APPENDICES

APPENDIX A

Joint committee on powder diffraction standard of $LaPO_4$

Name and formula

Reference code:	00-004-0635
PDF index name:	Lanthanum Phosphate
Empirical formula:	LaO_4P
Chemical formula:	$LaPO_4$

Crystallographic parameters

Crystal system:	Hexagonal
Space group:	P6222
Space group number:	180
a (?):	7.0420
b (?):	7.0420
c (?):	6.4490
Alpha (?):	90.0000
Beta (?):	90.0000
Gamma (?):	120.0000
Calculated density (g/cm^3):	4.12
Volume of cell (10^6 pm^3):	276.96
Z:	3.00
RIR:	-

Subfiles and Quality

Subfiles: Inorganic

Quality: Blank (B)

Comments

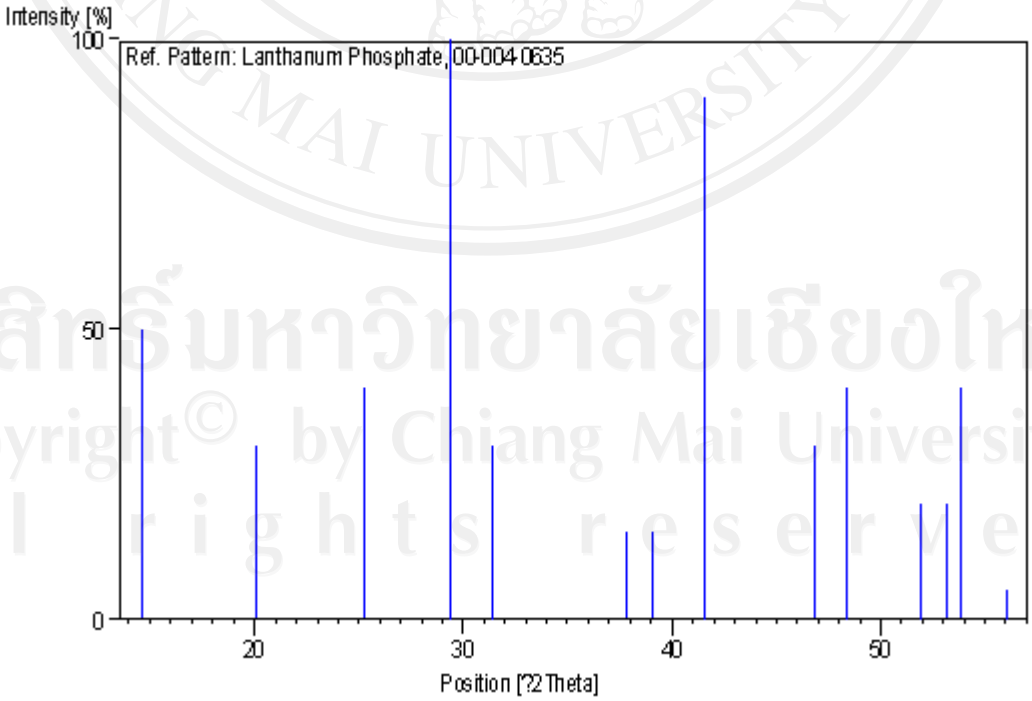
General comments: Cell parameters generated by least squares refinement.

Unit cell: Reference reports: a=7.081, c=6.468.

References

Primary reference: Mooney., *Acta Crystallogr.*, **3**, 338, (1950)

Stick Pattern



Peak list

No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	1	0	0	6.08000	14.557	50.0
2	1	0	1	4.41000	20.119	30.0
3	1	1	0	3.52000	25.281	40.0
4	2	0	0	3.04000	29.356	100.0
5	1	0	2	2.85000	31.362	30.0
6	1	1	2	2.38000	37.768	15.0
7	2	1	0	2.30500	39.046	15.0
8	2	1	1	2.17000	41.584	90.0
9	3	0	1	1.94000	46.789	30.0
10	2	1	2	1.88000	48.376	40.0
11	2	2	0	1.76000	51.911	20.0
12	3	0	2	1.72000	53.212	20.0
13	2	2	1	1.70000	53.888	40.0
14	3	1	1	1.64000	56.029	5.0

APPENDIX B

Joint committee on powder diffraction standard of $CePO_4$

1. $CePO_4$ monoclinic structure

Name and formula

Reference code:	01-077-0429
Mineral name:	Monazite
ICSD name:	Cerium Phosphate
Empirical formula:	CeO_4P
Chemical formula:	$Ce (PO_4)$

Crystallographic parameters

Crystal system:	Monoclinic
Space group:	P21/n
Space group number:	14
a (?):	6.7700
b (?):	6.9900
c (?):	6.4500
Alpha (?):	90.0000
Beta (?):	103.6000
Gamma (?):	90.0000
Calculated density (g/cm^3):	5.26
Measured density (g/cm^3):	5.20
Volume of cell (10^6 pm^3):	296.67

Z:	4.00
RIR:	3.39

Subfiles and Quality

Subfiles:	Inorganic
	Mineral
	Modelled additional pattern
Quality:	Calculated (C)

Comments

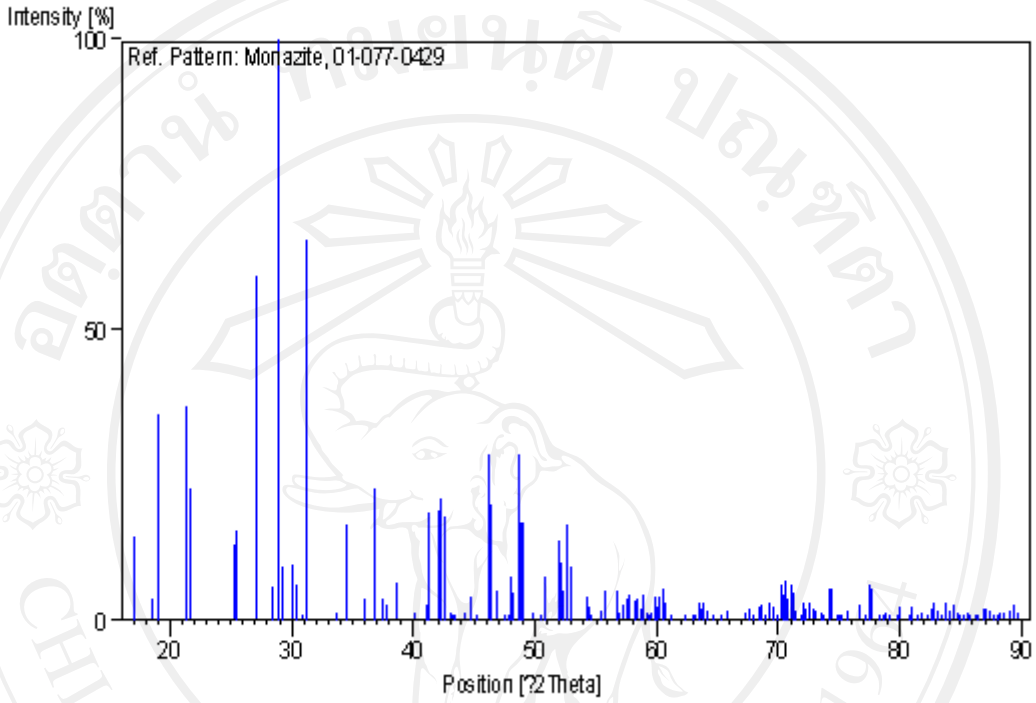
Sample source:	Specimen from Ishikawayama, Fukushima, Japan.
ICSD collection code:	039135
Test from ICSD:	At least one TF missing.
	No R value given.
	Calc. density unusual but tolerable.

References

Primary reference: *Calculated from ICSD using POWD-12++*, (1997)

Structure: Ueda, T., *Mem. Coll. Sci., Univ. Kyoto, Ser. B*, **20**, 227, (1953)

Stick Pattern



Peak list

No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	-1	0	1	5.18902	17.074	14.5
2	1	1	0	4.79122	18.504	3.7
3	0	1	1	4.66707	19.000	35.3
4	-1	1	1	4.16646	21.308	37.0
5	1	0	1	4.08454	21.741	22.8
6	1	1	1	3.52659	25.233	12.8
7	0	2	0	3.49500	25.465	15.4
8	2	0	0	3.29009	27.080	59.4
9	0	0	2	3.13457	28.452	5.9

10	1	2	0	3.08663	28.903	100.0
11	0	2	1	3.05267	29.232	9.4
12	2	1	0	2.97682	29.994	9.6
13	-2	1	1	2.94256	30.351	6.2
14	-1	2	1	2.89879	30.821	0.9
15	0	1	2	2.86016	31.248	65.8
16	1	2	1	2.65554	33.725	1.2
17	-2	0	2	2.59451	34.543	16.4
18	2	1	1	2.49151	36.018	3.9
19	1	1	2	2.43875	36.825	12.1
20	-2	1	2	2.43236	36.926	22.6
21	2	2	0	2.39561	37.513	3.7
22	-2	2	1	2.37764	37.807	2.8
23	0	2	2	2.33166	38.582	6.3
24	-3	0	1	2.24111	40.207	1.2
25	1	3	0	2.19637	41.062	2.5
26	0	3	1	2.18403	41.305	18.5
27	-1	0	3	2.14231	42.147	19.0
28	-3	1	1	2.13410	42.317	21.2
29	2	2	1	2.12003	42.611	17.9
30	3	1	0	2.09278	43.194	0.7
31	1	2	2	2.08724	43.314	0.9
32	-2	2	2	2.08323	43.402	0.5
33	-1	1	3	2.04827	44.181	0.4

34	2	0	2	2.04227	44.318	0.3
35	1	3	1	2.02386	44.743	4.0
36	0	1	3	2.00216	45.255	0.9
37	2	1	2	1.96032	46.276	28.4
38	-3	1	2	1.95479	46.414	20.1
39	3	0	1	1.93346	46.957	5.1
40	-2	1	3	1.91235	47.507	0.3
41	2	3	0	1.90147	47.796	0.8
42	-2	3	1	1.89245	48.038	7.5
43	-3	2	1	1.88656	48.197	4.9
44	-1	3	2	1.86901	48.679	28.7
45	3	1	1	1.86349	48.833	16.9
46	3	2	0	1.85784	48.991	16.8
47	-1	2	3	1.82649	49.889	0.9
48	1	1	3	1.80552	50.509	1.1
49	0	2	3	1.79356	50.869	7.3
50	-3	2	2	1.75927	51.934	13.6
51	2	3	1	1.75459	52.083	10.1
52	0	4	0	1.74750	52.310	5.2
53	1	3	2	1.73586	52.688	16.7
54	-3	0	3	1.72815	52.941	9.3
55	1	4	0	1.68896	54.269	4.1
56	0	4	1	1.68333	54.465	2.3
57	-3	1	3	1.67903	54.616	0.3

58	-1	4	1	1.65611	55.437	1.7
59	4	0	0	1.64436	55.867	5.0
60	-4	0	2	1.62200	56.707	5.1
61	-3	3	1	1.61521	56.967	1.3
62	1	4	1	1.60664	57.299	2.8
63	4	1	0	1.60130	57.508	3.6
64	3	3	0	1.59707	57.674	4.2
65	3	1	2	1.58411	58.191	3.6
66	-4	1	2	1.58002	58.356	3.7
67	-1	3	3	1.57703	58.477	1.2
68	-1	1	4	1.57123	58.714	2.0
69	0	0	4	1.56729	58.877	4.4
70	-2	0	4	1.56502	58.970	2.6
71	2	1	3	1.56127	59.126	1.3
72	0	3	3	1.55569	59.359	0.4
73	-3	2	3	1.55022	59.590	0.3
74	2	4	0	1.54331	59.884	4.0
75	-2	4	1	1.53848	60.091	2.3
76	-3	3	2	1.53315	60.322	3.9
77	0	1	4	1.52932	60.489	4.4
78	-2	1	4	1.52721	60.581	5.6
79	-4	2	1	1.52280	60.775	2.9
80	-2	3	3	1.51242	61.237	0.1
81	4	2	0	1.48841	62.334	0.4

2. $CePO_4$ hexagonal structure

Name and formula

Reference code:	01-075-1880
ICSD name:	Cerium Phosphate
Empirical formula:	CeO_4P
Chemical formula:	$CePO_4$

Crystallographic parameters

Crystal system:	Hexagonal
Space group:	P6222
Space group number:	180
a (?):	7.0550
b (?):	7.0550
c (?):	6.4390
Alpha (?):	90.0000
Beta (?):	90.0000
Gamma (?):	120.0000
Calculated density (g/cm^3):	4.22
Volume of cell (10^6 pm^3):	277.55
Z:	3.00
RIR:	5.23

Subfiles and Quality

Subfiles:	Inorganic
	Modelled additional pattern
Quality:	Calculated (C)

Comments

ICSD collection code:	031563
Test from ICSD:	No R value given.
	At least one TF missing.

References

Primary reference:	*Calculated from ICSD using POWD-12++*, (1997)
Structure:	Mooney, R.C.L., *Acta Crystallogr.*, **3**, 337, (1950)

Peak list

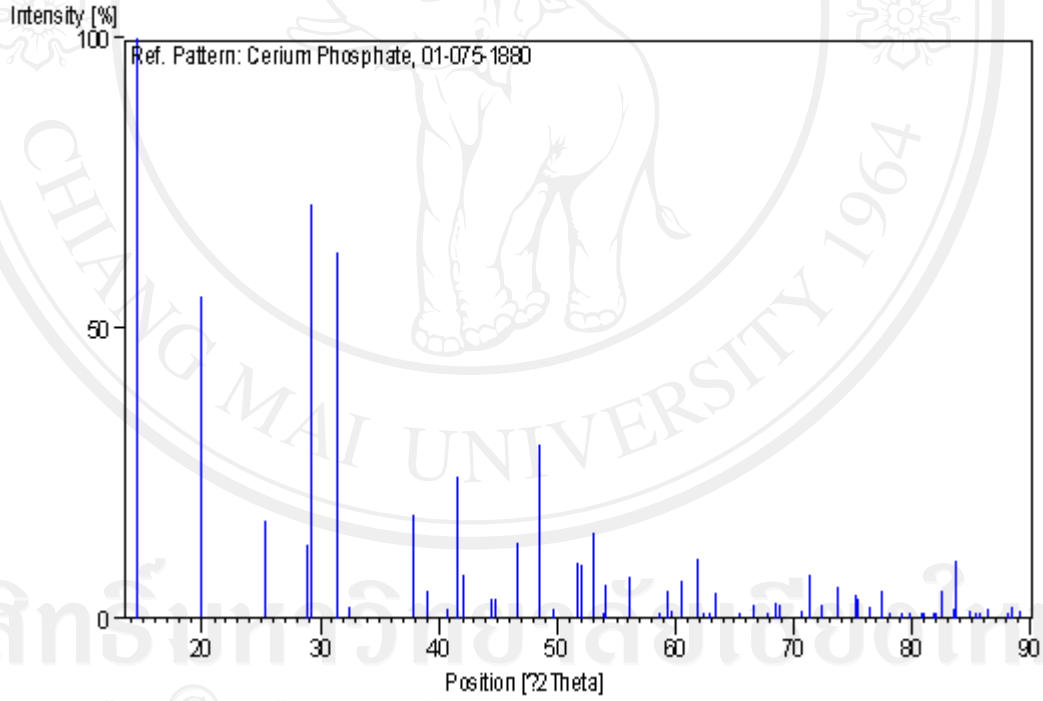
No.	h	k	l	d [A]	2Theta[deg]	I [%]
1	1	0	0	6.10981	14.486	100.0
2	1	0	1	4.43210	20.018	55.4
3	1	1	0	3.52750	25.227	16.8
4	1	1	1	3.09368	28.836	12.6
5	2	0	0	3.05490	29.210	71.4
6	1	0	2	2.84826	31.382	63.1
7	2	0	1	2.76003	32.412	1.9
8	1	1	2	2.37798	37.802	18.0

9	2	1	0	2.30929	38.971	4.5
10	2	0	2	2.21605	40.681	1.7
11	2	1	1	2.17372	41.510	24.5
12	0	0	3	2.14633	42.064	7.7
13	3	0	0	2.03660	44.448	3.6
14	1	0	3	2.02502	44.716	3.3
15	3	0	1	1.94179	46.744	12.8
16	2	1	2	1.87648	48.473	30.0
17	1	1	3	1.83359	49.682	1.6
18	2	2	0	1.76375	51.792	9.7
19	2	0	3	1.75621	52.031	9.3
20	3	0	2	1.72114	53.173	14.6
21	2	2	1	1.70109	53.850	0.6
22	3	1	0	1.69456	54.075	5.9
23	3	1	1	1.63876	56.075	7.0
24	2	1	3	1.57214	58.677	0.7
25	1	0	4	1.55663	59.320	4.7
26	2	2	2	1.54684	59.733	0.2
27	4	0	0	1.52745	60.570	6.3
28	3	1	2	1.49953	61.820	10.3
29	4	0	1	1.48621	62.436	0.2
30	3	0	3	1.47737	62.852	0.9
31	1	1	4	1.46447	63.470	4.2
32	2	0	4	1.42413	65.489	0.1

33	3	2	0	1.40169	66.672	2.2
34	4	0	2	1.38001	67.861	0.1
35	3	2	1	1.36961	68.447	2.9
36	2	2	3	1.36268	68.844	2.6
37	4	1	0	1.33327	70.585	0.6
38	3	1	3	1.33000	70.785	1.3
39	2	1	4	1.32057	71.367	7.6
40	4	1	1	1.30558	72.315	2.2
41	3	2	2	1.28517	73.650	5.3
42	3	0	4	1.26289	75.172	3.9
43	1	0	5	1.26011	75.367	3.4
44	4	0	3	1.24448	76.482	2.1
45	4	1	2	1.23182	77.414	4.8
46	5	0	0	1.22196	78.156	0.5
47	1	1	5	1.20971	79.102	0.5
48	5	0	1	1.20053	79.827	0.6
49	2	2	4	1.18899	80.761	0.1
50	2	0	5	1.18667	80.952	0.1
51	3	3	0	1.17583	81.856	0.4
52	3	2	3	1.17359	82.046	0.6
53	3	1	4	1.16709	82.602	4.5
54	3	3	1	1.15671	83.509	1.5
55	4	2	0	1.15465	83.692	9.8
56	5	0	2	1.14244	84.793	1.3

57	4	2	1	1.13652	85.339	0.1
58	4	1	3	1.13255	85.710	0.1
59	2	1	5	1.12473	86.452	1.4
60	4	0	4	1.10802	88.087	0.1
61	3	3	2	1.10448	88.443	1.9
62	5	1	0	1.09735	89.170	1.2

Stick Pattern



CURRICULUM VITAE

Name Miss Nuengruethai Ekthammathat

Date of Birth November 15, 1986

Education Background

2005 - 2008 Bachelor of Science (Chemistry), second Class honors, Chiang Mai University

Scholarship

2009 - 2010 The Center of Excellence for Innovation in Chemistry (PERCH-CIC)

Experiences

2009 Undergraduate Teaching Assistant, Chiang Mai University, General Chemistry Laboratory

International publication from MS.D. Thesis

1. **Nuengruethai Ekthammathat**, Titipun Thongtem, Anukorn Phuruangrat and Somchai Thongtem, "Microwave-assisted synthesis and characterisation of uniform $LaPO_4$ nanorods", *Journal of Experimental Nanoscience*, (2011) Accepted.

Other International publication

1. Anukorn Phuruangrat, **Nuengruethai Ekthammathat**, Titipun Thongtem, and Somchai Thongtem, "Microwave-assisted synthesis and optical property of $CdMoO_4$ nanoparticles", *Journal of Physics and Chemistry of Solids*, 72 (2011) 176-180**.**

Conference presentation

1. **Nuengruethai Ekthammathat**, Titipun Thongtem, Anukorn Phuruangrat and Somchai Thongtem, "Luminescence of uniform $LaPO_4$ nanorods synthesized by a facial microwave method", The 28th Annual Conference of the Microscopy Society of Thailand, 5-7 January 2011, Mea Fah Luang University, Chiang Rai, Thailand**.**
   (Proceeding of the 28th MST Annual Conference)
2. **Nuengruethai Ekthammathat**, Titipun Thongtem, Anukorn Phuruangrat and Somchai Thongtem, "Microwave-assisted synthesis of $CePO_4$ nanorods phosphor with violet emission", The 12th International Symposium on Eco-materials Processing and Design, 8-11 January 2011, Chiang Mai University, Chiang Mai, Thailand**.**
   (Excellence Award of Poster Presentation)

INTERNATIONAL PUBLICATION

Journal of Experimental Nanoscience
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Taylor & Francis

Microwave-assisted synthesis and characterisation of uniform $LaPO_4$ nanorods

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Lanthanum phosphate ($LaPO_4$) phosphor materials with one-dimensional (1-D) nanorods and nanoparticles were successfully synthesised from $LaCl_3 \cdot 7H_2O$ and $Na_3PO_4 \cdot 12H_2O$ with pH adjusted at the range 1–6 using 37% HNO_3 by the microwave radiation at 180 W for 60 min. X-ray diffraction patterns indicated that the as-synthesised products were hexagonal $LaPO_4$ nanostructures. Scanning electron microscopy and transmission electron microscopy analyses showed that these products were nanoparticles, short and long nanorods, controlled by pH of the precursor solutions. Asymmetric stretching and bending vibrations of the PO_4^{3-} groups were detected using Fourier transform-infrared spectroscopy. A possible formation mechanism of $LaPO_4$ phosphor materials with 1-D nanorods and nanoparticles was also proposed according to the experimental results.

Keywords: $LaPO_4$; 1-D nanorods; nanoparticles; microwave-assisted synthesis

1. Introduction

In recent years, scientists and researchers have focused on doing research on the properties of one-dimensional (1-D) nanomaterials, including nanorods, nanowires, nanobelts and nanotubes for use as phosphor materials, which have more efficient novel luminescent properties, due to their quantum size effect [1–3]. In comparison with 0-D structures, the space anisotropy of a 1-D structure provided a better model system to study the dependence of electronic transport, optical and mechanical properties on size confinement and dimensionality [4,5]. Their properties were controlled by the morphology and size which can be controlled by several parameters including temperature, holding reaction time, template/surfactant added, pH value, synthesised methods, starting materials and many others [6,7].

Lanthanide orthophosphate ($LnPO_4$) nanomaterials including lanthanum phosphate ($LaPO_4$), $CePO_4$, $EuPO_4$ and $GdPO_4$ belong to the group of phosphor materials, which are very interesting for researchers due to their unique chemical and physical properties [1,2]. They have a variety of potentially beneficial properties such as very high thermal stability (~2300°C), low solubility ($K_{sp} = 10^{-25} - 10^{-27}$) in water, high refractive index ($n \approx 1.5$) and high concentration of lasing ions (~1.8×10^{21} ionscm^{-3}). Because of these unique properties, $LnPO_4$ is capable of being used in various applications, such as luminescent or laser materials, magnets, ceramics, catalysts, proton conductors, moisture sensors, heat-resistant materials, hosts for radioactive nuclear waste, green ($^5D_4 - ^7F_5$ of Tb^{3+} at 543 nm) phosphor (for Tb^{3+} co-activated by bulk $LaPO_4$) in fluorescent lamps, scintillators for X-ray and gamma-ray detection in medical science, biochemical probes and medical diagnostics [8]. Among them, $LaPO_4$, with a unique 4f shell of La^{3+} ions, has been widely used in optoelectronic devices high-performance luminescent devices, magnets, catalysts, time-resolved fluorescence labels for biological detection and other functional materials [1,8,9].

There are a number of reports on the synthesis of $LaPO_4$ nanomaterials by various methods, such as liquid phase reaction [10], sonochemical method [11], precipitation [12], solvothermal/hydrothermal method [9, 13] and solid–liquid reaction [14], but they are not advantageous due to the high-temperature consumption and long-reaction time. In the past decades, microwave radiation has been considered as a new candidate, and is capable of being applied for the synthesis of nanomaterials such as YVO_4 [15], ZnO [16], CeO_2 [17] and $PbWO_4$ [18]. The microwave method consumes low power and has short-reaction time, and is able to produce highly purified products with controlled morphologies, compared to others [19].

In this research, hexagonal $LaPO_4$ nanorods have been synthesised by microwave radiation method at the precursor pH range of 1–6. The products were characterised by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) and Fourier transform-infrared spectrometry (FT-IR).

2. Experiment

To synthesise $LaPO_4$ nanostructures, chemical-grade reagents purchased from Merck Company, $LaCl_3 \cdot 7H_2O$, $Na_3PO_4 \cdot 12H_2O$ and 37% HNO_3, were used without further purification.

Each 0.003 mole of $LaCl_3 \cdot 7H_2O$ and $Na_3PO_4 \cdot 12H_2O$ was dissolved in 80 mL deionised (DI) water with vigorous stirring for 15 min, and followed by the addition of 37% HNO_3 to the precursor solutions for adjusting the pH to be in the range 1–6. Each of these solutions was continuously heated by a microwave oven (Electrolux, EMS 2820, 2.45 GHz) at 180 W and 60 min at a time, and naturally cooled down to room temperature. Finally, white precipitates were produced, filtered, washed with DI water and 95% ethanol several times and dried at 80°C for 12 h. The final products were collected for further characterisation.

Phase, morphologies and vibration modes of the products were characterised by XRD (Philips X'Pert MPD) using Cu-Kα line ($\lambda = 1.54056$ Å), and scanned at 2θ angle ranging from 10° to 60° with a scanning rate of 0.04° per step. The XRD data were analysed by X'Pert HighScore Plus program in combination with The Joint Committee on Powder

Diffraction Standards (JCPDS). Field emission scanning electron microscopy (FE-SEM, JEOL JSM-6335F) and TEM (JEOL JEM-2010) were operated at 15.0 and 200 kV with LaB_6 electron gun. FT-IR spectroscopy analysis (Perkin Elmer RX spectrophotometer) of the products with 40 times concentration diluted by KBr was performed over 400–4000 cm^{-1} range with 4 cm^{-1} resolution.

3. Results and discussion

Phase and crystalline structure of $LaPO_4$ synthesized by microwave radiation method with pH adjusted to be in the range 1–6 by the addition of 37% HNO_3 were investigated by XRD, as shown in Figure 1. These patterns were identified as purified hexagonal $LaPO_4$ phase, compared to the JCPDS No. 04-0635 ($a = b = 7.0420$ Å and $c = 6.4490$ Å) [20]. When the precursor pH values were lowered from 6 to 1, the XRD peaks became sharper, proving that the product crystal has improved. Therefore, it can be concluded that hexagonal $LaPO_4$ crystalline was influenced by the precursor pH. Lattice parameters of hexagonal $LaPO_4$ nanostructures were calculated from the equation of plane spacing for the hexagonal structure in combination with the Bragg's law for diffraction, as shown below.

$$\frac{1}{d^2} = \frac{4}{3a^2}(h^2 + hk + k^2) + \frac{1}{c^2}l^2 \tag{1}$$

$$\lambda = 2d\sin\theta \tag{2}$$

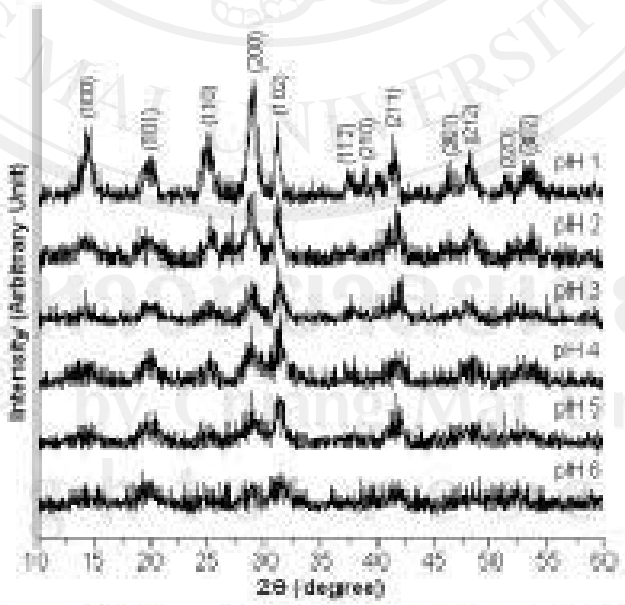

Figure 1. XRD patterns of $LaPO_4$ synthesized in the solutions with different pH values by microwave radiation at 180 W for 60 min.

where a and c are the lattice parameters, h, k and l the Miller indices, d the plane spacing and θ the Bragg's angle [21]. The calculated lattice parameters of the hexagonal $LaPO_4$ nanomaterials (pH = 1) are $a = b = 7.0859$ Å and $c = 6.5016$ Å, very close to those of the JCPDS standard values [20].

Consider the lanthanide phosphate compounds such as $CePO_4$, $LaPO_4$ and $GdPO_4$. Their different modes were originated from the vibration in the tetrahedral PO_4^{3-} units. The selection rule for the isolated PO_4^{3-} tetrahedrons shows different vibration modes as $A_1(R) + E(R) + 2F_2(IR + R)$. The $\upsilon_1(A_1)$ and $\upsilon_2(E)$ corresponding to the symmetric stretching and bending modes are Raman active. The $\upsilon_3(F_2)$ and $\upsilon_4(F_2)$ are the asymmetric stretching and bending modes, specified as Raman and IR doubly active. The symmetry of PO_4^{3-} ions in the lanthanide phosphate compounds decreases from T_d to C_1. Thus, the non-IR modes become IR active [22]. FT-IR spectra of $LaPO_4$ synthesised by the microwave-assisted synthesis at the pH levels of 1, 3 and 5 were recorded in the range 400–4000 cm^{-1} as shown in Figure 2. The broad bands at 3440 cm^{-1} and 1644 cm^{-1} were, respectively, assigned to be the O–H stretching and bending modes of absorbed water containing in the products. The other peaks at 1046, 614 and 540 cm^{-1} were seen in the vibrations in PO_4^{3-} of $LaPO_4$. The mode at 1046 cm^{-1} was ascribed as the asymmetric stretching vibration of P–O bonds. Those centred at 614 and 540 cm^{-1} were the P–O bending vibrations of the PO_4^{3-} groups [7,9,22]. There were no other functional groups, detected in these products.

The morphologies of $LaPO_4$ nanostructures were characterised by SEM and TEM. From SEM analysis (Figure 3), it is observed that the morphologies of the as-synthesised $LaPO_4$ products have different shapes (nanoparticles and nanorods), depending on the precursor pH of the system. At the pH range of 4–6, there were agglomerates of nanoparticles with size less than 50 nm. When the precursor pH was lowered to 3, the $LaPO_4$ nanoparticles arranged themselves and diffused to form short nanorods.

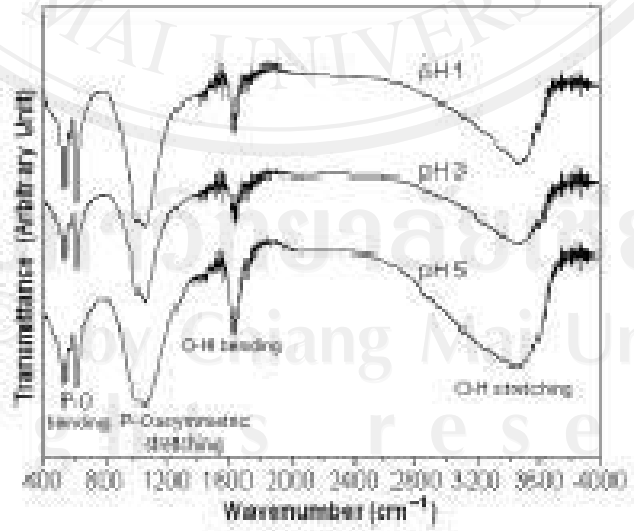

Figure 2. FTIR spectra of $LaPO_4$ synthesised in the solutions with pH values of 1, 3 and 5 by microwave radiation at 800 W for 60 min.

Finally, the short nanorods (~200 nm) and long nanorods (~600 nm) were respectively produced at the pH values of 2 and 1, under the microwave radiation. These results confirmed that pH is the key factor to control the $LaPO_4$ morphologies, by changing from nanoparticles to 1-D nanorods, due to the increase in the H^+ concentration of the solutions. The effect of pH on different morphologies of $LaPO_4$ nanostructures is illustrated in Figure 4.

The uniform shape of $LaPO_4$ nanorods was characterised by TEM, as shown in Figure 5. It indicates the nanomorphology of $LaPO_4$ at the pH values 1 and 2, showing that both these products are nanorods with different lengths and diameters. The $LaPO_4$ nanorods are 100–200 nm long with 10–12 nm diameter for pH value 2, and 600–1000 nm long, with 20–40 nm diameter for pH value 1 – in accordance with those of the SEM analysis. The lattice plane and growth direction of $LaPO_4$ nanorods were analysed by high-resolution transmission electron microscope (HRTEM), as shown in Figure 5(c) and (d). It shows lattice fringes, of which a number of planes are parallel to the $LaPO_4$ nanorods. These planes are 4.42 Å apart, corresponding to the (1 0 1) plane of hexagonal $LaPO_4$ structure (JCPDS No. 04-0635) [20]. This analysis demonstrated that the hexagonal $LaPO_4$ nanorods grew along the [−1 0 1] direction.

A possible formation mechanism of $LaPO_4$ phosphor nanomaterials is simply explained as follows:

$$PO_4^{3-} + nH^+ \rightarrow H_nPO_4^{(3-n)-} \quad (3)$$

$$La^{3+} + H_nPO_4^{(3-n)-} \rightarrow LaPO_4\,(\text{white precipitate}) + nH^+ \quad (4)$$

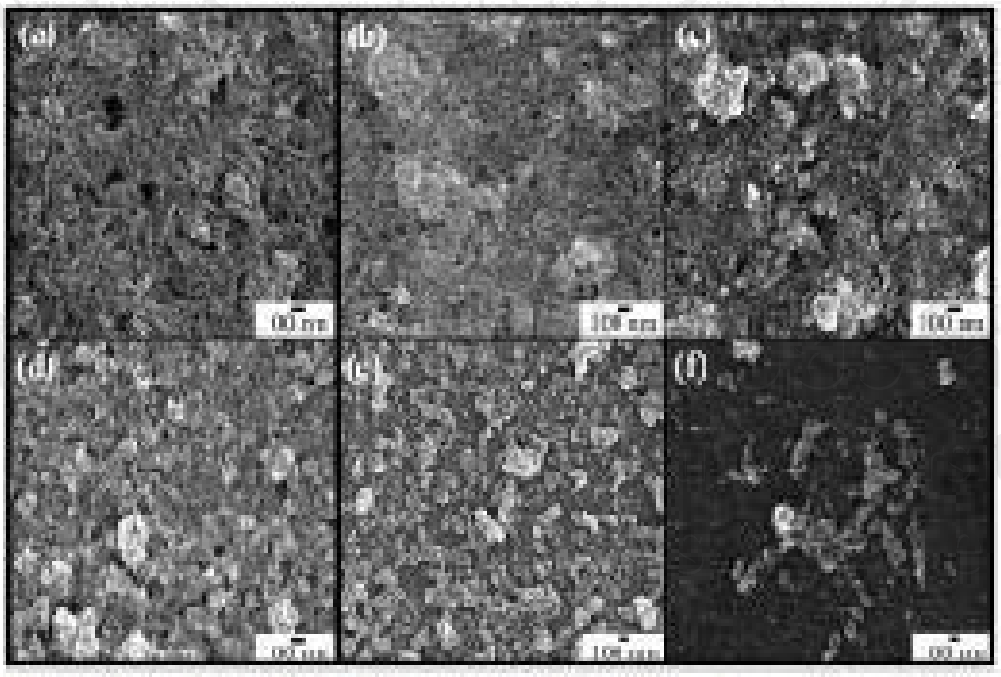

Figure 3. SEM images of $LaPO_4$ synthesised in the solutions with pH values of (a)–(f) 1, 2, 3, 4, 5 and 6 by microwave radiation at 180 W for 60 min, respectively.

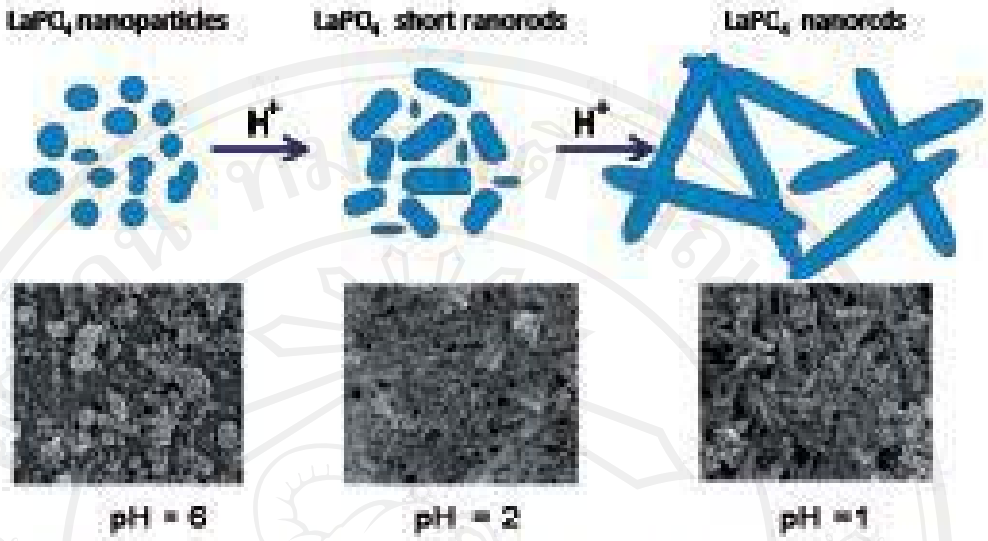

Figure 4. Schematic diagram for the formation of $LaPO_4$ products.

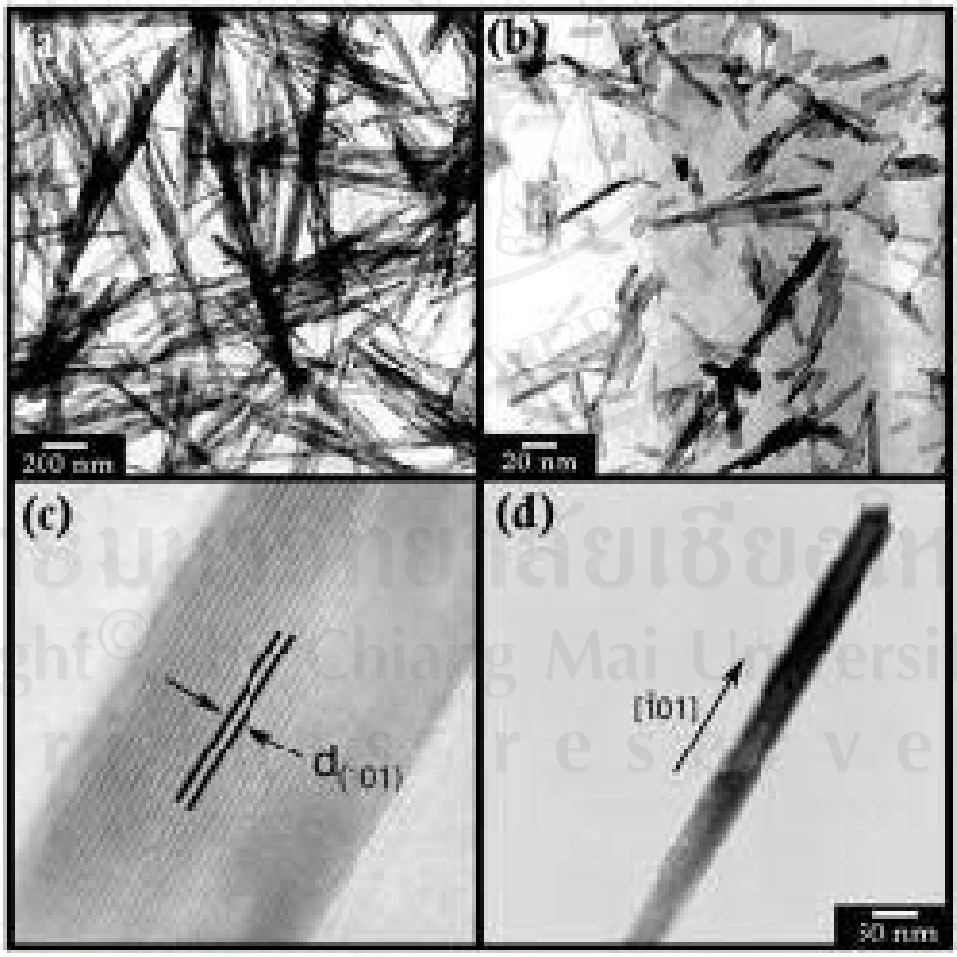

Figure 5. TEM and HRTEM images of $LaPO_4$ synthesized in the solutions with the pH of (a), (c) and (d) 1, and (b) 2, by microwave radiation at 180 W for 60 min.

In this research, $LaCl_3 \cdot 7H_2O$ and $Na_3PO_4 \cdot 12H_2O$ dissolved in DI water with the addition of HNO_3 (conc.). Thus, H^+ ions reacted with PO_4^{3-} ions to form $H_nPO_4^{(3-n)-}$, which subsequently reacted with La^{3+} to produce $LaPO_4$ white precipitates by the microwave radiation at 180 W for 60 min. The products have different morphologies for different pH values of the solutions. These implied that H^+ ions induced the system to be at the highest chemical potential and led the product to grow in 1-D $LaPO_4$ nanorods at pH value of 1 – in accordance with the report of Zhang and Guan [23]. Compared to the facile solution-precipitation process reported by Wang et al. [24], $LaPO_4$ products with different morphologies were synthesised. The effect of the precursor pH on the different morphologies was explained above. In this research, microwave radiation as a heating source has more advantages than the conventional heating method, due to the rapid volumetric heating, resulting in higher reaction rate and selectivity, reduction in reaction time often by orders of magnitude, and increasing yields of the products [18]. It is a candidate for the synthesis of nanomaterials.

4. Conclusions

Hexagonal $LaPO_4$ nanorods were successfully synthesised from $LaCl_3 \cdot 7H_2O$ and $Na_3PO_4 \cdot 12H_2O$ in DI water at pH level 1 by the 180-W and 60-min microwave radiation. This method is simple and inexpensive, with short-reaction time and can be applied for the synthesis of other nanomaterials.

Acknowledgements

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