

## TABLE OF CONTENTS

	<b>Page</b>
<b>ACKNOWLEDGEMENTS</b>	iii
<b>ABSTRACT (ENGLISH)</b>	iv
<b>ABSTRACT (THAI)</b>	vi
<b>LIST OF TABLES</b>	xii
<b>LIST OF LIST OF FIGURES</b>	xiii
<b>ABBREVIATIONS AND SYMBOLS</b>	xviii
<b>CHAPTER 1 INTRODUCTION</b>	
1.1 Introduction	1
1.2 Rare earth elements	4
1.3 Lanthanide phosphate	6
1.3.1 Structure	7
1.3.2 Photoluminescence properties	8
1.3.3 Application	12
1.4 Microwave irradiation method	14
1.4.1 Microwave irradiation	14
1.4.2 Microwave heating	18

	<b>Page</b>
1.4.3 Fundamental of Microwave Synthesis	21
1.4.4 Benefits of Microwave Chemistry	25
1.5 Hydrothermal / Solvothermal method	29
1.6 Literature review	31
1.7 Research Objectives	35
<b>CHAPTER 2 EXPERIMENTAL PROCEDURE</b>	
2.1 Chemical reagents, equipments and instruments	36
2.1.1 Chemical reagents	36
2.1.2 Equipments and instruments	36
2.2 Experimental procedure	38
2.3 Characterization	40
2.3.1 X-ray diffraction	40
2.3.2 Fourier transform infrared spectrometer	41
2.3.3 Field Emission Scanning Electron Microscopy	42
2.3.4 Transmission Electron Microscopy	43
2.3.5 Photoluminescence Spectroscopy	44
2.3.6 Ultraviolet- Visible Near-Infrared Spectroscopy	45

### CHAPTER 3 RESULTS AND DISCUSSION

3.1	Synthesis of Lanthanum Phosphate by microwave irradiation method	46
3.1.1	Formation of Lanthanum Phosphate complex	46
3.1.2	X-ray diffraction	47
3.1.3	Fourier transforms infrared spectroscopy	49
3.1.4	Scanning electron microscope	50
3.1.5	Transmission electron microscope	54
3.1.6	Possible formation mechanism of Lanthanum Phosphate complex	58
3.1.7	Ultraviolet-visible near-infrared spectroscopy	60
3.1.8	Photoluminescent spectroscopy	61
3.2	Synthesis of Cerium Phosphate by microwave radiation method	63
3.2.1	Formation of Cerium Phosphate	63
3.2.2	X-ray diffraction	64
3.2.3	Fourier transform infrared spectroscopy	66
3.2.4	Scanning electron microscope	67
3.2.5	Transmission electron microscope	71
3.2.6	Possible formation mechanism of Cerium Phosphate complex	74

	<b>Page</b>
3.2.7 Ultraviolet-visible near-infrared spectroscopy	76
3.2.8 Photoluminescent spectroscopy	78
<b>CHAPTER 4 CONCLUSIONS AND SUGGESTIONS</b>	
4.1 Conclusions	79
4.1.1 Microwave irradiation method	79
4.1.2 Synthesis of LaPO <sub>4</sub> by microwave irradiation method	79
4.1.3 Synthesis of CePO <sub>4</sub> by microwave irradiation method	80
4.2 Suggestions	82
<b>REFERENCE</b>	83
<b>APPENDICES</b>	
APPENDIX A Joint committee on powder diffraction standard of LaPO <sub>4</sub>	90
APPENDIX B Joint committee on powder diffraction standard of CePO <sub>4</sub>	93
<b>CURRICULUM VITAE</b>	104
<b>INTERNATIONAL PUBLICATION</b>	106

## LIST OF TABLES

Table		Page
1.1	Microwave Frequency Bands	16
1.2	Comparison of Reaction Duration	25
1.3	Comparison of Yields	26

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่  
Copyright© by Chiang Mai University  
All rights reserved

## LIST OF FIGURES

<b>Figure</b>	<b>Page</b>	
1.1	Pattern of world consumption of Rare earth elements	6
1.2	View of the LnPO <sub>4</sub> structures in: (a) the hexagonal phase and (b) the monoclinic phase, showing the connection of the cerium atom to the PO <sub>4</sub> <sup>3-</sup> tetrahedron	7
1.3	The process of photon excitation followed by photon emission is called Photoluminescence	8
1.4	Schematic representation of photophysical processes in lanthanide(III) complexes (antenna effect)	10
1.5	The electromagnetic spectrum	17
1.6	A microwave	17
1.7	Schematic of sample heating	18
1.8	Schematic of sample heating by microwaves	19
1.9	Reaction Coordinate	21
1.10	Methods of Heating by Microwave Radiation	22
1.11	Uniform Heating through Microwave Irradiation	27
1.12	Autoclave for used in Hydrothermal/Solvothermal method	30
2.1	Schematic diagram used for preparation of LaPO <sub>4</sub> (and CePO <sub>4</sub> )	39
2.2	X-ray diffractometer	40

<b>Figure</b>	<b>Page</b>
2.3 Fourier transform infrared spectrometer	41
2.4 Field Emission Scanning Electron Microscope	42
2.5 Transmission Electron Microscope	43
2.6 Photoluminescence Spectrophotometer	44
2.7 Ultraviolet- Visible Near-Infrared Spectrometer	45
3.1 Molecular structure of Lanthanum Phosphate	46
3.2 XRD patterns of LaPO <sub>4</sub> at pH values of 1-6 synthesized by microwave radiation method at 180 W for 60 min.	48
3.3 FT-IR spectra of LaPO <sub>4</sub> synthesized by microwave radiation method at the pH of 1, 3 and 5.	49
3.4 SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 6 by microwave radiation at 180 W for 60 min.	51
3.5 SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 5 by microwave radiation at 180 W for 60 min.	52
3.6 SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 4 by microwave radiation at 180 W for 60 min.	52
3.7 SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 3 by microwave radiation at 180 W for 60 min.	53
3.8 SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 2 by microwave radiation at 180 W for 60 min.	53

Figure		Page
3.9	SEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 1 by microwave radiation at 180 W for 60 min.	54
3.10	TEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 1 by microwave radiation at 180 W for 60 min.	55
3.11	TEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 2 by microwave radiation at 180 W for 60 min.	56
3.12	HRTEM image of growth direction of LaPO <sub>4</sub> synthesized in the solution with the pH of 1 by microwave radiation at 180 W for 60 min.	56
3.13	HRTEM image of layer of LaPO <sub>4</sub> synthesized in the solution with the pH of 1 by microwave radiation at 180 W for 60 min.	57
3.14	Schematic diagram for the formation of LaPO <sub>4</sub> products.	58
3.15	UV-vis NIR absorption of LaPO <sub>4</sub> nanorods synthesized by microwave radiation at 180 W for 60 min in the solution with the pH of 1.	60
3.16	PL spectrum of LaPO <sub>4</sub> nanorods synthesized by microwave radiation at 180 W for 60 min in the solution with the pH of 1.	62
3.17	Formation of Cerium Phosphate	63
3.18	XRD patterns of CePO <sub>4</sub> synthesized in the solutions with different pH values by microwave radiation at 180 W for 60 min.	64



Figure		Page
3.19	FTIR spectra of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 1, 3 and 5.	67
3.20	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 5 by microwave radiation at 180 W for 60 min.	68
3.21	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 4 by microwave radiation at 180 W for 60 min.	69
3.22	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 3 by microwave radiation at 180 W for 60 min.	69
3.23	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 2 by microwave radiation at 180 W for 60 min.	70
3.24	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 1.5 by microwave radiation at 180 W for 60 min.	70
3.25	SEM image of CePO <sub>4</sub> synthesized by microwave radiation method at the pH of 1 by microwave radiation at 180 W for 60 min.	71
3.26	TEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 1 by microwave radiation at 180 W for 60 min.	72
3.27	TEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 1.5 by microwave radiation at 180 W for 60 min.	72
3.28	TEM image of LaPO <sub>4</sub> synthesized in the solution with the pH of 2 by microwave radiation at 180 W for 60 min.	73
3.29	Schematic diagram for the formation of CePO <sub>4</sub> products.	74

Figure		Page
3.30	(a) UV-vis absorption, and (b) $(\alpha h\nu)^2$ vs $h\nu$ curve of $\text{CePO}_4$ nanorods synthesized in the solution pH of 1, using microwave radiation at 180 W for 60 min.	77
3.31	PL spectrum of $\text{CePO}_4$ nanorods synthesized in the solution pH of 1, using microwave radiation at 180 W for 60 min.	78

### ABBREVIATIONS AND SYMBOLS

$^{\circ}\text{C}$  = Degree Celsius

nm = Nanometer

$\mu\text{m}$  = Micrometer

ml = Milliliter

min = Minute

sec = Second

$\lambda$  = Wavelength

cm = Centimeters

W = Watt

$\alpha$  = Alpha

$\beta$  = Beta

$\gamma$  = Gamma

$\text{\AA}$  = Angstrom

h = Hour

La = Lanthanum

Ce = Cerium

FTIR = Fourier Transform Infrared Spectroscopy

PL = Photoluminescence

SEM = Scanning Electron Microscopy

ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่

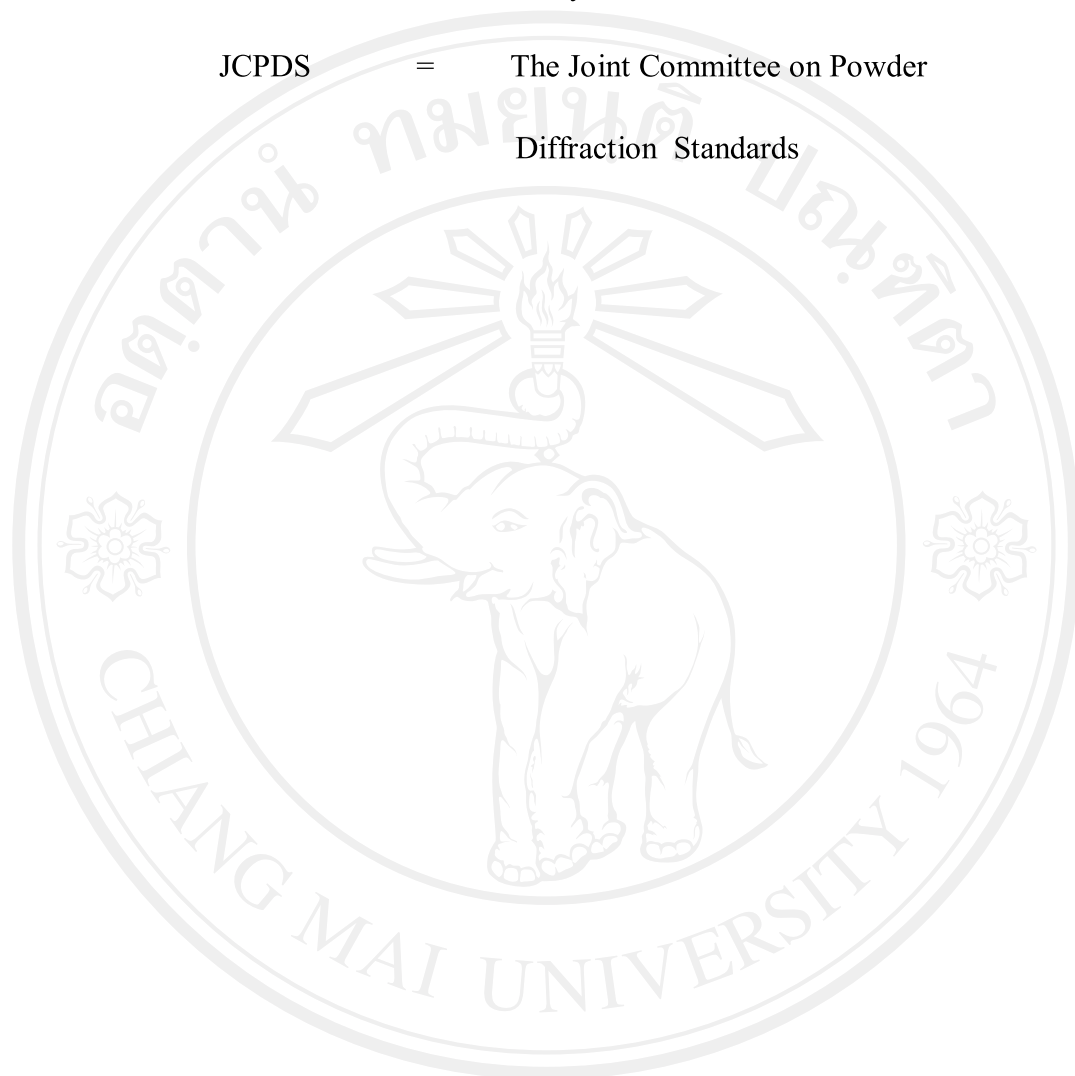
Copyright © by Chiang Mai University

All rights reserved

TEM = Transmission Electron Microscopy

XRD = X-ray Diffraction

JCPDS = The Joint Committee on Powder  
Diffraction Standards



ลิขสิทธิ์มหาวิทยาลัยเชียงใหม่

Copyright© by Chiang Mai University

All rights reserved