UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF CHELATING AGENTS ON STRUCTURAL, OPTICAL, AND ELECTROMGNETIC INTERFERENCE PROPERTIES OF COPPER SELENIDE NANOPARTICLES SYNTHESIZED VI MICROWAVEASSISTED METHOD

SHITU IBRAHIM GARBA

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By

SHITU IBRAHIM GARBA

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Doctor of Philosophy

December 2021

DEDICATION

This research work is dedicated to my late father Alhaji Garba Shitu Hadejia and to my lovely mother Hajiya Salamatu Muhammad ‘O’.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

INFLUENCE OF CHELATING AGENTS ON STRUCTURAL, OPTICAL, AND ELECTROMGNETIC INTERFERENCE PROPERTIES OF COPPER SELENIDE NANOPARTICLES SYNTHESIZED VIA MICROWAVE-ASSISTED METHOD

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Chairman : Josephine Liew Ying Chyi, PhD
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Recently, group II-VI binary semiconductor nanomaterials including copper selenide (CuSe), have garnered increased attention due to their remarkable properties that differ significantly from their bulk counterparts, as their functions are highly dependent on particle size, shape, and surface properties. To tune the overall properties of this nanoparticle, various surface modifications are required, such as capping the surface with organic, inorganic, and polymer-based chelating agents which can be used in various applications. In this work, CuSe nanoparticles were synthesized using a simple, low-cost, and environmentally friendly microwave method. Optimization of synthesis conditions such as microwave power, irradiation time, hydrazine hydrate concentration, and copper concentration was carried out to obtain a pure single-phase CuSe nanoparticle. Single-phase CuSe nanoparticles were obtained at 380 W microwave power, 20 minutes of irradiation time, 3 ml of hydrazine hydride, and 0.9:1 copper to selenium molar ratio. The effect of chelating agent concentrations on the structural, morphological, and optical properties of CuSe nanoparticles was fully investigated. Six different chelating agents were used: tartaric acid (TA), ethylenediaminetetraacetic acid (EDTA), citric acid (CA), cetyl ammonium bromide (CTAB), polyvinylpyrrolidone (PVP), and polyethylene glycol (PEG).

X-Ray Diffraction (XRD) analysis revealed that all samples formed a pure single-phase hexagonal (Klockmannite) crystal structure. The XRD analysis result is in agreement with the energy dispersive X-ray (EDX) and Raman analysis. At various concentrations of TA, EDTA, CA, CTAB, PVP, and PEG, the average crystallite size estimated using Scherer's method decreased from 73.10 to 16.10 nm, 73.10 to 16.80 nm, 73.10 to 18.20 nm, 73.10 to 43.60 nm, 73.10 to 14.00 nm, and 73.10 to 21.20 nm respectively. The Williamson-Hall method revealed an estimated crystallite size that is comparable to Scherer's method. The atomic force microscopy (AFM) and field emission scanning electron microscopy (FESEM) analysis agree with the obtained XRD results. At various

concentrations of TA, EDTA, CA, CTAB, PVP, and PEG, the optical band gap increased from 1.80 to 2.10 eV, 1.80 to 2.20 eV, 1.80 to 2.25 eV, 1.80 to 2.30 eV, and 1.80 to 2.24 eV, respectively. This is attributed to the decrease in particle size of the final product. Besides, photoluminescence (PL) maximum emission for all the samples was centered at 610 nm. The PL intensity was found to increase with increasing chelating agent concentrations in all samples.

Other than that, the effect of particle size of CuSe NPs as nanofiller loaded in the PVA polymer matrix on dielectric properties and electromagnetic interference shielding effectiveness (EMI SE) was investigated. The result showed that the dielectric constant, loss factor and loss tangent increases with the decrease in CuSe nanofiller size. This is because smaller particles fill the matrix evenly, forming a chain-like network in the PVA matrix. Moreover, the EMI SE measurement results showed that reflection loss (SE_R), absorption loss (SE_A), and total interference shielding (SE_T) decreases with an increase in frequency, which is attributed to the impedance mismatch of the EM waves as the applied frequency is increased from 8 to 12 GHz. Additionally, the nanocomposites exhibited a high absorption potential for electromagnetic waves (SE_A), but a significant portion of the EM wave was also reflected (SE_R). The contribution of SE_R and SE_A to SE_T increased as the size of the CuSe nanofiller is decreased. The nanocomposites showed the SE_T is higher than the target value of 20 dB. Thus, the results show that incorporating CuSe NPs of various sizes into a PVA polymer matrix significantly improves the total shielding effectiveness of EM waves, implying that the prepared nanocomposites can be used as lightweight, flexible, and low-cost material for electromagnetic interference shielding applications.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGARUH EJEN PENGIKAT PADA SIFAT STRUKTUR, OPTIK DAN GANGGUAN ELEKTROMAGNET UNTUK KUPRUM SELENIDE NANOZARAH YAND DISINTESIS MELALUI KAEDAH BANTUAN-GELOMBANG MIKRO

Oleh

SHITU IBRAHIM GARBA

Disember 2021

Pengerusi : Josephine Liew Ying Chyi, PhD
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Baru-baru ini, bahan nano semikonduktor binari kumpulan II-VI termasuk selenida kuprum (CuSe), telah mendapat perhatian yang lebih tinggi kerana sifat luar biasa mereka yang berbeza dengan ketara daripada rakan pukal mereka, kerana fungsinya sangat bergantung pada saiz zarah, bentuk dan sifat permukaan. Untuk menyesuaikan sifat keseluruhan zarah nano ini, pelbagai pengubahsuaian permukaan diperlukan, seperti menutup permukaan dengan bahan pengikat berasaskan organik, bukan organik dan polimer yang dapat digunakan dalam pelbagai aplikasi. Pada kajian ini, nanozarah CuSe telah disintesis menggunakan kaedah gelombang mikro yang mudah, kos rendah dan mesra alam. Keadaan sintesis optimum seperti kuasa gelombang mikro, masa penyinaran, kepekatan hidrazin hidrat, dan kepekatan kuprum telah dijalankan untuk mendapatkan nanozarah CuSe fasa tunggal tulen. Nanozarah CuSe fasa tunggal diperolehi pada kuasa gelombang mikro 380 W, 20 minit masa penyinaran, 3 ml hidrazin hidrida, dan nisbah molar kuprum kepada selenium 0.9:1. Kesan kepekatan bahan pengikat pada sifat struktur, morfologi dan optik nanozarah CuSe telah disiasat sepenuhnya. Enam bahan pengikat yang berbeza digunakan: asid tartarik (TA), asid etilenadiaminatetraasetik (EDTA), asid sitrik (CA), cetyl ammonium bromide (CTAB), polivinilpyrrolidone (PVP), dan poliethilene glikol (PEG).

Analisis XRD mendedahkan bahawa semua sampel membentuk struktur kristal heksagon (Klockmannite) fasa tunggal tulen. Keputusan analisis XRD adalah selaras dengan analisis EDX dan Raman. Pada pelbagai kepekatan TA, EDTA, CA, CTAB, PVP, dan PEG, saiz kristal purata yang dianggarkan menggunakan kaedah Scherer menurun daripada 73.10 hingga 16.10 nm, 73.10 hingga 16.80 nm, 73.10 hingga 18.20 nm, 73.10 hingga 43.60 nm, 73.10 hingga 43.60 nm, dan 73.10 hingga 21.20 nm masing-masing. Kaedah Williamson-Hall mendedahkan anggaran saiz kristal yang setanding dengan kaedah Scherer. Analisis AFM dan FESEM bersetuju dengan keputusan XRD yang diperoleh. Pada pelbagai kepekatan TA, EDTA, CA, CTAB, PVP dan PEG, jurang

jalur optik meningkat daripada 1.80 kepada 2.10 eV, 1.80 sehingga 2.20 eV, 1.80 sehingga 2.25 eV, 1.80 sehingga 2.30 eV, dan 1.80 sehingga 2.24 eV masing-masing. Keputusan ini adalah disebabkan oleh pengurangan saiz zarah produk akhir. Selain itu, pelepasan maksimum PL untuk semua sampel berpusat pada 610 nm. Keamatan PL didapati meningkat dengan peningkatan kepekatan bahan pengikat dalam semua sampel.

Selain daripada itu, kesan saiz zarah CuSe NPs sebagai pengisi nano yang dimuatkan dalam matriks polimer PVA ke atas sifat dielektrik dan keberkesanan perisai gangguan elektromagnet (EMI SE) telah dikaji. Keputusan menunjukkan bahawa pemalar dielektrik, faktor kehilangan dan tangen kehilangan meningkat dengan pengurangan saiz nanofiller CuSe. Ini kerana zarah yang lebih kecil mengisi matriks secara sama rata, membentuk rangkaian seperti rantai dalam matriks PVA. Selain itu, keputusan pengukuran EMI SE menunjukkan kehilangan pantulan (SER), kehilangan penyerapan (SEA), dan perisai gangguan keseluruhan (SET) berkurangan dengan peningkatan frekuensi, yang dikaitkan dengan ketidakpadanan impedans gelombang EM sebagai frekuensi yang digunakan, dinaikkan daripada 8 kepada 12 GHz. Selain itu, nanokomposit mempamerkan potensi penyerapan yang tinggi untuk gelombang elektromagnet (SEA), tetapi sebahagian besar gelombang EM juga dipantulkan (SER). Sumbangan SER dan SEA kepada SET meningkat apabila saiz pengisi nano CuSe dikurangkan. Nanokomposit menunjukkan SET lebih tinggi daripada nilai sasaran 20 dB. Oleh itu, keputusan menunjukkan bahawa penggabungan nanozarah CuSe yang pelbagai saiz dalam matriks polimer PVA meningkatkan keberkesanan keseluruhan perisai gelombang EM dengan ketara, membayangkan bahawa nanokomposit yang disediakan boleh digunakan sebagai satu bahan yang ringan, fleksibel, dan kos rendah untuk aplikasi perisai gangguan elektromagnet.

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This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfillment of the requirements for the degree of Doctor of Philosophy. The members of Supervisory Committee are as follows:

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LIST OF ABBREVIATIONS AND SYMBOLS

AA	Acetic acid
AFM	Atomic force microscopy
Ag_2Se	Aluminium selenide
CA	Citric acid
CB	Conduction band
CBD	Chemical bath deposition
CdS	Cadmium sulfide
CdSe	Cadmium selenide
CNT	Carbon nanotube
CTAB	Cetyltrimethyl ammonium bromide
Cu	Copper
$CuInSe_2$	Copper indium diselenide
CuS	Copper sulfide
CuSe	Copper selenide
DMF	N,N, dimethylformamide
DOS	Density of states
EDA	Ethylenediamine
EDTA	Ethylenediaminetetraacetic acid
EDX	Energy dispersive x-ray
E_g	Optical band gap
EM	Electromagnetic
EMI SE	Electromagnetic interference shielding effectiveness
EMI	Electromagnetic interference
EMP	Electromagnetic pollution

eV	Electron volt
FeSe	Iron selenide
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transform infrared spectroscopy
FWHM	Full width at half maximum
GHz	Giga Hertz
h	Planck constant
HRTEM	High resolution electron microscopy
K	Kelvin
k	Wave vector
LO	Longitudinal optic
MoSe	Molybdenum selenide
MWCNT	Multi walled carbon nanotube
NaOH	Sodium hydroxide
nm	Nanometre (10^{-9})
NPs	Nanoparticles
PANI	Polyaniline
PbSe	Lead selenide
PEG	polyethylene glycol
PL	Photoluminescence
PLA	Polylactic acid
PPy	Polypyrrole
PVA	Polyvinyl alcohol
PVDF	Polyvinylidene difluoride
PVP	Polyvinyl pyrrolidone

RS	Raman spectroscopy
Se	Selenium
SE_A	Absorption loss
SEM	Scanning electron microscopy
SE_R	Reflection loss
SE_T	Total electromagnetic shielding effectiveness
TA	Tartaric acid
Tanδ	Loss tangent
TEM	Transmission electron microscopy
TO	Transverse optical
UV-Vis.	Ultraviolet- visible
VB	Valence band
XRD	X-ray diffraction
ZnSe	Zinc selenide
ε''	Imaginary part of dielectric constant
ε'	Real part of dielectric constant

CHAPTER 1

INTRODUCTION

1.1 Nanomaterials

Nanotechnology and nanoscience have gained considerable interest in the field of research and technological advancement in the last two decades. The investigation of the properties of the material at a nanometre scale is referred to as nanoscience, whereas the design, fabrication, and application of the nanostructured material are referred to as nanotechnology. A nanoparticle is defined as a particle whose diameter is within the range of 1-100 nm. As a result of their nanometer dimension, there is an improvement in their surface to volume ratio which contribute to the enhancement of their physical and optical properties (Lalitha et al., 2012). Reducing the nanoparticle's size has a marked effect on the distribution of the energy level as the particle becomes more confined similar to a particle in a box with diminishing size, which leads to an increase in the energy separation between neighbouring energy levels. This phenomenon is more pronounced where the bandgap energy increases with reduced size and the bandgap transition starts to move to higher frequencies (Kang et al., 2000). The transformation between these states results in photon emission and absorption. Semiconductors have shown major changes in optical properties as a function of particle size (Rastei et al., 2007).

1.2 Semiconductor Nanomaterials

A semiconductor is a material whose electrical conductivity value falls between a conductor and an insulator. The conventional semiconductors have a bandgap between 1.00 and 1.50 eV, whereas wide bandgap semiconductors have a bandgap between 2.00 and 4.00 eV. The value of the bandgap of a material is dependent on particle size as well as the phase of the material obtained. Semiconducting nanofillers could be considered as a promising candidate for EMI shielding applications(Gosavi et al.,2008). The electromagnetic shielding effectiveness is primarily dependent on the conductivity as well as the dielectric and magnetic properties of a material. Semiconductors with electrical conductivity near 1 S/m are found to exhibit reasonable electromagnetic wave absorption behaviour (Sushmita et al., 2020). There are many semiconductors (e.g.: ZnO, CuSe, TiO_2, MoS_2, CoO, $BaTiO_3$, and $SrTiO_3$) that are known to have good dielectric properties which play a critical role in electromagnetic wave losses. However, some semiconductors have adequate magnetic properties (e.g., Fe_2O_3, Fe_3O_4, etc.), that support the electromagnetic wave absorption behaviour. Therefore, it is the mechanism of shielding thorough various types of losses such as dipolar, interfacial, conduction, eddy current and magnetic that will decide whether a certain semiconductor material is good for electromagnetic shielding application (Bayat et al., 2017; Cao & Hu, 2010; He et al., 2013). The shielding mechanism is dependent on the structure of each material in the polymer matrix as well as the size and morphological properties of the material (Pandey et al., 2018).

1.3 Microawave Assisted Synthesis Method

Researchers in both industries and academia are continuously challenged to come up with an environmentally friendly and safer method for the synthesis and fabrication of the desired product. Therefore, there is a need for the development of greener and sustainable methodologies for the synthesis of high-quality nanostructured materials for future use. Microwave energy is more effective in selective heating in many processes, which is beneficial to the environment because it uses less energy than traditional heating methods (Zhu & Chen, 2014). The microwave-assisted synthesis is considered to be one of the environmentally friendly novels chemical methods that relatively simplifies reactions and could be considered as another avenue for green synthesis of nanomaterials. Numerous features of microwave heating contribute to its greener characteristics, including lower energy consumption, shorter reaction time, and higher product yield. The microwave-assisted technique can be combined with several other green chemistry strategies to make green synthesis more appealing. For instance, ionic liquids, solvent-free reactions, and nontoxic precursors can be used in conjunction with microwave heating. When the energy needed for the synthesis of nanomaterial is high, it contributes to the formation of greenhouse gases which in turn leads to air pollution, global warming, and climate change (Lu et al., 2017).

1.4 Electromagnetic Interference Shielding (EMI)

On the other hand, the increasing demand for communication and electronic devices has led to the emergence of a new technological challenge called electromagnetic (EM) pollution in different sectors like military, industry, and commercial applications. To overcome this challenge, there is a need for the scientific and research community to fabricate new materials to attenuate the electromagnetic waves arising from electronics and telecommunication devices. EM wave is unwanted electromagnetic radiation that affects electrical and electronic devices. These EM radiations interfere with electronic and electrical devices and cause them to malfunction (Al-Saleh et al., 2013). It also affects the health of humans. When electromagnetic radiation falls on the human body, it tends to penetrate inside the body partially, thereby causing a net drop across the body tissues and in effect causes some electrical stress across the tissue which results in some disorders in the body. Polymer nanocomposites based materials are proven to be better than conventional metal-based materials owing to their easy processability, low density, flexibility, and corrosion resistivity. The field of polymer nanocomposites encompasses a broad variety of materials ranging from conductors and semiconductors nanofillers to a few insulating nanofillers. The commonly used conducting fillers that are used for electromagnetic interference (EMI) shielding applications are metal nanoparticle, carbon-based fillers such as carbon nanotubes (CNTs), graphene, carbon fibers (CFs), etc. (Geetha et al., 2009).

In this work, CuSe nanoparticles were synthesized via microwave-assisted synthesis technique. The effect of chelating agents' concentration on structural, morphological, and optical properties of CuSe nanoparticles is investigated. About six chelating agents were utilized which includes, tartaric acid (TA), ethylenediaminetetraacetic acid (EDTA), citric acid (CA), Cetyltrimethyl ammonium bromide (CTAB), polyvinylpyrrolidone (PVP), and polyethylene glycol (PEG). Additionally, the effect of particle size on dielectric properties and electromagnetic interference shielding effectiveness (EMI SE) of CuSe nanoparticles embedded in PVA polymer matrix was also investigated.

1.5 Significance of the Study

Nanostructured materials have created a high interest in recent years by their quantized electronic structures that lead to their fascinating properties. There is a large number of new opportunities that could be realized by dawn sizing existing structures into the nanometer scale, or by making new types of nanostructures. By decreasing the particle size and increase in surface to volume ratio can introduce many size-dependent properties. Their unique surface structure, electronic state, and largely exposed surface area greatly increase their activities for absorbing sunlight and promoting chemical reactions. The finite size of the particle confines the spatial distribution of the electrons, leading to a quantized energy level due to the quantum size effect. Such materials are extremely useful in a wide range of applications, particularly those that are size-dependent (Kolahalam et al., 2019).

In addition, the microwave-assisted synthesis of nanomaterials is a very emerging field of research that has received tremendous attention. This synthesis method can produce fine particles in the nanometer range and is intensively pursued due to its unique advantages over the remaining synthesis methods. The microwave-assisted synthesis technique is a rapid and environmentally friendly method for the formation of nanoparticles due to the fast and homogeneous heating effects of microwave energy. Compared with other synthesis methods, it has the advantage of short reaction time, high energy efficiency, ability to induce the formation of particles with smaller sizes, narrow size distribution, and high purity (Crane et al., 2013).

On the other hand, the proliferation of smart electronic devices and wireless communication in the artificial intelligent age are the source of electromagnetic pollution (EMP) which is of serious concern nowadays. The EMP creates complexities in the environment and the unwanted radiation which not only disturbs the general function of surrounding electronic systems but also threatens the well-being of humans. As a result, electromagnetic interference shielding (EMI SE) has become an unavoidable option around the world for mitigating the negative effects of electromagnetic pollution (Raagulan et al., 2020).

1.5 Problem Statement

Copper chalcogenides have recently gained a lot of attention due to their intriguing physical, chemical, and semiconducting properties, which are influenced by their chemical composition and crystalline structure, which may be tailored using intelligent synthesis conditions. Copper selenide is an essential chalcogenide material with a wide range of thermoelectric and photovoltaic energy harvesting applications (Singh et al., 2018). Furthermore, copper selenide is an intrinsic p-type semiconductor with direct and indirect bandgap energy. Non-stoichiometry in copper selenide nanoparticles impacts their electrical characteristics as well as their crystalline structure and cation exchange capabilities. Not only composition, but the arrangement of atoms in a given structural phase is also a crucial parameter to control electronic, thermal, and chemical properties of copper selenide. Because of these outstanding properties, compositional and structural control in copper selenide nanoparticles is highly desirable for their unique scientific and technological applications, posing a general challenge for researchers to develop new synthesis procedures (Mirzanezhad et al., 2019).

The preparation of nanomaterials with control over size requires a chelating agent, which primarily acts as a stabilizing agent and provides colloidal stability along with preventing agglomeration and stopping uncontrolled growth. The final morphology of a nanocrystal largely depends on the type of chelating agent that is used on the in the synthesis of nanomaterial. Thus, the use of chelating agents is key to obtaining the small-sized nanoparticles and they are frequently used in the colloidal synthesis of nanoparticles to avoid their overgrowth. Moreover, the aggregation and agglomeration of nanoparticles reduces the potential enhancement of mechanical properties in nanoparticles due to the restriction of interfacial area. Therefore, the main challenges in the production of nanoparticles include the achievement of small nanoparticles and the good dispersion of nanoparticles, which largely depends on the type of chelating agent used (Ashraf et al., 2018).

The conventional methods of manufacturing and processing of materials are becoming unneeded due to their higher energy consumption, long processing times, poor characteristics of processed materials, and increased costs. The limitations of traditional processing techniques for processing large quantities of advanced materials have compelled researchers to investigate alternative and novel manufacturing and material processing techniques that could overcome the limitations of traditional techniques. As a result, it is critical to develop a new methodology for the production of materials that can provide better properties, fewer defects, and of economic benefits in terms of energy and time savings. Microwave material processing has emerged as one of the innovative material processing methods that can meet current requirements and can deliver a better product at a lower cost and processing time.

Moreover, metallic materials with high electrical conductivity, such as copper and nickel, have been widely used as fillers in polymer matrixes in recent decades. However, metallic materials have some disadvantages, including their high weight, poor mechanical properties, and susceptibility to oxidation; these characteristics significantly limit their use. Therefore, inorganic binary nanomaterials such as CuSe, CuS, ZnSe, CdSe, CdS and so on have been the focus of attention in recent years because of their high electrical conductivity. The combination of these inorganic materials with polymer matrices can yield functional materials with enhanced electrical and thermal properties (Naghdi et al., 2018).

Conductive nanomaterials have long been the focus of research and may be employed as replacements to typical conductive and transparent metal oxides in the electrical and optoelectronic industries in the future. The use of conductive metallic nanoparticles as a thin film or coating rather than conventional conductive metal foils or metal coatings would have a significant impact on the cost, volume, weight, and mechanical properties of electronic, optoelectronic, and photovoltaic devices (Khan et al., 2019).

1.6 Research Hypothesis

1. The microwave-assisted synthesis technique is expected to help in the formation of pure single-phase CuSe nanoparticles. Microwave heating involves two main mechanisms, dipolar polarization, and ionic conduction. Microwaves generally heat any material that contains mobile electrical charges in a solvent or solid, such as polar molecules or ions. Polar molecules, such as water molecules, attempt to orient during microwave heating with the rapidly changing alternating electrical field; thus, heat is generated by molecular rotation, friction, and collision (dipolar polarization mechanism) (Arhancet et al., 2010). Therefore, it is believed that the microwave power, irradiation time, hydrazine hydride concentration, and copper precursor concentration will have a significant effect on the formation of single-phase copper selenide nanoparticles.

2. The CuSe nanoparticles prepared via microwave-assisted synthesis by varying the concentration of chelating agent is expected to affect the microstructural morphology as well as the particle size of the final products. Chelating agents are important stabilizers, which inhibit nanoparticles growth and prevent colloidal coagulation/aggregation. The chelating agent stabilizes the interface between nanoparticles and their preparation medium (Javed et al., 2020). Thus, it is believed that employing chelating agents in nanoparticle synthesis will have an impact on the microstructural morphology as well as particle szie of the final product.

3. Nanoparticles prepared via the microwave-assisted synthesis technique at different concentrations of chelating agents are expected to affect the optical properties of the final product. This is because the effect of reduced dimensionality in nanostructured systems on the electronic structure has a major impact on the energies of the highest occupied molecular orbit and the lowest unoccupied molecular orbital. The result of the transition between those states leads to an optical emission and absorption. Metals and semiconductors, in particular, exhibit great changes in optical properties as a function of particle size (Rastei et al., 2007). In semiconductor nanoparticles, the three-dimensional effect of quantum confinement may also be observed when the particle size approaches the exciton of the Bohr radius. This confinement leads to new optical characteristics. The optical properties like fluorescence emission also specifically depend on the size of the nanoparticles (Rastei et al., 2007).

4. It is believed that CuSe nanoparticles of different sizes embedded in PVA polymer matrix will affect the dielectric properties of CuSe/PVA nanocomposite. In general, composites reinforced with smaller particle size tend to possess a more significant interfacial area, leading to extra interfacial polarization, which increases the dielectric properties. Moreover, at the same filler content, the particulates in the smaller-sized filler are higher than that in the bigger sized filler. This occurrence leads to denser composites, which increases the permittivity of the overall composites (Alhaji et al.,2021).

5. It is believed that the embedding of CuSe nanoparticles of different sizes as a nanofiller in the PVA matrix will have a significant effect on the electromagnetic interference shielding performance of the CuSe/PVA nanocomposites. The filler dimensions have a significant effect on the permittivity of the nanocomposite. Certain nanostructures with a high porosity and a large surface area introduce multiple interfaces, which cause the Maxwell-Wagner effect. Additionally, several surfaces within complex geometries exhibit unsaturated bonds, which contribute to dipole polarization. Thus, multiple interfaces are advantageous for electromagnetic attenuation due to conductivity loss and polarization of the interfacial/dipole orientation.

1.7 Research Questions

Thus, this research will give answers to the following research questions based on the outlined hypothesis above.

1. What are the best synthesis conditions for making single phase copper selenide nanoparticles using the microwave-assisted synthesis method?

2. Would the addition of chelating agents at different concentrations affect the microstructural morphologies and optical properties of the as-synthesized final product?

3. Would different particle size embedded in PVA polymer matrices affect the dielectric properties of the nanocomposite?

4. Would embedding of CuSe nanoparticles of different sizes into the PVA matrix affect the electromagnetic interference shielding effectiveness of the nanocomposite?

1.8 Research Objectives

This work involves fundamental research on the effect of different chelating agents concentration on the structural and optical properties of CuSe nanoparticles synthesized via microwave-assisted technique. The final product is further used as a nanofiller in the polyvinyl alcohol (PVA) polymer matrix to investigate the effect of particle size on dielectric properties and electromagnetic interference shielding effectiveness (EMI SE) of the nanocomposite.

The primary objectives are as follows:

1) To optimize synthesis condition to form pure single-phase CuSe nanoparticles via the microwave-assisted method.

2) To investigate the effect of various chelating agents such as tartaric acid (TA), ethylenediaminetetraacetic acid (EDTA), citric acid (CA), cetyl ammonium bromide (CTAB), polyvinylpyrrolidone (PVP), and polyethylene glycol (PEG) on particle size of CuSe nanoparticles.

3) To investigate the effect of chelating agent concentrations on structural, morphological and optical properties of CuSe nanoparticles synthesized via microwave-assisted synthesis method.

4) To investigate the effect of particle size on dielectric properties and electromagnetic interference shielding effectiveness (EMI SE) of CuSe nanoparticles embedded in polyvinyl alcohol (PVA) polymer matrix.

1.9 Scope of the Research

This research will focus on gaining a better understanding of the effect of chelating agent concentration on copper selenide nanoparticles synthesized using the microwave-assisted method. The chelating agent used in this project includes tartaric acid (TA), ethylenediaminetetraacetic acid (EDTA), citric acid (CA), polyvinylpyrrolidone (PVP), polyethylene glycol (PEG) and cetyltrimethylammonium bromide (CTAB). To produce CuSe nanoparticles using the aforementioned chelating agents, a straightforward microwave-assisted method was used throughout the synthesis process. Additionally, the effect of CuSe nanoparticle size on dielectric properties and electromagnetic interference shielding (EMI SE) embedded in PVA polymer matrix is also investigated.

1.10 Thesis Outline

There are six chapters in this thesis. Chapter one starts with an overview of the research background, the significance of the study, the description of problems, and the objectives of the study. Chapter two reports the literature review; the general introduction of semiconductor materials, CuSe nanoparticles, and underlying literature on the synthesis method of semiconductor nanoparticles and related literature on the use of chelating agents for the synthesis of semiconductor nanoparticles. Chapter three is focused on the optical and electronic structure of semiconductors, theories of the dimensional semiconductor structure, microwave irradiation, and electromagnetic interference shielding. Chapter four gives a brief description of the experimental methodology and techniques used to design, synthesize and characterize CuSe nanoparticles. The microwave-assisted synthesis method used to synthesize the nanoparticles and solution casting method for the fabrication of CuSe/PVA composite is discussed in detail. A summary of the various characterization techniques used is also highlighted. This includes a description of the operation of each technique including X-ray diffraction

(XRD), Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, energy-dispersive X-ray spectroscopy (EDX), field emission scanning electron microscopy (FESEM), atomic force microscopy (AFM), UV-visible spectroscopy (UV-Vis), photoluminescence spectroscopy (PL), and rectangular wave guide for electromagnetic interference shielding application. Chapter five reports the major findings of the study, in which all the experimental results are presented, analyzed and discussed in detail. Finally, chapter six gives a summary of the results of the whole research work and suggestions for future work is also presented.

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