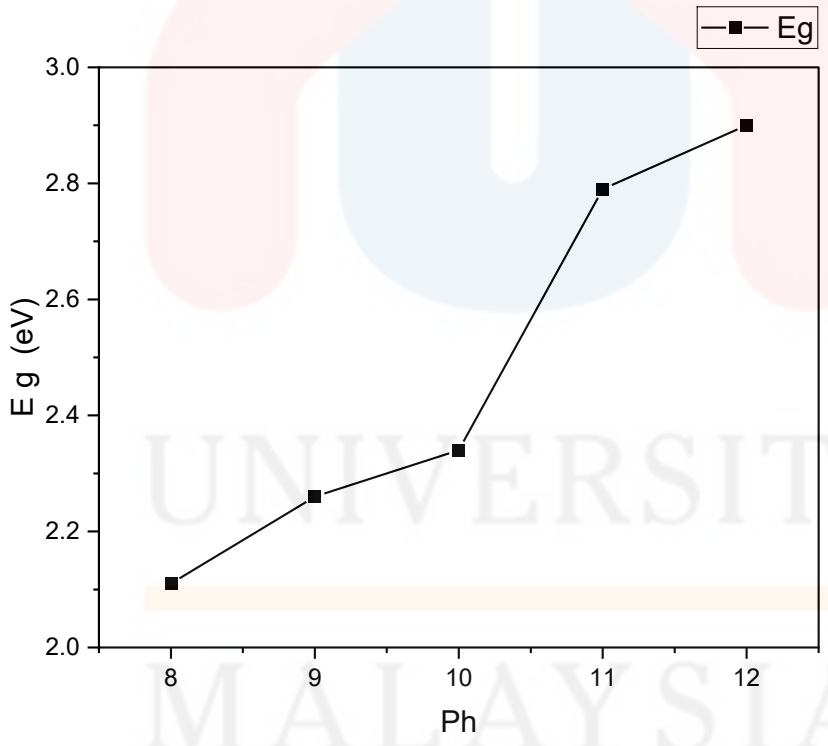

Figure 18: the uv-vis band gap of Zno/cellulose nanocomposite with different pH

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4.4 Scanning Electron Microscopy (SEM)

In figure 19, The ZnO nanorods decorating the cellulose substrate are shown in interesting detail in the SEM picture. These small troops are rather tall, with a consistent rod-like morphology that suggests were grown under regulated conditions using the hydrothermal process. Interestingly, they appear to arrange themselves into ranks, presumably shaped by the cellulose template they call home. Even without a size scale to help us visualise their potential in a variety of applications, we can still admire their amazing microscopic structure. The synthesis ZnO/cellulose nanostructured at were observed as the pH value of 8,9,10,11 and 12 are presented in figure 19. The synthesis of pH 8 were observed as geometric shape (3D) is observed. The size of this microstructure of pH 9 observed as geometric shapes (3D) and the nanorod are closer than pH 8.

After that, ZnO's adherence to cellulose is greatly hindered in high pH conditions (pH 10). There are several factors that come together to explain this phenomenon. First, the negative-charged hydroxyl groups on cellulose and the production of zincate ions (ZnO2-) from ZnO in the alkaline environment cause electrostatic repulsion. In Ph11 and 12 shows the SEM images of zinc oxide under higher magnification it clearly suggests that particle separation is not good enough and also particles with size in micron range was formed so it concludes that zinc oxide prepared using cellulose as the starting material does not produce particles with size in the nanometre range and the particle agglomeration is marginal compared to the previous method.

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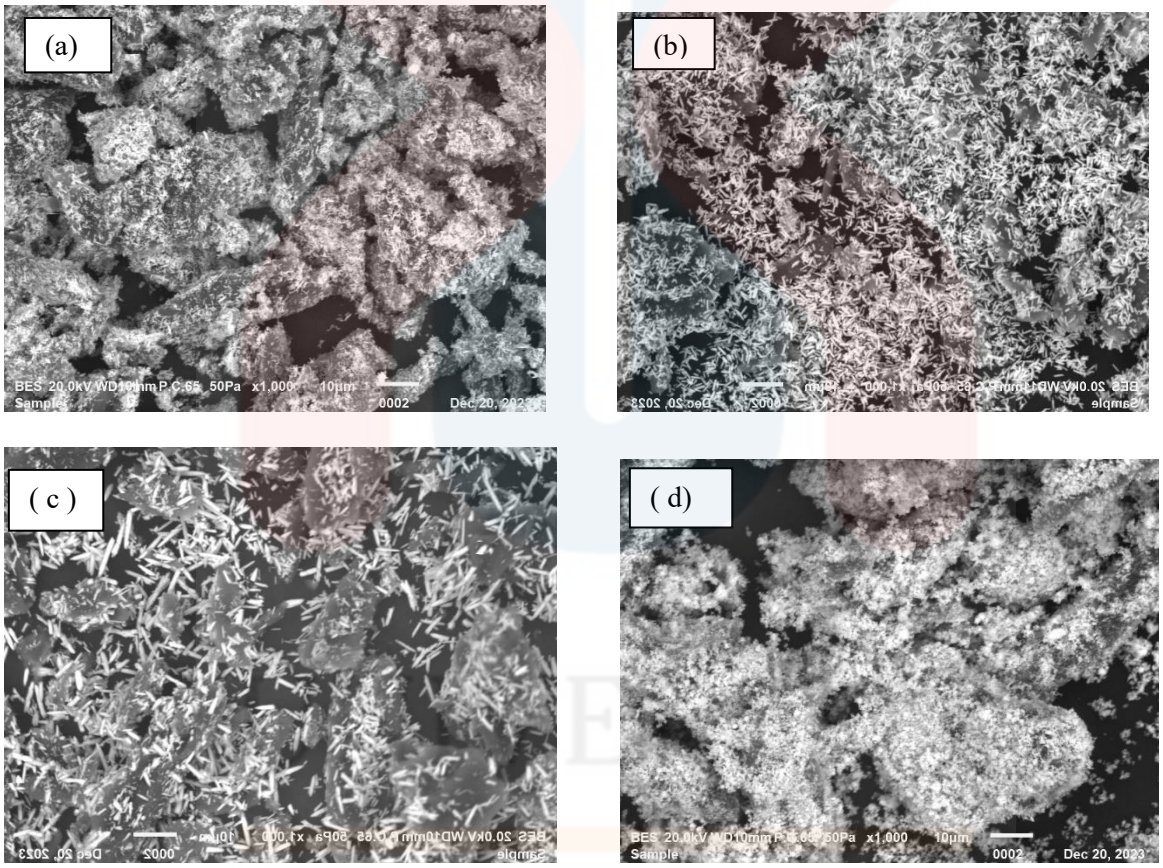
The literature claims that this reaction is what gives the twinned ZnO microparticles the distinctive form that is discussed in the SEM examination section. Because the results show a high degree of agreement with the literature of substrate-less synthesis, but the study was not set up in the realm of strongly time-lapsed reactions. As the initial precursor concentrations decrease, the process yields fewer particles with a higher form. Additionally, the production of dumbbells under the right circumstances has already been noted (Bažant et al., 2015).



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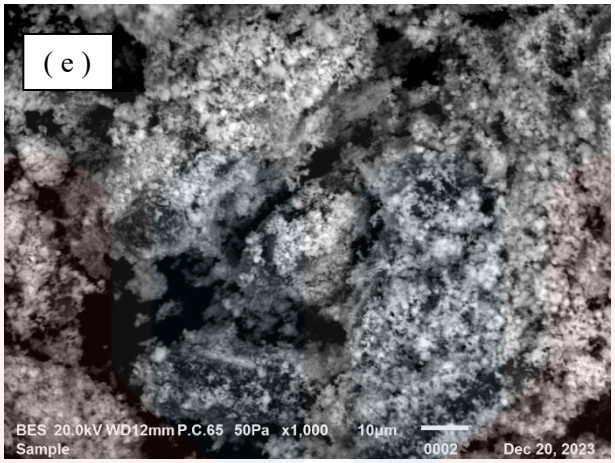

Figure 19: SEM image of ZnO/cellulose nanomposite prepared at different pH value (a) pH 8, (b) pH 9, (c) pH 10, (d) pH 11 and (e) pH 12

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

This study was to fully characterise zinc oxide (ZnO), a variety of analytical methods were used to investigate its optical, chemical, and physical characteristics. An essential technique for examining the phase purity and crystal structure of artificial ZnO nanoparticles was X-ray diffraction (XRD) examination. XRD signals were utilised to identify the hexagonal crystal shape of ZnO, and studies conducted on deposited films demonstrated the presence of a polycrystalline ZnO phases. This method revealed any contaminants or secondary phases in the material and helped determine the lattice characteristics. During XRD, scanning electron microscopy (SEM) was used to take in-depth pictures of a typical ZnO sample, either in thin-film or powder form. The SEM apparatus was carefully assembled, following the manufacturer's guidelines. Calibration then sample holder preparations were crucial stages. UV-Vis spectrophotometry was used for optical characterisation, going beyond structural investigation.

The ZnO powder's XRD pattern changed with pH levels, indicating a strong influence of pH on the crystal structure. ZnO powder dissolved at a very alkaline pH of 12, creating $Zn(OH)_4^{2-}$ ions that subsequently re-precipitated as ZnO particles had a different crystal structure from the initial particles. Because of this, ZnO either pH 12 has a different XRD pattern than at pH 10 or pH 8. It's interesting to note that the cellulose structure did not change at all during the analysis, indicating that most cellulose particles were not substantially altered during the manufacturing process. But at different pH levels (8, 9, 10, 11, and 12), cellulose's presence

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was visible in the XRD pattern, especially at 22°, suggesting that it had an impact on the observed pure phase maxima.

The manufactured ZnO nanoparticles were investigated, and it was found that the predicted band gap energies were higher than those of the bulk material. This finding highlights the appearance of the quantum confinement effect. ZnO in its pure state had an optical band gap of 3.30 eV, however the ZnO/cellulose combination showed variable band gap energies at varied pH levels. Energy band gaps of 2.11 eV, 2.26 eV, 2.34 eV, 2.79 eV, and 2.90 eV were found for ZnO/cellulose at pH values of 8, 9, 10, 11, and 12, respectively, according to the extrapolated straight-line graph. This pattern suggested that pH had a significant impact on the composite's optical characteristics. The quantum confinement effect brought about by smaller particle sizes and a higher surface-to-volume ratio was identified as the cause of the difference between the estimated band gap energies at nanoparticles and bulk material. This work provided insights for potential use in optoelectronic devices by highlighting the importance of pH control in adjusting the optical properties of ZnO/cellulose composites.

The intricate SEM study shown in Figure 15 sheds fascinating light on the ZnO nanorods that adorn the cellulose substrate. The uniform rod-like shape of these nanorods points to hydrothermally controlled development. Interestingly, they group themselves into ranked configurations, maybe as a result of the cellulose template. The creation of ZnO/cellulose nanostructures at different pH values (8, 9, 10, 11, and 12) is further demonstrated in Figure 16. Three-dimensional (3D) geometric forms are discovered at pH 8 and 9, with pH 9 nanorods being closer together than pH 8.

However, at pH 10, the formation of zincate ions (ZnO_2^-) in the alkaline environment and electrostatic repulsion from negatively charged hydroxyl groups on cellulose substantially reduce ZnO's adhesion to cellulose. The use of cellulose was the starting material seems not to yield nanoscale particles, as evidenced by SEM pictures at pH 11 and 12, which show problems

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with particle separation and the creation of bigger particles in the micron range. All things considered, our results highlight how pH affects the shape and aggregation behaviour of ZnO/cellulose nanostructures, offering insightful information for possible uses in a variety of industries.

5.2 Recommendations

1. The semiconductor properties of ZnO/cellulose, take into account surface modification methods. For the purpose of enhancing electron mobility and conductivity, dopants or functional groups may be added.

2. For ZnO nanorods to develop in a regulated manner, concentrate on improving the hydrothermal synthesis parameters. To maintain consistency and alignment of the semiconductor structures, this involves optimising the pH value.

3. Maintained the Teflon by following best practices, ensuring its functionality and minimizing data inconsistencies due to potential corrosion.

4. Investigate the application of ZnO and cellulose composites in batteries or supercapacitors. ZnO's special qualities, such its large surface area and strong conductivity, might enhance the structural stability that cellulose offers.

5. To identify the numerous distictive functional group, associate with the synthesis zinc/cellulose composite, Fourier Transform Infrared Spectroscopy (FTIR) investigation perform. The peak will show the functional group that are characteristic of the ZnO/Cellulose nanocomposite.

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