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Carbon Black-ZnO Composite Prepared Using Solid State Method As Counter Electrode.

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Carbon Black-ZnO Composite Prepared Using Solid State Method As Counter Electrode.

ABSTRACT

Counter electrode based on carbon black (CB)-ZnO composite is proposed as cost effective alternative to conventional Pt counter electrodes used in dye-sensitized solar cell (DSSC) applications. CB-ZnO Composite counter electrodes with different CB weight percentages (5wt%,10wt%,15wt%,20wt%) were prepared using solid state method and coated on fluorine-doped tin oxide (FTO) glass using the doctor blade method. The experimental results revealed that CB-ZnO composites influence photovoltaics performance by increasing electrocatalytic activity. As the amount of CB increases, the catalytic activity improves due to the increase in surface area which then leads to a low charge transfer resistance at electrolyte/CB electrode interface. Due to the use of a modified photoanode together with natural dye sitizers, the counter electrode based on the 15 wt% CB-ZnO composite is able to produce the highest energy.

Keywords: CB-ZnO composite, counter electrode, solid state method, dye-sensitized solar cell.

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Komposit Karbon Hitam-ZnO Disediakan Menggunakan Kaedah Keadaan Pepejal Sebagai Elektrod Pembilang.

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ABSTRAK

Elektrod pembilang berasaskan komposit karbon hitam (CB)-ZnO dicadangkan sebagai alternatif kos efektif kepada elektrod pembilang Pt konvensional yang digunakan dalam aplikasi sel suria peka pewarna (DSSC). Elektrod pembilang komposit CB-ZnO dengan peratusan berat CB yang berbeza (5wt%,10wt%,15wt%,20wt%) telah disediakan menggunakan kaedah keadaan pepejal dan disalut pada kaca oksida timah (FTO) berdop fluorin menggunakan kaedah bilah doktor. Keputusan eksperimen mendedahkan bahawa komposit CB-ZnO mempengaruhi prestasi fotovoltaik dengan meningkatkan aktiviti elektrokatalitik. Apabila jumlah CB meningkat, aktiviti pemangkin bertambah baik disebabkan oleh peningkatan dalam kawasan permukaan yang kemudiannya membawa kepada rintangan pemindahan cas yang rendah pada antara muka elektrolit/CB elektrod. Disebabkan oleh penggunaan fotoanod yang diubah suai bersama dengan sitizers pewarna semula jadi, elektrod kaunter berdasarkan komposit CB-ZnO 15 wt% mampu menghasilkan tenaga yang paling tinggi.

Kata kunci: komposit CB-ZnO, elektrod pembilang, kaedah keadaan pepejal, sel suria peka pewarna.

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DECLARATION

I declare that this thesis entitled “Carbon Black-ZnO Composite Prepared Using Solid State Method As Counter Electrode.” is the results of my own research except as cited in the references.

Signature :

Student’s Name : Aimin Nurhakim bin Adnan

Date :

Verified by signature :

Supervisor’s Name : Dr. Hidayani binti Jaafar

Stamp : ____________________________

Date : ___________________________

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LIST OF ABBREVIATIONS

DSSC	Dye-Sensitized Solar Cell
ZnO	Zinc oxide
Pt	Platinum
XRD	X-ray diffraction
UV-vis	Ultraviolet –visible spectroscopy
CB	Carbon black
ITO	Indium-Tin-Oxide
FF	Fill factor
ISC	Short circuit current
JSC	Short circuit current density

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LIST OF SYMBOLS

°C	Degree Celsius
%	Percentage
nm	Nanometre
µm	Micrometre
eV	Electronvolt
~	Approximation
Å	Angstrom
mA	Milliampere
mV	Millivolt
a.u.	Arbitrary uni

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Carbon Black-ZnO composites made utilising solid-state techniques have been offered as a possible replacement to typical platinum-based counter electrodes in electrochemical systems such as dye-sensitized solar cells (DSSCs). (Zatirostami, 2021). Dye-sensitized solar cells (DSSCs) are a form of photovoltaic technology that turns sunlight into energy. DSSC is made up of various components, including photosensitive dyes, semiconductors, electrolytes, and two electrodes, the working electrode and the counter electrode. Su'ait et al. (2015).

The counter electrode of a dye-sensitized solar cell (DSSC) manufactured using the solid-state approach plays a significant role in the device's electrochemical performance. The counter electrode acts as a location for the reduction of the electrolyte's oxidised state, completing the DSSC circuit and generating power. Due to its high electrical conductivity and good catalytic activity, platinum (Pt) has traditionally been utilised as a counter electrode material. However, because to the high cost and unavailability of carbon black, researchers have looked at other materials for use as counter electrodes.

Carbon black, any group of dense black, finely divided amorphous forms of carbon, usually obtained as soot from partial combustion of hydrocarbons, used primarily as a reinforcing agent in car tyres and other rubber products, but also as a black pigment with high hiding power (Marsh & Rodrguez-Reinoso, 2006). However, in this experiment, carbon black

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produced from soot from the partial combustion of hydrocarbons from the burning of coconut shells will be responded to utilising a solid-state technique as a counter electrode.

A carbon black-ZnO composite material is made up of carbon black nanoparticles and zinc oxide (ZnO). Carbon black is a kind of amorphous carbon that is commonly utilised as a conductive additive in a variety of applications, including electrochemical devices. Zinc oxide, on the other hand, is a semiconductor material with good optical and electrical characteristics that makes it a viable material for use in solar cells and sensors.

The use of carbon black and ZnO nanoparticles in composite materials has various benefits, including increased electrical conductivity, surface area, and mechanical stability. Composites may be made utilising a number of techniques, including solid-state, hydrothermal, and sol-gel procedures. Carbon black-ZnO composites have received a lot of attention in the realm of electrochemistry. It has demonstrated good electrocatalytic activity and stability, allowing it to be used as a counter electrode in a variety of electrochemical devices.

As a counter electrode, carbon black-ZnO composite may be made utilising the solid-state approach. The powdered precursor materials are mixed and heated at high temperatures to initiate a chemical reaction between the components in the solid-state approach. The final product is crushed into a fine powder and utilised as a counter electrode.

The carbon black-ZnO composite generated utilising the solid-state technique as a counter electrode has been the topic of electrochemical study. The creation of effective and low-cost counter electrodes for electrochemical devices such as solar cells and sensors is critical for enhancing their performance and lowering their costs. One interesting option is the use of a carbon black-ZnO composite as a counter electrode.

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The solid-state approach is a simple and low-cost way for creating a carbon black-ZnO composite counter electrode. (Narudin and colleagues, 2021). The powdered precursor materials are mixed and heated at high temperatures to enhance the chemical interaction between the components. The final product is crushed into a fine powder and utilised as a counter electrode. Several investigations have been carried out to explore the electrochemical characteristics of a carbon black-ZnO composite generated by the solid-state technique as a counter electrode. These experiments have revealed that the composite has outstanding electrocatalytic activity and stability, making it an attractive candidate for use as a counter electrode in a variety of electrochemical devices.

Furthermore, using a carbon black-ZnO composite as a counter electrode has various advantages, including strong electrical conductivity, a large surface area, and superior mechanical stability. Because of these characteristics, it is an excellent material for use in electrochemical devices that demand high performance and long-term stability.

Carbon black-ZnO composite generated using the solid-state technique as a counter electrode is a promising material for usage in a variety of electrochemical applications, it may be said. More research is required to optimise the preparation procedure and fully explore its potential in various electrochemical devices.

1.2 Objective

The objective of this study:

1. To investigate different weight percentage of carbon black using solid state method.
2. To determine photovoltaic properties of carbon black.

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1.3 Problem statement

A problem statement for using Carbon black-ZnO composites prepared using solid-state methods as counter electrodes can be framed in the context of improving the performance and reducing the cost of electrochemical devices. Conventional counter electrodes such as platinum are expensive and not readily available in large quantities, making them unsuitable for mass production. (Apetrei & Ghasemi-Varnamkhasti, 2013).

As a result, alternative materials that are cost-effective, easily accessible, and exhibit high electrocatalytic activity and stability are required. Because of their inexpensive cost, simple manufacturing process, and outstanding electrochemical characteristics, carbon black-ZnO composites made utilising solid-state methods provide a possible solution to this problem.

However, there are several issues that must be solved before the promise of carbon black-ZnO composites as counter electrodes can be completely realised. Optimisation of preparation procedures, for example, is critical in order to generate homogenous and well-dispersed composites with appropriate electrochemical characteristics. Furthermore, the composite's long-term stability and endurance under a variety of operating situations should be examined.

1.4 Expected outcome

Recent research suggests carbon black-based counter electrodes as a cost-effective alternative to Pt counter electrodes in dye-sensitized solar cells (DSSCs). In addition, the production of high-energy and low-cost, low-resistance composites show potential in various applications and increase cell efficiency.

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1.5 Scope of study

The study's goal is to look into the potential of carbon black-ZnO composites made utilising solid-state processes as counter electrodes in dye-sensitized solar cells (DSSC) or other electrochemical systems. The goal of this research is to replace the traditional platinum counter electrode with a more cost-effective and efficient alternative. The solid-state approach will be used to make composite counter electrodes with varying weight percentages of carbon black and ZnO. The performance of the carbon black-ZnO composite counter electrode will be assessed in terms of energy conversion efficiency, conductivity, and electrocatalytic behaviour. This research seeks to contribute to the creation of extremely stable and cheap DSSCs using various counter electrode materials.

1.6 Significant of study

The investigation of the Carbon Black-ZnO Composite made using the solid-state approach as a counter electrode is relevant because it seeks to evaluate the possibility of this composite as a substitute for standard platinum counter electrodes in dye-sensitized solar cells (DSSCs). The composite will be made with various weight percentages of carbon black and ZnO, and its performance will be measured in terms of energy conversion efficiency, conductivity, and electrocatalytic behaviour. Carbon materials are useful for creating counter electrodes in DSSCs because of their high catalytic activity, resistance to iodine corrosion, and stability, and they are less costly than platinum-based counter electrodes. As a result, the study's goal is to contribute to the development of economical and stable DSSCs using different counter electrode materials, which has a lot of promise for future applications in solar energy conversion.

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CHAPTER 2

2 LITERATURE REVIEW

2.1 Dye Sensitized Solar Cell

DSSCs are a type of solar cell from the third generation. The DSSC's operating concept is derived from the principle of photosynthesis in plants. Since their initial 1991 study, Grätzel and coworkers have extensively developed the dye-sensitized solar cell (DSSC), It demonstrates the advantage of fabricating the dye sensitised photoanode utilising nanocrystalline TiO2 layer. (Raïssi et al., 2020). In comparison to the far more established silicon-based solar cells, DSSCs provide various advantages such as a quicker energy payback time, transparency, compatibility with flexible substrates, and, most crucially, superior performance in low light circumstances, notably artificial lighting. Figure 2.1 depicts a schematic illustration of DSSCs.

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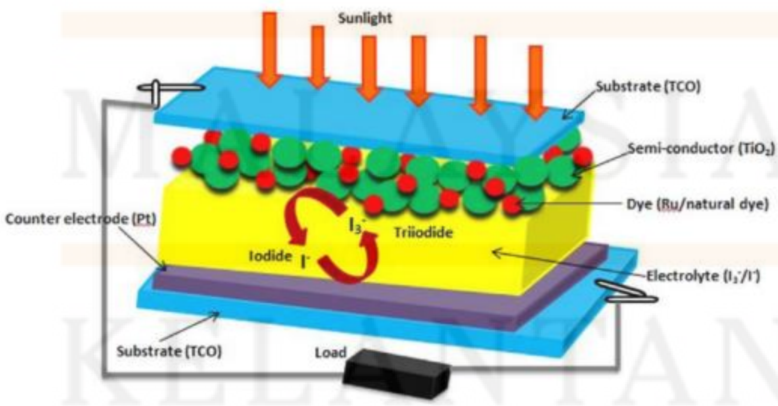

Figure 2.1: Schematic diagram of a dye-sensitized solar cell.

Dye-sensitized solar cells (DSSCs) are a type of photovoltaic technology that converts sunlight into electrical energy. (Mohanty & Tyagi, 2015). They are a relatively new technology that was initially created in the 1990s and are currently being explored as a potentially cost-effective and environmentally friendly alternative to standard silicon solar cells. The fundamental building block of a DSSC is a thin coating of a semiconductor material, such titanium dioxide (TiO2), that has been painted with a dye molecule. Sunlight is absorbed by the dye, which then transmits the energy to the semiconductor substance, which produces an electric current. An external load is powered by the current, which is collected via a conductive substrate that is commonly constructed of a transparent material like glass or plastic.

One benefit of DSSCs is that they can be produced using inexpensive components and straightforward production techniques, which might result in a price difference between them and conventional silicon solar cells. They are excellent for a variety of applications since they work well in low light circumstances and may be produced in flexible or transparent forms. But there are certain restrictions on DSSCs as well. Their potential for practical use may be constrained by their normally lower efficiency than silicon solar cells. In addition, the dye molecules are made from scarce and pricey substances like ruthenium, which can increase the cost of manufacturing them in bulk. Despite these drawbacks, DSSC research and development are ongoing, and the technology might eventually provide a viable substitute for conventional solar cell technology. (Wu et al., 2017b).

2.2 Electrolyte

A standard electrolyte is embodied between the two glass substrates, thereby functionalizing the pigment cation reduction and subsequent electron infusion thereby achieving the electron process in DSSC in line with Mohamad et al., (2017). Approximately,

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the electrolyte may be classified into three kinds: liquid electrolyte, quasi solid-state electrolyte, and solid-state electrolyte. It may also be classified into two liquid electrolytes: organic solvent electrolyte (acetonitrile, ethylene carbonate, etc.) and ion liquid electrolyte.

The electrolyte is a vital part of DSSCs. Continually renewing both the dye and itself, it is in charge of the inner charge carrier transit between electrodes during DSSC functioning. Gonçalves et al. (2008). The effectiveness of converting light into electricity and the long-term durability of the devices are both greatly influenced by the electrolyte. Efficiency (FF) of a DSSC device is influenced by photocurrent density (JSC), photovoltage (VOC), and fill factor. The positive and negative electrodes of an electrochemical device can conduct pure ions more easily when a material called an electrolyte is present. The majority of electrochemical devices require electrolytes in order to function.

While holes migrate via electrolytes or hole conductors, electrons in the electrochemical circuit of DSSCs pass through TiO2 crystals. The hole-transport mediators in this method are electrolytes or hole conductors. In order to renew dye and themselves, DSSCs generally employ electrolytes or hole conductors. The iodide/tri-iodide I-/I3 redox pair system is one example of the liquid phase electrolyte under investigation by these DSSCs. A liquid electrolyte based on I/I3 has a low diffusion through the transparent semiconductor surface, a rapid stain renewal rate, and a low recombination disturbance rate during photochemical processes inside cells.

2.3 ZnO properties

The Carbon Black-ZnO composite, prepared using the solid-state method as a counter electrode, exhibits a range of properties that make it a promising material in various

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applications. The combination of carbon black and zinc oxide (ZnO) brings together their individual characteristics, resulting in enhanced performance and functionality.

Zinc oxide is a wide-bandgap semiconductor with excellent electrical and optical properties. It is known for its high electron mobility and good transparency in the visible range of the electromagnetic spectrum. When incorporated into the composite, ZnO contributes to its electrical conductivity, making it suitable for electrochemical applications. One of the notable properties of ZnO is its electrocatalytic activity. It can efficiently catalyze various electrochemical reactions, such as the reduction and oxidation of species in electrolyte solutions. This property is crucial for the composite's role as a counter electrode, where it facilitates the transfer of electrons during electrochemical processes.

Furthermore, ZnO exhibits a high specific surface area, which increases the available surface for electrochemical reactions. This property enhances the composite's electrocatalytic efficiency and promotes faster reaction kinetics, leading to improved performance in energy storage and conversion devices. Carbon black, on the other hand, is a highly conductive form of carbon with a large surface area. It possesses excellent electrochemical stability and acts as a conductive network within the composite. The incorporation of carbon black enhances the electrical conductivity of the composite, ensuring efficient electron transfer during electrochemical reactions.

The composite's solid-state preparation method offers several advantages. It allows for the intimate mixing of carbon black and ZnO at the nanoscale, leading to a homogenous distribution of the two components. This uniform distribution enhances the synergistic effects between carbon black and ZnO, maximizing their collective properties and overall performance. The Carbon Black-ZnO composite, prepared using the solid-state method as a counter electrode, combines the electrocatalytic activity of ZnO with the excellent conductivity

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and stability of carbon black. This composite exhibits high electrical conductivity, electrochemical stability, and enhanced reaction kinetics. These properties make it a promising material for various applications, including energy storage devices, electrochemical sensors, and catalysis.

2.4 Counter electrode

The counter electrode's role is to minimize the oxidized species of redox couples as a buffer (Mubarak et al., 2018). It can be achieved with as minimum resistance as practicable in order to produce an effective DSSC. A counter-electrode in the DSSCs should have mesoporous, strong catalytic capacity, and chemical stability. Platinum (Pt) nanoparticles have been thermally deposited in the most popular, efficient counter electrode substrate used by the redox iodide / triiodide device. For the iodide / triiodide device, the thermal deposited Pt counter electrode is very efficient. Fajar & Endarko, (2017) also revealed that Pt has strong catalytic electrical activity to suppress triiodide. Because Pt is rare, costly material and also has low retention in the corrosive I- /I3 - redox method, a variety of research groups are interested in seeking a substitute to Pt.

In a dye-sensitized solar cell (DSSC), the counter electrode is an important component that completes the circuit by collecting electrons generated in the photoanode. (Arifin et al., 2012b). The counter electrode is usually made of a conductive material, such as platinum or a conductive polymer, and is usually coated with a catalytic material, such as platinum nanoparticles. The photoanode's dye molecules absorb light, which results in excited electrons being transported to the photoanode and holes remaining in the dye molecules. An electric current is subsequently created when the excited electrons pass through the photoanode and

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into an external circuit. The counter electrode, which serves as the DSSC's cathode, has to gather electrons for the electrical circuit to be complete. (Park & Seo, 2011).

The selection of the counter electrode material is important for the performance and efficiency of the DSSC. The counter electrode material should have high electrical conductivity, large surface area, and excellent catalytic activity to facilitate electrochemical reactions in the device. (Westbroek, 2005). In addition, the counter electrode should be stable and durable in operating conditions, such as high temperature, humidity and exposure to light. Overall, the counter electrode is an important component in many electrochemical devices, including DSSCs, batteries and fuel cells. The choice of counter electrode materials and their optimization is an area of active research to improve the efficiency, cost-effectiveness and environmental impact of these devices.

2.4.1 Platinum

Platinum is a highly sought after precious metal known for its exceptional properties and wide range of applications. With its striking silver-white color and exceptional resistance to corrosion, platinum holds a special place in various industries and fields. One of the most notable properties of platinum is its extraordinary catalytic activity. Platinum-based catalysts are widely used in chemical reactions. Platinum also exhibits excellent electrical conductivity, making it an important component in many electronic devices. Its use can be found in a variety of applications, including electrical contacts and electrodes.

The counter electrode in DSSC should have mesoporous catalytic capacity, strength, and chemical stability. Platinum (Pt) nanoparticles have been thermally deposited in the most popular and efficient counter electrode substrates used by iodide/triiodide redox devices. (Wu et al., 2017c). For iodide / triiodide devices, thermally deposited platinum counter electrodes

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are very efficient. It is also traditionally revealed that platinum has strong catalytic electrical activity to block triiodide and excellent catalytic activity. However, due to its high cost and scarcity, researchers have explored alternative materials for use as counter electrodes, including carbon black, graphene and other metal oxides.

2.4.2 Carbon black

Carbon black is an amorphous form of carbon produced by the incomplete combustion or thermal decomposition of hydrocarbons, such as shell burning. It is a finely divided black powder consisting mainly of carbon particles ranging in size from less than 10 nm to several micrometers. (Carbon Black, n.d.).

How ever for use Carbon Black-ZnO composites have several desirable properties as counter electrodes, including high electrical conductivity, large surface area, and excellent catalytic activity. These properties allow the composite to efficiently catalyze the reduction of the oxidized form of the electrolyte and facilitate electron transfer, leading to higher overall DSSC efficiency. The addition of zinc oxide (ZnO) to the composite further improves its properties. ZnO is a wide band gap semiconductor with excellent electrochemical stability. It acts as a co-catalyst with carbon black, promoting electron transfer and facilitating electrochemical reactions. The combination of carbon black and ZnO in the composite produces a synergistic effect, leading to enhanced electrocatalytic performance. Furthermore, carbon black is a conductive material with a high surface area that can serve as a catalyst for the reduction of oxidized electrolyte species in DSSCs.

The use of carbon black has several advantages as a counter electrode material in dye-sensitized solar cells (DSSC), including that carbon black is a low-cost and readily available material, making it an attractive alternative to conventional platinum electrodes. In addition,

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carbon black has a high surface area, which allows more dye molecules to be absorbed onto the electrode surface, leading to higher light absorption and more efficient electron transfer and also carbon black has good electrical conductivity, which ensures electron transfer. . efficient between the counter electrode and the electrolyte, leading to higher energy conversion efficiency in DSSCs. (Gomathi et al., 2022). Overall, the use of carbon black as a counter electrode material in DSSCs is a promising research area, as it offers a potential alternative to platinum and other precious metal catalysts. However, further studies are needed to optimize the performance and stability of carbon black-based DSSCs.

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2.5 Method used

SOLID STATE	HYDROTHERMAL	SOL GEL
The solid-state method is relatively simple, involving mechanical mixing and a heat treatment step. (Thorogood et al., 2022).	The hydrothermal method involves several reaction steps, making it more complex and time-consuming than other methods. (Pan et al., 2015c)	The sol-gel method involves various steps, including preparation of the solution requires careful control and optimization. (Landau, 2008)
This method facilitates good interfacial contact between Carbon Black and ZnO nanoparticles, promoting potential synergistic effects and better composite properties.	Hydrothermal synthesis requires specialized high-pressure autoclaves and precise control over temperature and pressure.	The use of certain precursors and solvents in the sol-gel process can increase costs and limit scalability for large-scale production.
1000-1500°C (it depending on the specific synthesis method and materials used).	100 – 250°C (under the vapour pressure).	50- 80°C (depending on the specific materials and reaction conditions used).

Table 2.5: Differences of method used.

Different techniques for carbon black synthesis namely solid state, sol-gel and hydrothermal methods differ in the way the precursor materials are prepared and processed. For the solid-state method, the precursor material is heated in the absence of air or oxygen to a high temperature, resulting in the formation of carbon black. The solid-state method is simple,

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cost effective and can produce high quality carbon black with controlled particle size and morphology on the properties of carbon black which resulted.

In addition, the sol-gel method is that the precursor material is dissolved in a solvent to form a solution, which is then converted into a gel through the addition of a cross-linking agent. The gel is then heated to an oxygen-free temperature to form carbon black. This method usually involves the use of liquid precursor materials, such as metal salts, and allows the formation of highly dispersed and uniform carbon black particles, but it can be complex and expensive compared to other methods.

The hydrothermal method involves heating a liquid precursor material, such as glucose or sucrose, in a solvent under high pressure and temperature to induce thermal decomposition and the formation of carbon black. The hydrothermal method can produce highly pure and uniform carbon black nanoparticles with well-controlled particle size and crystallinity, but it can be complex, time-consuming and expensive compared to other methods.

However, the best method for carbon black synthesis depends on the specific requirements of the application, including desired particle size, morphology, purity and cost. However, the solid-state method is the most cost effective and has the highest yield of carbon black. The solid-state method enables precise control over the properties of the resulting carbon black, such as particle size, surface area, and porosity, by adjusting the heating temperature, the duration of the heating process, and the composition of the precursor material. Carbon black produced by the solid-state method is usually more highly structured and crystalline than carbon black produced by other methods, such as the furnace or lampblack method. This structure results in unique properties, such as high electrical conductivity, high surface area and excellent light absorbing properties, which make carbon black a valuable material in various industrial applications. Overall, the solid-state method is an important technique for

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the production of high-quality carbon black, and it offers a level of control over the properties of the resulting material that is difficult to achieve with other methods.

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CHAPTER 3

3 MATERIAL AND METHOD

3.1 Material

Carbon Black-ZnO composites are innovative materials with excellent electrochemical properties that make them suitable candidates for use as counter electrodes in various electrochemical devices such as batteries, fuel cells and supercapacitors. Black carbon material produced from coconut shell that will be mixed with ZnO. The production procedure of this composite material is important in determining its overall characteristics and performance. This article will go through the solid-state process to make Carbon Black-ZnO Composite and its electrochemical characteristics as a counter electrode. Figure 3.1 displayed the carbon black.

Figure 3.1: Carbon black

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3.2 Method

Carbon Black-ZnO composites produced through solid state techniques are highly conductive materials with good electrochemical properties. Carbon black, a type of amorphous carbon, and zinc oxide nanoparticles are used to make the composite. The solid-state method offers a simple and convenient approach to synthesize carbon black-ZnO composites. The preparation of carbon black requires a carbonization process and then grinding to obtain a fine powder. This next process involves physical mixing of carbon black powder and zinc oxide, followed by heat treatment or sintering at high temperatures between 1000-1500°C. The carbon black-ZnO composite was carefully ground using a mortar or milling process to ensure a uniform distribution of carbon black in the ZnO precursor. The process of collection and characterization with various characterization techniques such as X-ray diffraction (XRD), Ultraviolet–visible spectroscopy (UV-VIS), energy scattering to analyze the structure, morphology and composition of composite elements.

The research flow chart illustrates the simplest procedures of preparation, characterisation, and fabrication portrayed in figure 3.2.

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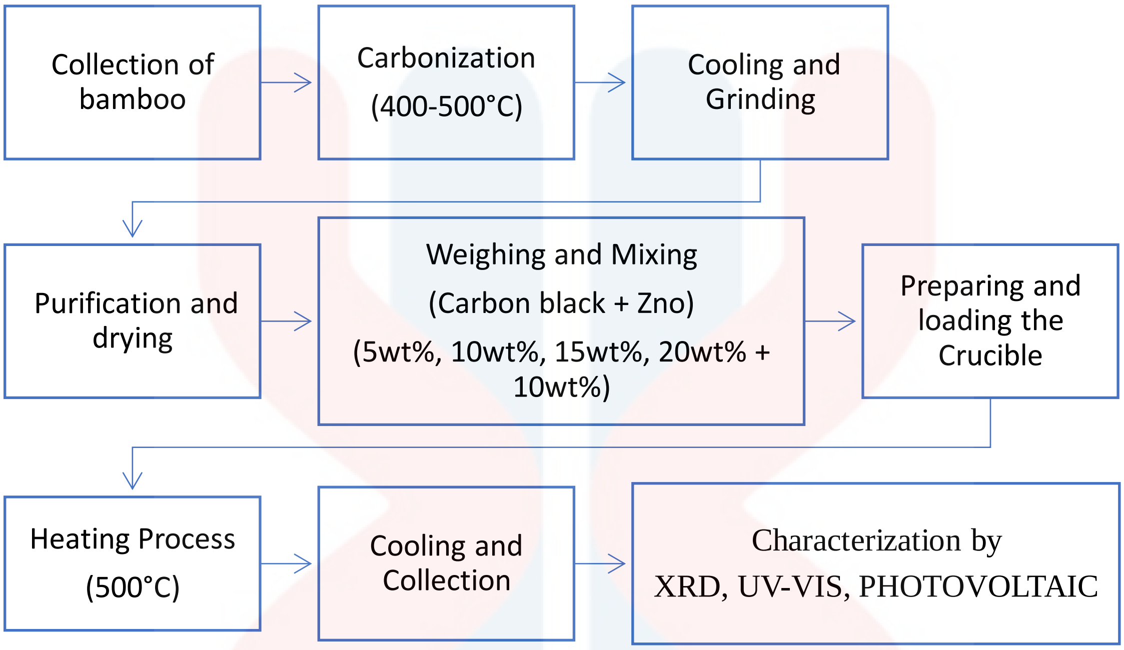

Figure 3.2: Research flow for carbon Black-ZnO and characterization

3.3 Characterization

3.3.1 X-ray diffraction (XRD)

X-ray diffraction (XRD) is a powerful analytical technique used to determine the crystal structure and characteristics of materials. It is based on the principle that when X-rays interact with a crystalline sample, they undergo constructive interference, resulting in a distinct pattern of diffracted X-rays. The process of XRD involves directing a beam of X-rays onto a sample at various angles and measuring the intensity and angle of the diffracted X-rays. These measurements are then used to determine the arrangement of atoms within the crystal lattice, as well as other important properties such as crystal symmetry, crystal size, and orientation. The diffraction pattern obtained from XRD is a series of peaks, each corresponding to a unique

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set of crystal planes within the sample. The positions and intensities of these peaks provide valuable information about the crystal structure. By comparing the experimental diffraction pattern with a database of known patterns, scientists can identify the crystalline phase(s) present in the sample.

In the context of Carbon Black-ZnO composites prepared using solid state methods as counter electrodes, XRD can provide valuable insight into the structural properties of composite materials. To fully understand these qualities, it is necessary to characterize the structural characteristics of composites using methods such as X-ray diffraction (XRD). (King et al., 2022). XRD is a non-destructive technology that offers information on the crystal structure and lattice parameters of composites, as well as the size and distribution of ZnO nanoparticles. The phase composition and degree of crystallinity of the composite can be determined using XRD patterns.

When analyzing Carbon Black-ZnO composites using XRD, this technique involves bombarding the sample with X-rays and measuring the diffraction pattern of the scattered X-rays. These patterns provide information about the arrangement of atoms in the material and can be used to determine the crystal structure, phase composition and crystallinity of the composite. In the case of Carbon Black-ZnO composites, XRD can help identify the presence of different phases or crystalline forms of carbon and zinc oxide. It can determine whether the composite consists of amorphous or crystalline carbon, as well as the crystal structure of the ZnO component. Diffraction patterns obtained from XRD can be compared to a database of known crystal structures to accurately identify the phases present in the composite.

Additionally, XRD can provide information about the degree of crystallinity of the composite material. By analyzing the intensity and width of the diffraction peaks, it is possible to evaluate the nature of the crystals and the size of the crystals in the composite. This

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information is important in understanding the structural characteristics and properties of Carbon Black-ZnO composites. Additionally, XRD can be used to investigate any possible changes in the crystal structure or phase composition of the composite after certain treatments or modifications. By comparing XRD patterns before and after certain processes, such as heat treatment or chemical reactions, researchers can gain insight into the structural transformations occurring in composite materials.

3.3.2 Ultraviolet–visible spectroscopy (UV-VIS)

The use of advanced materials in electrochemical applications has led to the development of new composites, which aim to improve the performance of devices such as dye-sensitized solar cells and electrochemical sensors. Among these materials, Carbon Black-ZnO composite, synthesized by solid-state methods, appears as a promising candidate for use as a counter electrode. Characterizing the optical properties of these composites through UV-Visible (UV-Vis) spectroscopy provides valuable insight into their structure, composition and potential applications.

In the UV-Vis spectrum, distinctive features arise from the interaction of light with composite constituents. Carbon black contributes to a broad absorption band spanning both the UV and visible regions. At the same time, ZnO introduces its characteristic absorption advantage in the UV region. The intensity of the absorption peak reflects the concentration and dispersion of carbon black and ZnO. A higher intensity may indicate a higher concentration or better dispersion, an important factor that affects the overall performance of the composite in electrochemical applications.

A shift in the absorption peak or a change in the shape of the absorption band can indicate a structural change in the composite. For example, modifications in the absorption

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edge of ZnO may suggest that variations in crystallite size shed light on the stability and durability of composites. Additionally, estimating the optical band gap of composites is important to understand their electronic properties. ZnO's absorption edge helps in defining the band gap, allowing researchers to tailor the material for specific applications by manipulating its electronic structure. The overall color of the composite, as observed in the UV-Vis spectrum, provides insight into the interaction of carbon black and ZnO. Changes in color may indicate changes in composition or structure. Additionally, transparency is an important factor for applications such as transparent conductive films, where higher transparency is often desired.

In conclusion, UV-Vis spectroscopy serves as a powerful tool to characterize Carbon Black-ZnO composites prepared using solid state methods. Insights gained from UV-Vis spectra contribute to a comprehensive understanding of the optical properties of composites, guiding further research and development for enhanced electrochemical performance in various applications. Integrating UV-Vis spectroscopy with other characterization techniques ensures a holistic approach to material analysis and paves the way for the advancement of innovative materials in the field of electrochemistry.

3.3.3 Photovoltaic

Photovoltaic (PV) refers to the technology that converts sunlight directly into electricity using solar cells. Photovoltaic systems play a crucial role in harnessing renewable energy and have gained significant attention as a sustainable and environmentally friendly power generation option. The fundamental component of a photovoltaic system is the solar cell, which is typically made of semiconductor materials.

The XRD study reveals the composite material's amorphous character as well as the dispersion of ZnO nanoparticles in the carbon black matrix. This information may be utilised

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to determine the composite material's phase composition and degree of crystallinity. The solid-state approach is a low-cost method for producing Carbon Black-ZnO composites for solar applications. This material might be utilised to create high-performance, low-cost solar cells. 2014b (Thomas et al.).

The development of efficient and cost-effective materials for renewable energy applications has become an important research focus in recent years. One such area of exploration is the use of carbon black-zinc oxide (ZnO) composites as counter electrodes in photovoltaic devices. The solid-state method has emerged as a promising technique for the preparation of these composites, offering advantages in terms of simplicity and scalability.

The use of carbon black in composites is driven by its excellent electrical conductivity and high surface area, which are important for efficient charge transfer in photovoltaic systems. By incorporating ZnO nanoparticles into the carbon black matrix, the composites can benefit from the unique properties of ZnO, such as favorable energy band structures and light harvesting capabilities. The solid state method involves a simple synthesis process, usually starting with mixing carbon black powder and ZnO followed by compaction and sintering. This method ensures good interfacial contact between the two components, promotes electron transport and facilitates electrochemical reactions at the counter electrode.

Moreover, the resulting carbon black-ZnO composite exhibits enhanced electrocatalytic activity, which is important for efficient light-to-electricity conversion. The composite structure enables rapid regeneration of redox couples involved in the photovoltaic process, which leads to increased overall performance and device stability. Furthermore, solid-state preparation methods offer advantages in terms of scalability and cost-effectiveness. It can be easily adapted for large-scale production, making it a viable option for future industrial applications.

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CHAPTER 4

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4 RESULT AND DISCUSSION

4.1 Characterization of Carbon black-ZnO

This chapter has discussed about different %wt for calcined carbon black zinc oxide composites using X-Ray Diffraction (XRD), crystal structure properties, including crystal size and lattice strain have been discussed. Then, UV-VIS spectroscopy is used to investigate the visible part of the spectrum and identify band gaps. In addition to using photovoltaic techniques for characterization

4.1.1 Structural and Properties of CB-ZnO Composite

The quest for efficient solar energy conversion explores materials at the atomic level. Carbon Black-ZnO composites, synthesized via solid-state methods, appear as potential contenders for the important role of counter electrodes in these devices. Decoding its secrets requires not only the embrace of light, but also a powerful X-ray diffraction (XRD) instrument. These XRD results reveal the crystal fingerprint of the composite and its implications for solar cell performance. XRD patterns, with their distinctive peaks, offer valuable insight into the crystal structure and composition of composite materials.

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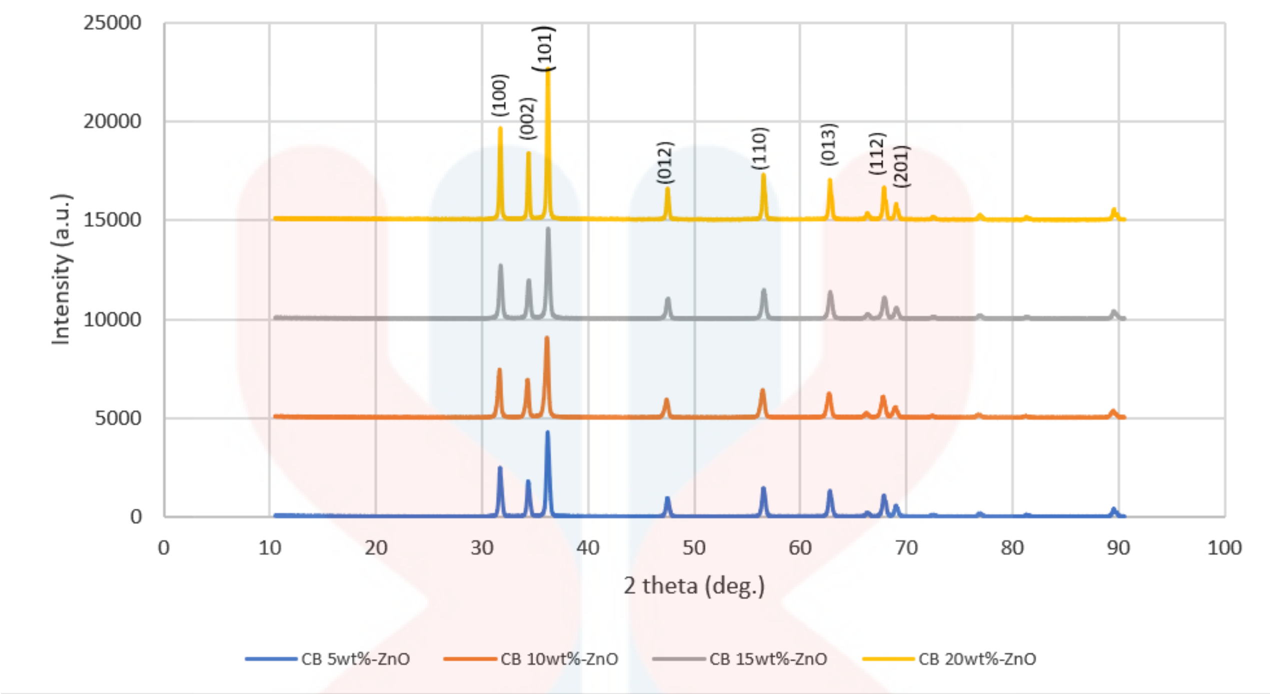

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Figure 4.1: XRD Spectra of Carbon black – ZnO

The XRD pattern shows a prominent peak standing high at a certain angle, like a sentinel indicating the presence of ZnO in its hexagonal wurtzite phase with the presence of ZnO at prominent peaks at 31.7°, 34.4°, 36.4°, 47.5°, 56.7°, 62.9°, 67.9 °, and 69.2° with delegated to the diffraction of lattice planes (1 0 0), (0 0 2), (1 0 1), (0 1 2), (1 1 0), (0 1 3), (1 1 2), and (2 0 1) planes respectively corresponds to the characteristic reflection of ZnO in its hexagonal wurtzite phase. This confirms the successful incorporation of ZnO into the composite. This diffraction peaks were reflect to tetragonal anatase crystalline phase of CB-ZnO. According to Zheng et al. (2021), his value with the standard data Joint Committee on Powder Diffraction Standards (JCPDS) Card No. 21-1272).

Crystallization sizes with different 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO were calculated from diffraction line broadening using the Debye Scherrer formula (Rahman et al., 2019);

$$D = K\lambda / \beta Cos\theta \qquad \text{Equation 4.2}$$

Where D is the mean size of the crystallites (nm), K is the crystallite form variable and the appropriate approximation is 0.9 (Scherrer's constant) , λ wavelength (0.154060), β full width at the half maximum intensity (FWHM) in radians of the X-ray diffraction peaks and the Bragg angle is θ. Crystallization sizes with different 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO were shown in the Table 4.1.

Table 4.1 : Lattice parameters, average crystallite size and d spacing of 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO.

Samples	Average crystallite size, D (nm)	Cell Volume (Å^3)	a(Å)	c(Å)
CB 5wt%-ZnO	27.64	47.61	3.25	5.21
CB 10wt%-ZnO	25.82	47.61	3.25	5.21
CB 15wt%-ZnO	26.51	47.61	3.25	5.21
CB 20wt%-ZnO	35.08	47.61	3.25	5.21

Based on Table 4.1, it has been identified that the average crystal size of carbon black-zinc oxide shows variation based on 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO, measuring 27.64 nm, 25.82nm, 26.51 nm and 35.08nm. According to Hossain and Ahmed (2023), the average crystal size is subject to variation influenced by both and the specific conditions used in the synthesis process. High wt % values have the potential to foster crystal growth, thus contributing to the observed difference in average crystal size in the

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Carbon black-ZnO composites. The average crystal size appears to be affected by the amount of carbon black added to the ZnO.

In the carbon black-ZnO sample, the average crystal size of CB 20wt%-ZnO is larger (35.08 nm) compared to other samples. This suggests that the amount of wt% carbon black can act as a variable in which crystals grow.

In conclusion, the carbon black content of the crystal landscape and performance comparison with other composites synthesized with different methods signal paths for exploration and XRD results show the preparation of Carbon Black-ZnO composites with ZnO present in its crystalline wurtzite phase and carbon black likely present . The absence of impurities and the potential to control the crystal size through synthesis parameters are promising aspects to further optimize the composite properties for counter electrode applications.

4.2 Absorption Spectra of ZnO doped with carbon black (CB)

Figure 4.3 represents the visible absorption spectrum of 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO.

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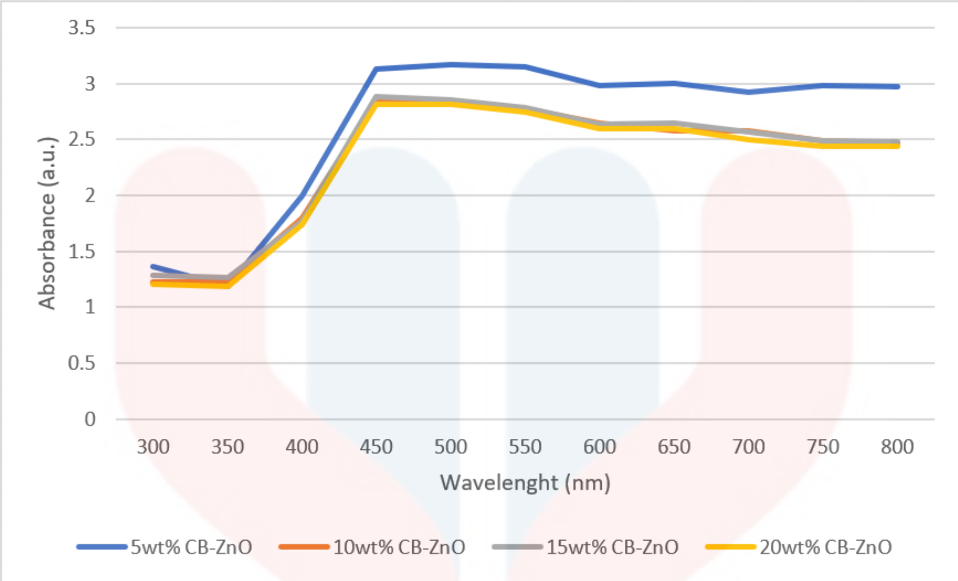

Figure 4.2: UV visible absorption spectra of carbon black-ZnO

The exploration of composite materials with tailored properties has emerged as a promising avenue. An interesting combination is the Carbon Black-ZnO composite, synthesized using a solid-state method, and its potential as a counter electrode in solar cells.

UV-vis spectra reveal the light absorption behavior of Carbon Black-ZnO composites. All four of these composites have improved absorption properties with visible wavelengths in the deep light area 300-800 nm. The visible absorption band shifts to higher energy states, revealing a different range of about 380 nm with optimal absorption for pigments from 5wt% CB-ZnO where it absorbs more light in the blue range in sampping for 20wt% CB-ZnO , the absorption peak is observed around 370 nm and the broad absorption scale is around 370–800 nm which is relatively less compared to 5wt% CB-ZnO where it absorbs less light showing in the yellow range. The prominent peak centered in the UV range speaks volumes for the exceptional ability of carbon black nanoparticles to capture UV light.

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4.2.1 Band Gap Determination

The energy band gap of carbon black-zinc oxide with different amount (CB 5wt%, CB 10wt%, CB 15wt%, CB 20wt%) was analyzed through UV Vis spectrophotometer. According to Mursyalaat et al. (2023), the band gap (eg) of this sample is calculated using equation 4.1:

$$(\alpha h\nu) = k(Eg - h\nu)^n \qquad \text{Equation 4.1}$$

where :

α optical absorption coefficient

hv photon energy

E_g absorption coefficient

n constants (1/2 and 2 respectively for indirect and direct band gap)

This method was developed by Davis and Mott for semiconductor materials to find the band gap using a Tauc plot. Table 4.2 above shows presents data on the absorption and photon energy of Carbon Black-ZnO composites prepared using the solid state method, with varying weight percentages of carbon black (5wt%, 10wt%, 15wt% and 20wt%).

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Table 4.2 : Absorbance and photon energy of 5wt% CB-ZnO, 10wt% CB-ZnO, 15wt% CB-ZnO and 20wt% CB-ZnO.

Samples	Peak Absorption (nm)	Absorbance (a.u.)	Photon Energy (eV)
CB 5wt%-ZnO	425	3.24	3.34
CB 10wt%-ZnO	423	3.00	3.41
CB 15wt%-ZnO	427	2.96	3.36
CB 20wt%-ZnO	429	2.92	3.40

carbon black-ZnO states that the photon energy at the peak absorption wavelength of CB 5wt%-ZnO is 425 nm which is the lowest wavelength compared to other samples. In addition, CB 10wt%-ZnO has the highest absorption peak, at 423 nm with an energy band gap of 3.41 eV. The peak absorption intensity decreases with increasing carbon black content. This trend suggests that higher concentrations of black carbon may inhibit overall light absorption, potentially due to light scattering or clumping.

Another important aspect revealed by the spectrum is the peak width. This indicates a heterogeneous size distribution of carbon black nanoparticles in the composite. Although the exact implications of this heterogeneity require further investigation, it opens up exciting possibilities for tailoring material properties for specific applications. By manipulating the synthesis process or using size selection techniques, the size distribution of nanoparticles can potentially be optimized to achieve the desired electronic and optical properties. The combined strength of strong UV absorption and visible transparency positions Carbon Black-ZnO

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composites as promising candidates for counter electrodes in dye-sensitized solar cells (DSSCs).

In conclusion, the UV-vis spectra of Carbon Black-ZnO composites prepared using the solid state method paint a picture of an interesting material with great potential for solar energy applications. Its strong UV absorption, visible transparency, and potential for further optimization position it as a promising candidate for counter electrodes in DSSCs.

4.3 Modified of working Photoanode for DSSCs application

Table 4.3 illustrates the photoelectric dimensions for DSSC sensitization applications in four different percentages of carbon black mixtures. As formulated by Mohamad et al. (2017), photovoltaic properties are measured using the following equation:

$$FF = \frac{(I_{max} \times V_{max})}{I_{sc} \times V_{oc}} \quad \text{(Equation 4.1)}$$

$$\eta = \frac{(I_{sc} \times V_{oc} \times FF)}{P_{in}} \quad \text{(Equation 4.2)}$$

Where, JSC is short-circuit current density at zero voltage, VOC is the open-circuit voltage at zero current density, Vm is maximum voltage, ISC is short-circuit current at zero voltage, Im is the maximum current, Pin is the radiation power incident on the cell and FF is the fill factor.

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Table 4.3: Photovoltaic parameters of counter electrodes based on different amounts of CB-ZnO composite.

Sample	Voc (mV)	Isa (mA)	Jsc (mA/cm^2)	Fill factor, FF (%)	η Efficiency (%)
5wt% CB-ZnO	0.1074	0.35	0.35	2.22	0.08
10wt% CB-ZnO	0.1052	0.26	0.26	2.94	0.1
15wt% CB-ZnO	0.1127	0.41	0.41	2.26	0.18
20wt% CB-ZnO	0.1187	0.50	0.50	2.24	0.13

The table shows the photovoltaic parameters of counter electrodes based on different amounts of CB-ZnO composites. The parameters shown are Voc (open circuit voltage), Isc (short circuit current), Jsc (current density), FF (fill factor), and η (efficiency). Overall, the efficiency of the counter electrode increases with the amount of CB-ZnO composite up to 15 wt%, and then decreases at 20 wt%. The highest efficiency is 0.18%, achieved with 15 wt% CB-ZnO composite.

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Here are some specific observations that show that Voc increases with the amount of CB-ZnO composites up to 15 wt%, and then decreases at 20 wt%. This indicates that the CB-ZnO composite is effective in improving the charge separation in the counter electrode, but too much CB-ZnO can cause loss of recombination. This is likely because the CB-ZnO composite helps to increase the conductivity of the counter electrode, but the high probability value of CB-ZnO can block the light and reduce the efficiency.

Isc increases with the amount of CB-ZnO composite up to 20 wt%. This shows that the CB-ZnO composite is effective in increasing the light absorption in the counter electrode. Jsc increases with the amount of CB-ZnO composite up to 15 wt%, and then decreases at 20 wt%. In addition, FF is relatively constant for all samples. This indicates that the CB-ZnO composite has no significant effect on the recombination loss in the counter electrode. These results indicate that CB-ZnO composites are promising materials for counter electrodes in dye-sensitized solar cells. However, it is important to optimize the amount of CB-ZnO composites to achieve the highest efficiency.

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CHAPTER 5

5 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Investigations into the structural and optical properties of Carbon Black-ZnO composites, synthesized via solid-state methods, have revealed materials with promising potential as counter electrodes in solar energy conversion devices. Comprehensive analysis, using X-ray diffraction (XRD) and UV-Visible spectroscopy, has provided valuable insights into the crystal structure, composition and optical behavior of the composite.

The XRD pattern has served as a crystal fingerprint, confirming the successful incorporation of ZnO into the composite in its hexagonal wurtzite phase. The calculated average crystal size shows variation based on the weight percentage of carbon black, indicating that the synthesis conditions and the amount of carbon black affect crystal growth. UV-Vis spectroscopy revealed enhanced light absorption properties of Carbon Black-ZnO composites across visible wavelengths (300-800 nm). Visible absorption bands shift to higher energy states, indicating better absorption in the blue range for lower weight percentages of carbon black. In evaluating the photovoltaic properties for dye-sensitized solar cell (DSSC) applications, the efficiency of the counter electrode increased with the amount of CB-ZnO composite up to 15 wt%, indicating better charge separation. However, an excessive amount (20 wt%) leads to a decrease in efficiency, indicating a delicate balance in optimizing the amount of carbon black to avoid recombination loss.

In summary, the highest efficiency was achieved with 15 wt% CB-ZnO, highlighting the potential of the material for enhanced DSSC performance. The observed trends in open-circuit voltage (Voc), short-circuit current (Isc), and current density (Jsc) suggest that CB-ZnO composites help in improving charge separation and light absorption, even with optimal concentrations.

5.2 Recommendation

Based on the data collected, there are several recommendations made for the future of work. for future studies, some suggestions will be presented. First, to have a better photovoltaic devices must use powders in the size of nanomaterials to enlarge the surface as to increase photovoltaic efficiency. Furthermore, to have a better understanding in morphology, the next study can be analyzed using SEM. SEM offer topographical and elemental information at magnifications ranging from 10x to 300,000x, with an almost infinite depth of focus.

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APPENDIX A

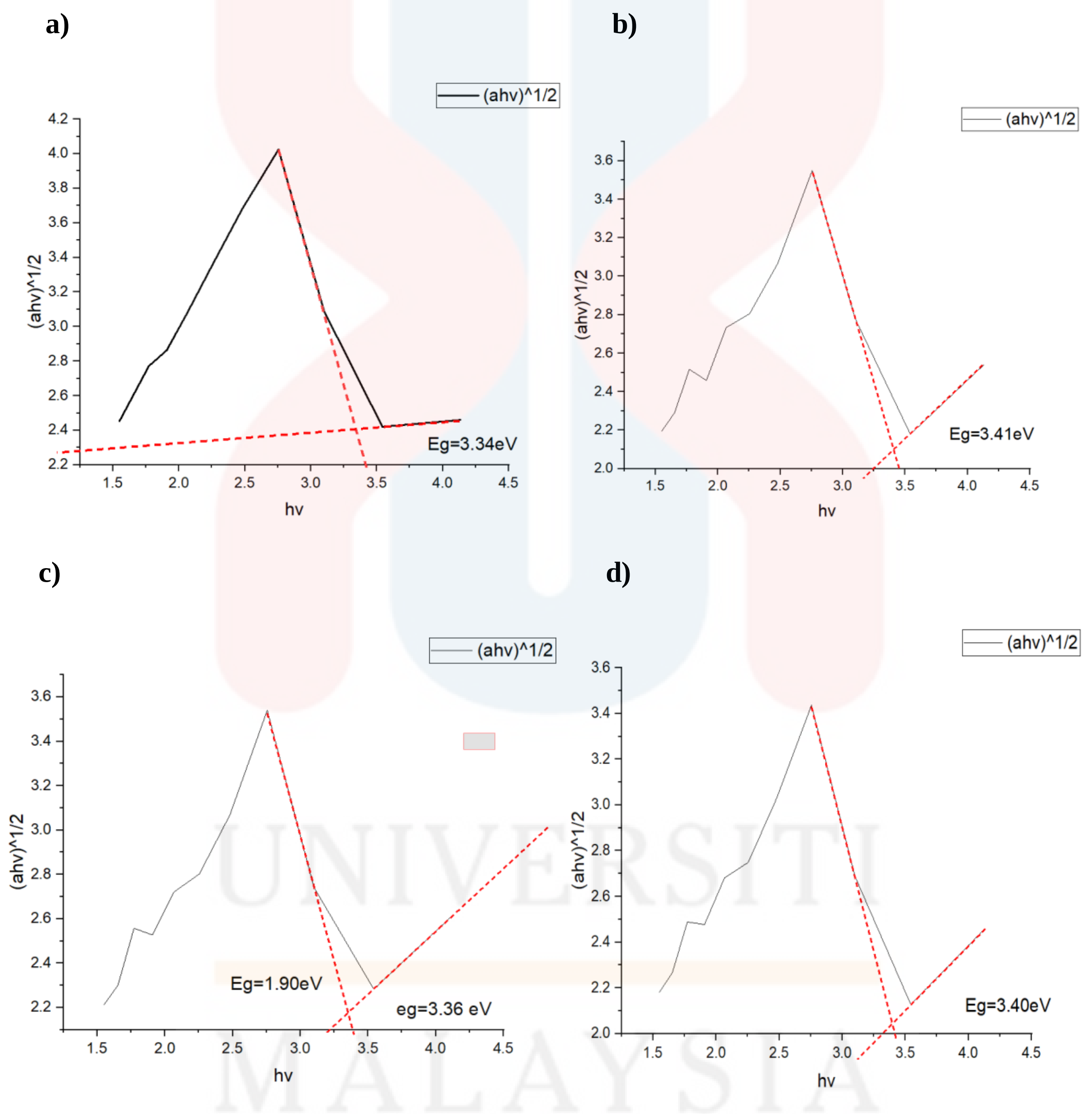

Figure A.1 : photon energy of a) 5wt% CB-ZnO, b) 10wt% CB-ZnO, c) 15wt% CB-ZnO and d) 20wt% CB-ZnO.

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APPENDIX B

Figure B.1 : Preparation carbon black from bamboo

Figure B.2 : Carbon black

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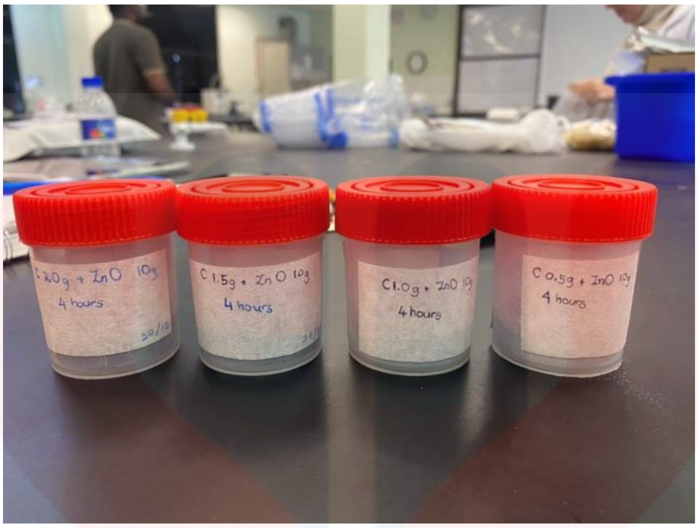

Figure B.3 : Collection of mixed composite CB-Zno

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