UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND OPTICAL CHARACTERIZATIONS OF THERMAL LUMINESCENCE MATERIALS LiF:Mg,Cu,P AND LiF:Mn,Co,P NANOPARTICLES

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By

FAROOQ ABDULKHALEQ NAJEEB

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

March 2017

DEDICATION

"and of knowledge ye have been vouchsafed but little"
Holy Qur'an

In appreciation of their love, sacrifices, faith and eternal goodness

To my wife, my children (Rahaf and Yousif), my father & in memorial my mother

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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March 2017

Chairman : Professor Elias Saion, PhD
Faculty : Science

Varieties of materials have been used in the thermo luminescence dosimeters (TLD_S). The physical and chemical properties of these materials have shown great enhancement especially in environmental, medical and industrial applications. Thermoluminescence materials have been synthesised using different methods which include solid state, gaseous phase, hydrothermal, combustion and sol gel methods. However, the application of most of these methods on large-scale production is difficult due to the complex nature of their procedures, toxic reagents, and longer reaction time, difficult to achieve high purity, and toxic by-products, which can harm the environment.

In this research, the TLD_S was fabricated using lithium fluoride (LiF) doped with Mg/Mn, Cu/Co, which is the tissue equivalency of this material (Z_{eff}=8.04) suitable for use in personal dosimetry. Firstly, LiF:Mg,Cu,P and LiF:Mn,Co,P nanoparticles were synthesized separately using a modified co-precipitation and thermal treatment methods. In TGA, the weight of low molecular compounds is reduced at temperature ranging between 150 °C and 250 °C. Next, the factors of thermal stability including initial decomposition temperature (T_{onset}) are between 200 °C and 500 °C. Conversely, 470 °C (T_{max}) is the required temperature for major weight loss of up to 86.67% due to PVP as capping agent. The formations of LiF nanoparticles were confirmed using FT-IR analysis showing two principle absorption bands at 370 and 850 cm^{-1} which can be attributed to Li-O and F-O, respectively. The XRD patterns of the LiF nanoparticles which have been synthesized indicated the peak positions at 2θ values of 38.797°, 45.104°, 65.691°, 78.988°and 83.254° corresponding with (111), (002), (022), (113) and (222) crystalline plans, which indicate cubical lithium fluoride nanoparticles. The calculation of the average crystallite size of all samples was done using the Scherer's formula and this corresponded with the TEM images that possessing homogenous particle size distributions and morphology. It was observed that an increase in calcination temperature the average particle size increased from 1.66 nm at 450 °C to 3.34 nm at 750 °C. The UV-vis reflectance

with the increase in the calcination temperature. The energy gap was calculated to be 4.28 eV at 450 °C to 4.20 eV at 750 °C for LiF:Mg,Cu,P nanoparticles while the energy band gap of LiF:Mn,Co,P nanoparticles reduced from 4.28 eV at 450 °C to 4.21 eV at 750 °C as a result of an increase in particle size.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

STRUKTUR DAN PENCIRIAN OPTIK BAHAN NANO LUMINESEN LiF:Mg, Cu, P DAN LiF:Mn, Co, P

Oleh

FAROOQ ABDULKHALEQ NAJEEB

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Jenis bahan telah digunakan dalam dosimeter termo luminescence (TLDS). Sifat-sifat fizikal dan kimia bahan-bahan ini telah menunjukkan peningkatan yang besar terutama dalam aplikasi persekitaran, perubatan dan industry. Bahan thermoluminescence telah disintesis menggunakan kaedah yang berbeza termasuk keadaan pepejal, fasa gas, hidroterma, pembakaran dan kaedah gel sol. Walau bagaimanapun, penggunaan yang paling kaedah ini pada pengeluaran besar-besaran adalah sukar kerana sifat kompleks prosedur mereka, reagen toksik, dan masa tindak balas lebih lama, sukar untuk mencapai kesucian yang tinggi, dan toksik oleh-produk, yang boleh merosakkan alam sekitar .

Dalam kajian ini, TLDS telah direka menggunakan fluorida litium (LIF) didopkan dengan Mg / Mn, Cu / Co, yang bersamaan tisu bahan ini (Zeff = 8.04) sesuai untuk digunakan dalam dosimetri peribadi. Pertama, LIF: Mg, Cu, P dan LIF: Mn, Co, P nanopartikel telah disintesis secara berasingan menggunakan diubahsuai bersama hujan dan kaedah rawatan haba. Dalam TGA, berat sebatian molekul yang rendah dikurangkan pada suhu berjulat antara 150 °C dan 250 °C. Seterusnya, faktor kestabilan haba termasuk suhu penguraian permulaan (Tonset) adalah di antara 200 °C dan 500 °C. Sebaliknya, 470 °C (Tmax) adalah suhu yang diperlukan untuk menurunkan berat badan utama sehingga 86.67% disebabkan oleh PVP sebagai ejen menetapkan siling. Pembentukan nanopartikel LIF disahkan menggunakan analisis FT-IR menunjukkan dua band penyerapan prinsip di 370 dan 850 cm-1 yang boleh dikaitkan dengan Li-O dan F-O, masing-masing. Corak XRD daripada nanopartikel LIF yang telah disintesis menunjukkan kedudukan puncak pada 2θ nilai 38,797°, 45,104°, 65,691°, 78,988° dan 83,254° sepadan dengan (111), (002), (022), (113) rancangan kristal dan (222), yang menunjukkan kubik nanopartikel lithium fluorida. Pengiraan saiz crystallite purata semua sampel telah dilakukan dengan menggunakan formula Scherer dan ini mengenai dengan imej TEM yang memiliki taburan saiz zarah seragam dan morfologi. Ia adalah diperhatikan bahawa peningkatan suhu

pengkalsinan saiz zarah purata meningkat daripada 1.66 nm pada 450 °C hingga 3.34 nm pada 750 °C. UV-vis pantulan spektrum telah digunakan dalam menentukan jurang jalur tenaga yang didapati berkurangan dengan peningkatan suhu pengkalsinan. Jurang tenaga telah dikira dan dipastikan 4.28 eV pada 450 °C hingga 4.20 eV pada 750 °C untuk LIF: Mg, Cu, P nanopartikel manakala jurang jalur tenaga LIF: Mn, Co, P nanopartikel dikurangkan daripada 4.28 eV pada 450 °C hingga 4.21 eV pada 750 °C akibat daripada peningkatan dalam saiz zarah.

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"In the name of Allah, the most beneficent and the most merciful"

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I certify that a Thesis Examination Committee has met on 7 March 2017 to conduct the final examination of Farooq Abdulkhaleq Najeeb Al-Gburi on his thesis entitled "Structural and Optical Characterizations of Thermal Luminescence Materials LiF:Mg, Cu,P and LiF:Mn,Co,P Nanoparticles" 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 Master of Science.

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LIST OF ABBREVIATIONS

TL	Thermoluminescence
TSL	Thermo Stimulated Luminescence
TLD	Thermoluminescence Dosimeter
TSC	Thermally Stimulated Conductivity
LiF	Lithium Fluoride
Mg	Magnesium
Mn	Manganese
Cu	Copper
Co	Cobalt
P	Phosphor
PVP	Polyvinylpyrrolidone
^{60}C	Cobalt-60
^{137}Cs	cesium-137
Z_{eeff}	Effective Atomic number
LET	Linear energy transfer
eV	Electron volt
°C	Degree Celsius
a	Lattice parameter
ESR	Electron Spins Resonance
TEM	Transmission electron microscopy
FT-IR	Fourier Transform Infrared
XRD	X-ray Diffraction
TGA	Thermo Gravimetric Analysis

UV-vis	Ultraviolet visible
SEM	Scanning Electron Microscope
VB	Valence Band
CB	Conduction Band
CPE	Charged Particles Equilibrium
EthoH	Ethanol
FWHM	Full Width Half Maximum
K	Boltzmann’s constant
OSLD	Optically Stimulated Luminescence Dosimeter
PMT	Photomultiplier
R	Recombination centres
Wt	Weight

CHAPTER 1

INTRODUCTION

1.1 Introduction of the Thermoluminescence

Thermally Stimulated Luminescence (TSL) or as called generally Thermally Luminescence (TL) is the event whereby solids or semiconductors or insulators that have been exposed to ionizing radiation under situations of rising heat emit lights (Mandavia, 2011). Several of studies have been conducted in order to improve the properties of TL and a number of commercial thermoluminescent dosimeters exist for the purpose of such researches (Salah et al., 2009b). It is a known fact that a limited dose is found in the phosphor materials depending on their sensitivity to ionizing radiation. As a result of TL signal saturation while at low doses, estimating dose response accurately at high doses becomes difficult as lower signal to noise makes the assessment of the dose amount complex (Salah et al., 2007). The increasing interest in the application of TLDs for clinical, personal and environmental use has led to an extensive research on how new and high efficiency TLD materials can be produced for a large range of doses (Kitis et al., 2002). In recent times various fields especially the fields of radiation monitoring have focused on TL materials that possess nanometer dimensions like nanorods, nanowires, quantum dots and nanotubes (Shama et al., 2009a). Findings of research have shown that there is a significant difference between the physical properties of individual nanoparticles and traditional macroscopic materials. The unique characteristics of different nanocrystalline materials such as response to high linear dose and high sensitivity, which cannot be found in traditional microcrystalline phosphors, have been revealed in the findings of recent studies (Salah et al., 2009b). Research on new materials that possess sufficient dosimetric properties has been stimulated by the increase in the application of radiation processing in medical, industrial and agricultural sectors (Li et al., 2005). Also, due to the fact that unique features, which cannot be gotten from conventional macroscopic materials, have been found in nanostructure materials, interest in nanosized phosphors has increased because of its ability to detect high energy ionizing radiations (Kortov, 2010). The increase in their surface to volume ration, which transforms the electronic structure as a result of quantum confinement effect, is responsible for the unique properties which they possess. The surface states are of great importance to the physical characteristics particularly the nanoparticles optical characterization (Sharma et al., 2011). As the particles decrease, an increase in the surface area to size ratio and the surface states occurs while a reduction in the excited emission occurs through recombination of non-radiative surface. Further study of the TL features of various TL nanomaterials for high dose ionizing can benefit from the remarkable results of preliminary derived from such nanomaterials (Sahare et al., 2007; Salah, 2011).

In recent times, researchers have conducted studies on LiF as a radiation dosimeter which they used in measuring the doses absorbed by workers that work in the radiation fields for in vivo dosimetry as a way of overcoming the limitations of spatial resolution of existing dosimetry systems (Yaakob et al., 2011a,b,; Hashim et al., 2009;). The presence of reasonable atomic quantity in lithium fluoride compounds seems promising for medical applications as well as personal dosimeters (Mayles et al., 2007). One of the materials that is suitable but less researched is LiF:Mg,Cu,P; the use of this material can be employed in the detection of high and low-dose ionizing radiation for radiation dosimetry.

1.2 Problem Statement

It is widely accepted that TLD is the most frequent used technique of radiation dosimetry due to its cost-effectiveness. More so, it is the commonly used technique for regular monitoring of occupational radiation exposure (Portal, 1986). The presence of particular dopants must enhance the TL signal considering the fact that dopants serve as the defect centers that provide TL signal. TL materials structure is also regarded as a huge problem for the performance of TL and so therefore, the requirement for higher performance of TL is a well-established structure (glow curve).

This thesis presents an extensive investigation of the morphological, structural, thermal and optical properties of LiF: Mg, Cu, P and LiF: Mn, Co, P at different calcination temperatures in a modified co-precipitation and thermal treatment methods and cost effective material for TLD application.

1.3 Scope of the study

This study was conducted under different synthesizing conditions, processes of surface modification and the thermoluminescence features for the development of new nanomaterial for radiation dosimetry. The earlier focus of this thesis is the examination of the optical features of doped and un-doped lithium fluoride nanocrystals used in monitoring radiation in various dosimetry areas of low to high level of exposure. The techniques, which were used in the production of this new nanomaterial, include co-precipitation technique, synthesis approach and heat treatment. Through the use of co-precipitation technique small-sized, homogenous and amorphous particles with more homogeneity and less-broader distribution is used in this study for the production of the new nanomaterial. The amorphous particles were modified to nanocrystalline particles using heat treatment. The aim of this modification was to enhance their efficiency in dosimetric applications.

An epitaxial organic layer of polyvinyl pyrrolidone was formed for the purpose of modifying the surface of the particles. The particles were surrounded by the formed layer, which exhibited good level of control of the morphology of nanoparticles that were synthesized after undergoing heating process as well as increased stability of particles which were synthesized from agglomeration. The shape and volume of the produced nanoparticles were greatly influenced by the conditions of experiment like calcination temperatures. Due to the fact that the properties of prepared powder have great influence on the luminescence properties of materials, the researcher in this study used an easy method to prepare TL materials as a new technique as well as cost effective to produce luminescent material rather than using the traditional solid states methods. In the precipitation stage, PVP was adjusted to water ratio 3 wt% and afterwards the newly produced nanoparticles went through the process of heating. In the second step, various calcination temperatures ranging from 450 to 750 C° were used in the detection of the best conditions for synthesizing lithium fluoride phase structure. Furthermore, the use of maximum calcination temperature was employed in the development and characterization of doped nanoparticles. The morphological, structural, thermal and optical properties of TL materials are investigated at different calcination temperatures. The last stage involves the study of the dosimetric capability of synthesized doped and un-doped lithium fluoride nanoparticles subjected to a wide range of gamma exposures was not able to investigated due to unavailability of TL reader machine during this study.

1.4 Objectives of Research

The objectives of this study are as given below:

I. To conduct the synthesis lithium fluoride based: LiF:Mg,Cu,P and LiF:Mn,Co,P nanoparticles using a modified co-precipitation and thermal treatment method.

II. To present an extensive investigation of the morphological, structural, thermal and optical properties of LiF:Mg,Cu,P and LiF:Mn,Co,P nanoparticles.

1.5 Thesis outline

This thesis includes of six chapters. Chapter 1 contains the research introduction, scope, problem statement and study objectives. In chapter II the historical background of thermoluminescence dosimetry is discussed alongside a review of related literature on bulk and nano thermoluminescence dosimetry materials and their thermoluminescence properties on medical, individual and environmental radiation monitoring. Chapter III contains a discussion of the basic characteristics and structural properties of lithium fluoride families, the physical principles of the phenomenon of thermoluminescence as well as the theories related to TL parameters. Chapter IV contains the methodology and materials of the study, experimental details of method of synthesis as well as the description of the different characteristics of the various techniques used in the study. The major of the thesis is contained in chapter V in which the results of experiments are also presented, analyzed and discussed extensively. Chapter VI, which is the last chapter, presents the conclusion, summary of study and recommendation for future study.

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