***STRUCTURAL AND OPTICAL PROPERTIES OF WILLEMITE NANOPARTICLES SYNTHESIZED BY POLYMER THERMAL TREATMENT METHOD***

**ALIBE IBRAHIM MUSTAPHA**

**ITMA 2019 2**

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By

**ALIBE IBRAHIM MUSTAPHA**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

**February 2019**

**DEDICATION**

This thesis solely dedicated to my beloved parent, Hon. Mustapha Ali Benishiekh and Zara Kellu Alibe.

Abstract of a thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree Doctor of Philosophy.

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**February 2019**

**Chairman : Khamirul Amin Matori, PhD**
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Willemite is an inorganic phosphor material used for optoelectronic applications. Much attention has been paid to the synthesis of willemite nanoparticles in the last two decades. This includes the application of new methods or modification of the existing ones. The present study proposes a polymer thermal treatment method involving calcination temperature between 500 to 1000 °C to fabricate the willemite nanoparticles. The effects of synthesis parameters such as the calcination temperature, calcination holding time, PVP concentration, and the precursor concentration were extensively studied and optimized. The FT–IR, Raman and the XRD analysis revealed that the samples were amorphous at room temperature and further confirmed the formation of pure willemite nanoparticles upon the calcination process. The crystallite size of the materials ranges between 21.60–32.15 nm and increases with the increment of the calcination temperatures, calcination holding time and precursor. While the crystallite size was found to be reduced from 36.70–23.80 nm with the increase in the PVP concentration (2–5 g) for the willemite nanoparticles produced at 900 °C. This is in a good agreement with the particle size determined by HR–TEM and FESEM micrographs. The $E_{opt}$ values decreased with the increased of holding times over the range of 5.39 eV at 1 h to 5.27 at 4 h. The $E_{opt}$ of the material was also found to be increasing from 5.24–5.32 eV with the corresponding increase in the PVP concentration. The PL emission spectra reveal a blue emission at 485 nm due to zinc interstitial. For all the synthesis condition, the PL emission was found to be depended on the particle size of the willemite. The current findings provide a pathway to reduce the high energy consumed in the synthesis of willemite nanoparticles, and the wide band gap energy of the material may have key potential applications for future optoelectronic lighting devices.

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

**CIRI-CIRI STRUKTUR DAN OPTIK NANOPARTIKEL WILLEMITE YANG DISINTESIS DENGAN KAEDAH RAWATAN HABA POLIMER**

Oleh

**ALIBE IBRAHIM MUSTAPHA**

**Februari 2019**

**Pengerusi : Khamirul Amin B Matori, PhD**
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Willemite adalah bahan fosfor tidak organik yang digunakan untuk aplikasi optoelektronik. Terdapat banyak perhatian telah diberikan terhadap penghasilan nanopartikel willemite sejak dua dekad yang lalu. Ini termasuk penerapan kaedah baru atau pengubahsuaian yang sedia ada. Penyelidikan ini bertujuan untuk menghasilkan nanopartikel willemite yang melibatkan suhu kalsinasi antara 500 hingga 1000 °C menggunakan kaedah rawatan termal polimer baru. Kesan parameter seperti suhu kalsinasi, masa pemangkinan kalsin, kepekatan PVP dan kepekatan prekursor telah dikaji dan dioptimumkan secara meluas. FT–IR, Raman dan analisis XRD menunjukkan bahawa sampel adalah amorfus pada suhu bilik dan selanjutnya mengesahkan pembentukan nanopartikel willemite tulen apabila proses pengkalsinan dijalankan. Saiz kristal bahan adalah antara 21.60–32.15 nm dan bertambah dengan peningkatan suhu kalsinasi, masa pemangkinan kalsinasi dan prekursor. Walaupun saiz kristal didapati berkurang dari 36.7–23.8 nm dengan peningkatan kepekatan PVP (2–5 g) untuk nanopartikel willemite terhasil pada suhu 900 °C. Hasil ini bersesuaian dan bertepatan dengan saiz zarah yang ditentukan oleh HR–TEM dan mikrograf FESEM. Nilai $E_{opt}$ yang diperolehi berkurangan dengan peningkatan tempoh masa iaitu antara 5.39 eV pada 1 jam ke 5.27 eV pada 4 jam. Tenaga jurang jalur optik bahan didapati meningkat daripada 5.24–5.32 eV dengan peningkatan yang sama dalam kepekatan PVP. Spektra pelepasan PL mendedahkan pelepasan biru pada 485 nm disebabkan oleh interstitial zink. Untuk semua keadaan sintesis, pelepasan PL bergantung kepada saiz zarah willemite. Penemuan dalam kajian ini memberi laluan baru untuk mengurangkan tenaga yang tinggi yang digunakan dalam sintesis nanopartikel willemite, dan tenaga jurang jalur lebar bagi bahan mungkin mempunyai aplikasi utama yang penting untuk peranti pencahayaan optoelektronik di masa depan.

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I certify that a Thesis Examination Committee has met on 18 February 2019 to conduct the final examination of Alibe Ibrahim Mustapha on his thesis entitled "Structural and Optical Properties of Willemite Nanoparticles Synthesized by Polymer Thermal Treatment Method" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| CB | Conduction band |
| DCCAs | Drying control chemical additives |
| D.I | Deionized |
| $E_{opt}$ | Optical band gap |
| FESEM | Field Electron Scanning Microscopy |
| FT–IR | Fourier Transforms Infrared Spectroscopy |
| HCl | Hydrochloric acid |
| HR–TEM | High Resolution transmission electron microscopy |
| JCPDS | Joint Committee on Powder Diffraction Standards |
| KM | Kubelka–Munk |
| NPs | Nanoparticles |
| PL | Photoluminescent |
| PBNM | Phosphors based nanomaterials |
| PEG | Polyethylene glycol |
| PVA | Polyvinyl pyrrolidone alcohol |
| PVP | Poly(vinyl pyrrolidone), |
| SLS | Soda lime silica |
| TGA | Thermogravimetric analysis |
| VB | Valence band |
| UV | Ultraviolet |
| UV–vis | Ultraviolet–visible spectroscopy |
| XRD | X–ray diffraction |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Phosphors based nanomaterials (PBNM) have demonstrated a substantial interest from scientist and researchers in several fields across the globe, due to their attractive physical and chemical properties (Horikoshi and Serpone, 2013). Nanomaterials such as the PBNM offers exceptional properties like quantum confinement effects couple with an increased surface area to volume ratio (Chander, 2005). These properties uniquely differentiate them from those in their bulk amount (Wu et al., 2002).

PBNM are studied broadly for numerous interdisciplinary applications (Alivisatos, 1996; Klimov et al., 2000; Chandan et al., 2018; Garakyaraghi and Castellano, 2018). Conceivably, the most interesting characteristic of these materials, from both an academic research point of view and at an industrial standpoint, is their unique size-dependent electronic nature, the ability to create structure and devices with tailor–made electronic characteristics by modifying the size of one of the constituent materials (Norris and Bawendi, 1996; Chen et al., 2017). Most often, the properties of PBNM are treated with other materials of interest in a system or device; for instance, functionalization of PBNM with biomolecules for medical imaging (Kairdolf et al., 2017; McHugh et al., 2018); creating nanostructured electronic arrays by linking several PBNM with short–chain molecules (Talapin et al., 2009; Gupta et al., 2018); producing fine nanoparticles for optoelectronic displays and sensors devices (Coe–Sullivan, 2009; Chen et al., 2018); or synthesizing PBNM systems with other semiconducting material to form cheaper next–generation photovoltaic devices (Selopal et al., 2016; Zhao et al., 2016). In each of the applications above, PBNM are exploited for their size–dependent electronic structure. Zinc silicate or willemite, for instance, is considered to be one of the earlier identified as the naturally occurring PBNM for phosphors applications. Inorganic material especially in nano–form like the willemite nanoparticles are considered to be a good host for phosphor ions because of their chemical and physical properties and have attracted attraction researcher's interest (Takesue et al., 2009).

Willemite is associated with the wide band gap oxide based materials (Omri et al., 2013; El Mir and Omri, 2014; Bharti et al., 2018; Omri et al., 2017). Willemite exists as naturally occurring minerals combined with the little number of zinc ores exhibiting a phenacite structure in which Zn–O tetrahedral shares corners with Si–O tetrahedral thus making hollow tubes appearing parallel to the plane [0001] (Klaska et al., 1978, Chang et al., 1999). This structure is a rigid lattice which enables the atoms to conquer the whole position and composed of the tetrahedral structure.

Having possessing this structure, willemite offers distiguished chemical and physical characteristics (Takesue et al., 2009). In light of this, willemite become one of the famous green phosphor material in optical and lighting devices and laser crystals applications (Veremeichik et al., 2003; Wan et al., 2005; Yu and Wang, 2009; Zaid et al., 2016; Vitkin et al., 2017; El Mir and Omri, 2018; Onufrieva et al., 2018). Willemite additional potentials applications include but not limited to, their usage as an adsorbents (Wang et al., 2012; Dai et al., 2017), optoelectronics application (Zaid et al., 2016; Effendy et al., 2017), and photonic devices (Romanov et al., 2000; Tarafder et al., 2016).

Furthermore, willemite is fame for its suitability serving as good host material used for several guest ions of rare–earth and transitional metal to attain higher effectiveness in displaying wider range of multi–colours for luminescence application (Omri et al., 2013; El Mir and Omri, 2014; Al–Nidawi et al., 2017; Effendy et al., 2017; Zaid et al., 2016; Omar et al., 2017; Omar et al., 2016; Rasdi et al., 2017;). For instance, the luminescence characteristics of rare earth ions ($RE^{3+}$) like the long emission lifetime, sharper luminescence array and the characteristic spectral behavior as they are filled by 4f shells and shielded by the outer $5s^2$ and $5p^6$ orbitals are some of the motivating drivers for their choice in nowadays optoelectronic application. For this reason, willemite doped with $RE^{3+}$ is creating major attention for its luminescence efficiency and colour purity for lighting devices (Omar et al., 2017). Furthermore, Takesue et al. (2009) reported the optical performance of willemite doped manganese ($Zn_2SiO_4:Mn^{2+}$) phosphors where the $3d^5$ electron transition in $Mn^{2+}$ ions is characterized as activating centre in the $Zn_2SiO_4$ structure. The strong green emission obtained under Ultraviolet light is directly due to the excitation from the lowest state $^4T_1(G)$ to $^6A_1(S)$ of the ground state (Lukić et al 2008; Uegaito et al., 2012; Park et al., 2015).

## 1.2 Problem Statement

The earth is globally faced with tough environmental dreadful degradations and energy resources because of continues usage of conventional fossil fuels which lead to the greenhouse effects. The great challenges of the 21st century are undoubtedly the need to create a system that reduces high energy consumption and carbon gas emission to the environment. Manufacturing industries including phosphor industry have been criticized for higher energy consumption and greenhouse gases emission, attributed to the high temperature of production (Zhang and Cheng, 2009; Canadell and Raupach, 2008). In recent years, willemite based phosphors are fascinating a remarkable attention in optical, and lighting devices (Omar et al., 2017; Rasdi et al., 2017; Mbule et al., 2017; Dang et al., 2018; Nakamura et al., 2018; Omri et al., 2018; Wei et al., 2018; Zaitseva et al., 2018). Willemite nanoparticles have previously been produced using several synthesis techniques such as the conventional sol–gel and solid state techniques. Other established methods include combustion method, a mechanical method, hydrothermal method, spray pyrolysis method wet–chemical method, and microwave method. However, most of these methods have some setback that is limiting their choices, such as high production temperature involved, complexity in procedures, the lengthier period for the reaction to take part, poisonous substances and a harmful effluent that are possibly detrimental to the environments. In light of the reason above, this study is focused on developing new environmental friendly synthesis technique, yet simple and low cost effective for producing willemite nanoparticles. Polymer thermal treatment method offers a remedy and to curb the drawback of the previous methods of synthesis. The study investigates the effects of the synthesis conditions on the formation and properties of these willemite nanoparticles by polymer thermal treatment method and followed by the characterization of their properties. The findings in this study are anticipated to find a potential application as phosphor material for optoelectronic devices.

## 1.3 The significance of the study

In recent times, willemite nanomaterials open greater prospects for creating new materials and fascinating an essential interest in phosphors and optoelectronic. A higher attention of research have been pointed on the production of willemite nanoparticles to achieve useful luminescent materials for oscilloscopes, multi–color light emitting diode and several displays panels and lighting devices (Takesue et al., 2009; Ding et al., 2015)

Willemite has been considered suitable phosphor host matrix for several guest ions such as rare earth and transitional metal as dopant ions for effective optical performance (Azman et al., 2018; Omri et al., 2018). The structure in willemite is such that the entire atoms occupying the overall position and made up of the tetrahedral structure where zinc and silicon are placed in three different fourfold crystallographic sites. Thus, such rigid lattice provides the chances to attain improved optical properties (Takesue et al., 2009; Tarafder et al., 2014).

To the best of the author’s knowledge, there are not yet available literature reports on the production of willemite based phosphor using a polymer thermal treatment technique. Thus, this work presents an optimized production of willemite nanoparticles by controlling parameters like calcination temperature and holding times, precursor and PVP concentration. Consequently, the enhanced structural and optical properties of the materials may be possible as a candidate for optoelectronics devices applications.

## 1.4 Research Objective

This study aims to fabricate and optimize willemite nanoparticles by polymer thermal treatment method. This project employs the design of appropriate metallic precursor and polymer compositions, development of the calcination process, a series of fundamental studies on the willemite crystallization and formation process.

This study was conducted based on the following objectives:

1) To determine the effects of calcination temperature on the formation, structural and optical properties of the willemite nanoparticles
2) To examine the effects of the calcination holding time on the formation, structural and optical properties of the willemite nanoparticles.
3) To study the influence of PVP concentrations on the formation, structural and optical properties of the willemite nanoparticles.
4) To investigate the impact of precursors concentrations on the formation, structural and optical properties of the willemite nanoparticles.

## 1.5 Hypothesis

Polymer thermal treatment method will reduce the problems involves in high calcination temperature and the use of lethal chemical reagent and large agglomeration in the production of willemite nanoparticles. The purity of the willemite nanoparticles produced depends on the interaction between the metallic ions and a capping agent (PVP) that undergo to the calcination process which causes the nucleation and growth of the nanoparticles. In this technique, the role of PVP is to stabilize the metallic ions, so that the agglomeration of the nanoparticles could be diminished. Higher concentration of the PVP leads to more capping ability, and produced smaller nanoparticles. Consequently, the optical properties of the willemite nanoparticles produced such as the wide band gap energy may have possible applications for future semiconductor lighting devices.

## 1.6 Scope of the study

This work is limited to the stated aim and objective which is the production of willemite nanoparticles by polymer thermal treatment method only. In the present study, willemite nanoparticles were synthesized by the polymer thermal treatment method considering several synthesis parameters such as calcination temperature and holding times, the concentration of the metal precursor and the polymer effects. Apart from that, the influence of these parameters on the microstructural, morphological and optical characteristics of the willemite nanoparticles was as well lengthily studied and discussed. The investigation of other properties like dielectric, photocatalytic, and magnetic properties of willemite nanoparticles did not fall within the scope of the study.

To realize the stated objective, the following are considered as the scopes of the study:

1) A series of metallic precursor of zinc acetate dihydrate and silicon tetraacetate ($Zn(CH_3COO)_2.2H_2O$ and $Si(OCOCH_3)_4$) with several concentration based on the stoichiometric molar ratio of Zn:Si, 1:1, 1.5:1, 2:1 and 3:1 has been used to prepare willemite nanoparticles, while keeping the PVP concentration constant.
2) Several PVP concentrations between 0–5 g in 100 ml of deionized water were used to obtain the optimum polymer concentration in the synthesis of the material.
3) The thermal decomposition and degradation properties of metallic precursor embedded in PVP before the calcination process has been measured using thermogravimetric analysis and differential thermal analysis (TGA–DTA) spectroscopy.
4) The structural and crystallinity of willemite based phosphor has been analyzed using XRD, HR–TEM, FESEM, Raman, and FT–IR spectroscopy.
5) The optical properties of willemite based phosphor have been analyzed using UV–Vis and PL spectroscopy.

## 1.7 Thesis outline

Synthesis and characterization of willemite nanoparticles by polymer thermal treatment method are the central focus and features of assessment in this study. Chapter 1 of this thesis consists of a brief background on phosphors based nanomaterials and willemite based phosphors. Other features in Chapter 1 includes the problem statement, the significance of the study, the research objectives, scope of the study and the thesis outline. The review of the previous literature reported on the production of willemite based phosphors, metal oxide nanoparticles by other researchers are covered in Chapter 2. The materials and tools used in the experiment, in addition to the detailed description of the procedure for the production of willemite based phosphor are discussed in Chapter 3. The results discussion on the effect of metallic precursor and PVP concentration, the progression of calcination temperatures and various proportion cerium doping amount on the structural and optical properties of the willemite based phosphor are studied in Chapter 4. The research conclusion and recommendations for possible future works are stated in Chapter 5. The last part of the thesis contains a list of references, publications, and conferences attended by the author.

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