UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF LITHIUM FLUORIDE-DOPED Mg/ Cr, Cu/ Ni NANOPARTICLES

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By

ALI JASSEM ABDULHUSIAN

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

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DEDICATION

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

I would like to dedicate my thesis to my dear mother, father and

To my wife and my children (Rawan and Rahaf)

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

Tissue equivalent thermoluminescent dosimeters (TLD_S) are an effective device to measure low and high absorbed doses of ionizing radiation in protected area, medical and industrial applications or as a personal monitoring dosimeter. A number of commercially available TLD_S are common for this purpose where the TL intensity is proportional to absorbed dose but they are of a narrow dose range. In this study effort were made to enhance the present TL performance of these materials to a wider dose range by employment of nanosynthesis method and introducing impurities to the TL materials. In this research the TLD_S are fabricated from lithium fluoride doped by Mg/Cr, Cu/Ni and phosphor. LiF:Mg,Cu,P and LiF:Cr,Ni,P nanocrystales were synthesized by thermal treatment method from aqueous solution including lithium chloride (LiCl), ammonium fluoride (NH_4F), deionized water, with the doping magnesium nitrate ($MgNO_3$), copper nitrate ($CuNO_3$), chromium nitrate ($CrNO_3$), nickel nitrate ($NiNO_3$) and ammonium hydrogen phosphate ($(NH_4)H_2PO_4$) and polyvinyl pyrrolidone as surfactant agent. The samples were annealed from 723 to 1023 K. The characterization of the prepared samples of LiF:Mg,Cu,P and LiF:Cr,Ni,,P were done by using X- ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), Transmission Electron Microscopy (TEM), (UV-vis) and Thermo Gravimetric Analysis (TGA). In TGA, the solvent evaporation caused weight loss in synthesized nanoparticles which had initial weight of 53.46 °C, the weight loss was about 5.19%. The temperature for initial decomposition (T_{onset}) is between (250 °C and 500 °C). Nevertheless, the greatest weight loss of 86.67% occurs at temperature of 445.53°C (T_{max}) as a result of loss of a part of the organic material likes CO_2 and gases. The XRD patterns of synthesized LiF nanoparticles showed the peak positions at 2θ values of 38.797°, 45.104°, 65.691°, 78.988°and 83.254° matching with (111), (002), (022), (113) and (222) crystalline plans. The average crystallite size of all samples was calculated from the line broadening of the diffraction peaks and most intense using Scherer's formula. The TEM images which show cubical lithium fluoride nanoparticles with uniform morphology and particle size distributions. The TEM results showed that the particle size increased with the calcination

temperature increases from 1.29 nm at 723 K to 3.19 nm at 1023 K. The FT-IR analysis proves two principle of absorption band around 350 and 750 cm^{-1} which attributed to the Li-O and F-O respectively. The band gap energy was determined from UV-vis reflectance spectra were found to decrease with increase in calcination temperature from 4.28 eV at 723 K to 4.20 eV at 1023 K in LiF:Mg,Cu,P nanocrystals while in LiF:Cr,Ni,P nanocrystals the band gap decreased from 4.26 eV at 723 K to 4.22 eV at 1023 K due to particle size increased.

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

SINTESIS DAN PENCIRIAN LITHIUM FLUORIDA DIDOPKAN Mg/Cr, Cu / Ni NANOPARTIKEL

Oleh

ALI JASSEM ABDULHUSAIN

Mac 2017

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Tisu dosimeter thermoluminescent setara (TLDS) adalah alat yang berkesan untuk mengukur dos diserap rendah dan tinggi sinaran mengion dalam kawasan perlindungan, aplikasi perubatan dan industri atau sebagai pemantauan dosimeter peribadi. Beberapa boleh didapati secara komersial TLDS adalah biasa untuk tujuan ini di mana intensiti TL adalah berkadar dengan dos diserap tetapi mereka julat dos yang sempit. Dalam usaha kajian ini telah dibuat untuk meningkatkan prestasi TL ini bahan-bahan ini untuk julat dos yang lebih luas oleh guna kaedah nanosynthesis dan memperkenalkan kekotoran kepada bahan-bahan TL. Dalam kajian ini yang TLDS adalah direka dari fluorida litium didopkan dengan Mg/Cr, Cu/Ni dan fosfor. LiF:Mg,Cu,P dan LiF:Cr,Ni,P nanocrystales telah disintesis melalui kaedah rawatan haba daripada larutan akueus termasuk litium klorida (LiCl), ammonium fluorida (NH_4F), air ternyahion, dengan nitrat doping magnesium ($MgNO_3$), nitrat tembaga ($CuNO_3$), nitrat kromium ($CrNO_3$), nitrat nikel ($NiNO_3$) dan fosfat ammonium hidrogen ((NH_4) H_2PO_4) dan polyvinyl pyrrolidone sebagai ejen surfactant. Sampel anil 723-1023 K. Pencirian sampel bersedia daripada LiF:Mg,Cu,P dan LiF:Cr,Ni,P telah dilakukan dengan menggunakan X- ray Diffraction (XRD), Fourier Transform Infrared Spektroskopi (FT-IR), Bahagian Penghantaran Electron Microscopy (TEM), (UV-vis) dan Thermo gravimetrik Analisis (TGA). Dalam TGA, penyejatan pelarut menyebabkan kehilangan berat badan dalam nanopartikel disintesis yang mempunyai berat badan awal 53,46 °C, kehilangan berat badan adalah kira-kira 5.19%. Suhu penguraian permulaan (Tonset) adalah antara (250 °C dan 500 °C). Walau bagaimanapun, penurunan berat badan yang paling besar 86.67% berlaku pada suhu 445,53 °C (Tmax) akibat daripada kehilangan sebahagian daripada bahan organik suka CO_2 dan gas. Corak XRD nanopartikel LiF disintesis menunjukkan kedudukan puncak pada 2θ nilai 38,797°, 45,104°, 65,691°, 78,988° dan 83,254° sepadan dengan (111), (002), (022), (113) dan (222) rancangan kristal. Purata saiz crystallite semua sampel dikira dari memperluas garis puncak pembelauan dan paling sengit menggunakan formula Scherer ini. Imej-imej yang menunjukkan TEM kubik nanopartikel lithium fluorida dengan morfologi seragam dan taburan saiz zarah. Keputusan TEM

menunjukkan bahawa saiz zarah meningkat dengan peningkatan suhu pengkalsinan dari 1.29 nm pada 723 K kepada 3.19 nm pada 1023 K. Analisis FT-IR membuktikan dua prinsip band penyerapan sekitar 350 dan 750 cm^{-1} yang disebabkan oleh Li-O dan FO. Tenaga jurang jalur ditentukan daripada UV-vis spektrum pantulan didapati berkurangan dengan peningkatan suhu pengkalsinan dari 4.28 eV pada 723 K hingga 4.20 eV di 1023 K dalam LiF:Mg,Cu,P nanokristal manakala di LiF: Cr, Ni, P nanokristal jurang jalur menurun daripada 4.26 eV pada 723 K kepada 4.22 eV di 1023 K kerana saiz zarah meningkat.

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I certify that a Thesis Examination Committee has met on 10 March 2017 to conduct the final examination of Ali Jassem Abdulhusian on his thesis entitled "Synthesis and Characterization of Lithium Fluoride-Doped Mg/Cr, Cu/Ni 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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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

TL	Thermoluminescence
TSL	Thermo Stimulated Luminescence
TLD	Thermoluminescence Dosimeter
TSC	Thermally Stimulated Conductivity
LiF	Lithium Fluoride
Mg	Magnesium
Cr	Chromium
Cu	Copper
Ni	Nickel
P	Phosphor
PVP	Polyvinyl pyrrolidone
^{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
OSLD	Optically Stimulated Luminescence Dosimeter
PMT	Photomultiplier
R	Recombination centres
wt	Weight
K	kelvin

CHAPTER 1

INTRODUCTION

1.1 Background of study

One of the most relevant tasks in health physics and radiation therapy is radiation dosimetry. The prediction of biological results is the major purpose of radiation dosimetry. In radiation dosimetry it is important that the dose for human tissues or water be known even though majority of radiation detectors cannot be equated to tissue. However, this may not be much of a concern for megavoltage photons but problems can emerge with photons of low to medium energy. This problem is as a result of heavy reliance of the photoelectric effect on the material's effective atomic number which energy is deposited into by ionizing radiation. One of the techniques of radiation which has among the wide range of radiation dosimeters that may be used in radiation treatment become a standard technique for radiation dosimetry is thermoluminescence dosimetry (TLD). In recent times the use of radiotherapy has been employed in the destruction of cancer. It is used as one of the cancer-destroying modalities so that the possibility of total destruction of the tumour is maximized by applying high dose of radiation to the cancer tissue (Oberhofer & Scharmann., 1981). Characteristics like long high sensitivity, optical fading, energy dependence, large linearity between thermoluminescence (TL) signal and dose should be possessed by TL material which will be used for dosimetric purposes. High glow peak temperatures as well as deep electron traps which enhance stability for months are associated with majority of commercially available. However, these characteristics pose an unending challenge for TLD. More so, the TL materials which are tissue equivalent possessing a Z_{eff} that is close to biological tissue are not many (Li & Harris., 2005).

1.2 Significant of research

Research on new materials that possess sufficient dosimetric properties have been driven by the increase in the application of radiation processing in medicine, agriculture and industrial use (Li & Harris, 2005). It has been found that conventional microscopic materials do not possess the unique features which nanostructured materials have; this can explain the reason why there is a growing interest in nano-sized phosphors. One of the unique capabilities of nano-sized phosphors is their applicability in the high energy ionizing radiation detection (Kortov, 2010). These unique properties are derived of the change in the electronic structure and increase in their surface to volume ratio and caused by effect of quantum confinement. The surface states are crucial to the physical features particularly the Nanoparticles optical properties (Sharma et al., 2011). An increase in the surface state and surface to volume ratio as well as a reduction in excited emission through non-radiative surface recombination occurs when particles become smaller. Further study of the TL properties of various TL nanomaterials used in high dose ionizing radiation can be conducted using the initial results generated from such nanomaterials (Sahare et al., 2007; Salah, 2011). Fluoride compounds are promising candidates that can be applied in personnel and medical dosimeters because of the effective atomic number they possess which can be compared with human tissue (Mayles et al., 2007). One of the materials that are most suitable yet under investigated is lithium fluoride (LiF). This material is capable of detecting high-dose ionizing radiation for radiation dosimetry.

1.3 Problem Statement

One of the popular and widely used techniques of radiation dosimetry which is also affordable is TLD. It is also known as the most common technique applied in monitoring occupational radiation exposure (Portal, 1986). Although there are more than 2000 TL materials available, only 8 are used as they are more appropriate for measuring radiation dose. Four of them, lithium fluoride (LiF), lithium borate ($Li_2B_4O_7$), beryllium oxide (BeO) and magnesium borate (MgB_4O_7) they have low atomic number (Z_{eff}), a respond similar of human tissue (7.4) and they are used for medical application as well as for personal monitoring for industrial application. The other four materials over-respond due to their higher (Z_{eff}). Thus, they have higher sensitivity and characterized as non-tissue equivalent material. These materials are calcium sulphate ($CaSO_4$), calcium fluoride (CaF_2), aluminium oxide (Al_2O_3) and magnesium orthosilicate (Mg_2SiO_4) and are used for environmental monitoring. Specific dopants must be used to enhancing the TL signal to improve its cost-effectiveness because of the fact that dopants serve as defect centres that produce the TL. However, due to non-availability of TLD reader, in this study we only examines the morphological, structural and optical properties of LiF:Mg,Cu,P and LiF:Cr,Ni,P at various annealing temperatures by co-precipitation and thermal treatment methods.

1.4 Scope of Study

In this study the morphological, structural and optical properties of doped and un-doped lithium fluoride (LiF) nanocrystals at various annealing temperatures. The new nanomaterial developed in this study is developed using a new approach of synthesis which is a combination of co-precipitation technique and thermal heat. Small-sized particles that are homogenous and amorphous possessing narrow size distribution alongside more uniformity are produced using co-precipitation. The amorphous particles were modified to nanocrystalline particles for better performance in dosimetric applications through the use of heat treatment. An epitaxial organic layer of polyvinyl pyrrolidone was formed in order to modify the surface of the particles. The particles were surrounded by this layer that was formed; good control of the morphology of nanoparticles that were synthesized subsequent to heating was displayed by the particles. Particles that were synthesised from agglomeration exhibited increased stability.

The size and shape of newly produced nanoparticles are influenced by conditions of experiment like annealing temperature, capping agent concentration and annealing time. The characteristics of prepared powder are believed can greatly influence the luminescence properties of materials. Several attempts have made to produce particles that have homogenous distribution, regular shapes. An easy method of preparing TL materials was investigated in the first part of this study as novel method of developing luminescent nanomaterials rather than the traditional solid states technique. PVP was adjusted to water weight ratio 3% in the precipitation step in order to monitor the effect of PVP concentration to the nanoparticles obtained from this procedure after heat treatment. In order to determine the ideal condition of synthesising fluoride phase structure, the second step involved annealing at different temperatures ranging from 723 to 1023 K.

1.5 Study objectives

1- To prepare un-doped and magnesium/chromium Mg/Cr, copper/nickel Cu/Ni doped lithium fluoride (LiF) nanoparticles.
2- To characterize the physical properties (structural and optical) of LiF:Mg,Cu,P and LiF:Cr,Ni,P nanoparticles.

1.6 Thesis Outline

The entire thesis is made up of six chapters and the contents of the chapter are given as follows: Chapter one consist of the overview of the research, background of study, problem statement, scope of study and objectives. Chapter two contains a review of literature on radiation dosimetry of large and nanomaterials as well as their properties. In chapter three the basic characteristics and structural properties of lithium fluoride families are discussed alongside the physic fundamental of TL phenomenon. More so, theories related to TL parameters are discussed. The study design and experimental procedure is discussed in chapter four. Chapter five is the major part of this work in which the analysed results of experiments performed are discussed in detail. In the last chapter which is chapter six, the conclusion, summary and suggestion for future work is presented.

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