CHAPTER 3

RESULTS AND DISCUSSION

3.1 POWDERS CHARACTERIZATION

BNT and BLNT powders synthesized from a hydrothermal process were characterized by the following methods.

3.1.1 CRYSTALLINE STRUCTURE DETERMINATION

3.1.1.1 BNT Powders from Hydrothermal Process

3.1.1.1.1 Effect of Holding Period at 200 ^{o}C

The effect of synthesis time on perovskite BNT formation is shown in Figure 3.1. The synthesis temperature was fixed at 200 °C and 10 M NaOH was used as a mineralizer. The holding period was varied from 5 to 20 hours. For all holding periods, the XRD patterns showed rhombohedral of BNT. It can be clearly seen from the XRD patterns that the critical holding period for BNT powders by a hydrothermal process at this condition was 5 hours. Rhombohedral BNT was formed with a holding period of 5 hours and longer.

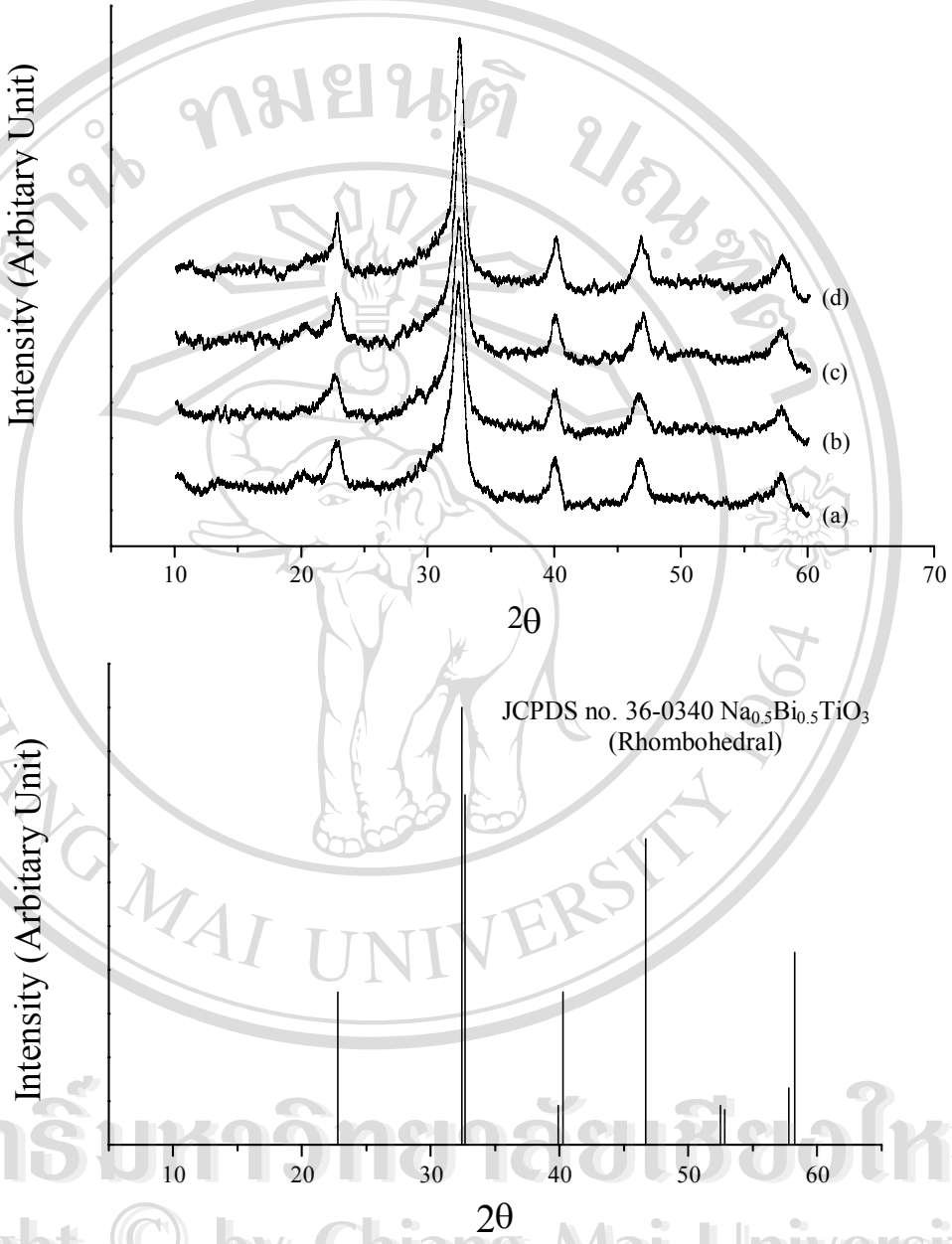


Figure 3.1 XRD patterns of BNT powders synthesized by hydrothermal process at 200 °C using 10 M NaOH as a mineralizer with different holding periods of (a) 5 h, (b) 10 h, (c) 15 h, and (d) 20 h.

3.1.1.1.2 Effect of Holding Period at 150 ^{o}C

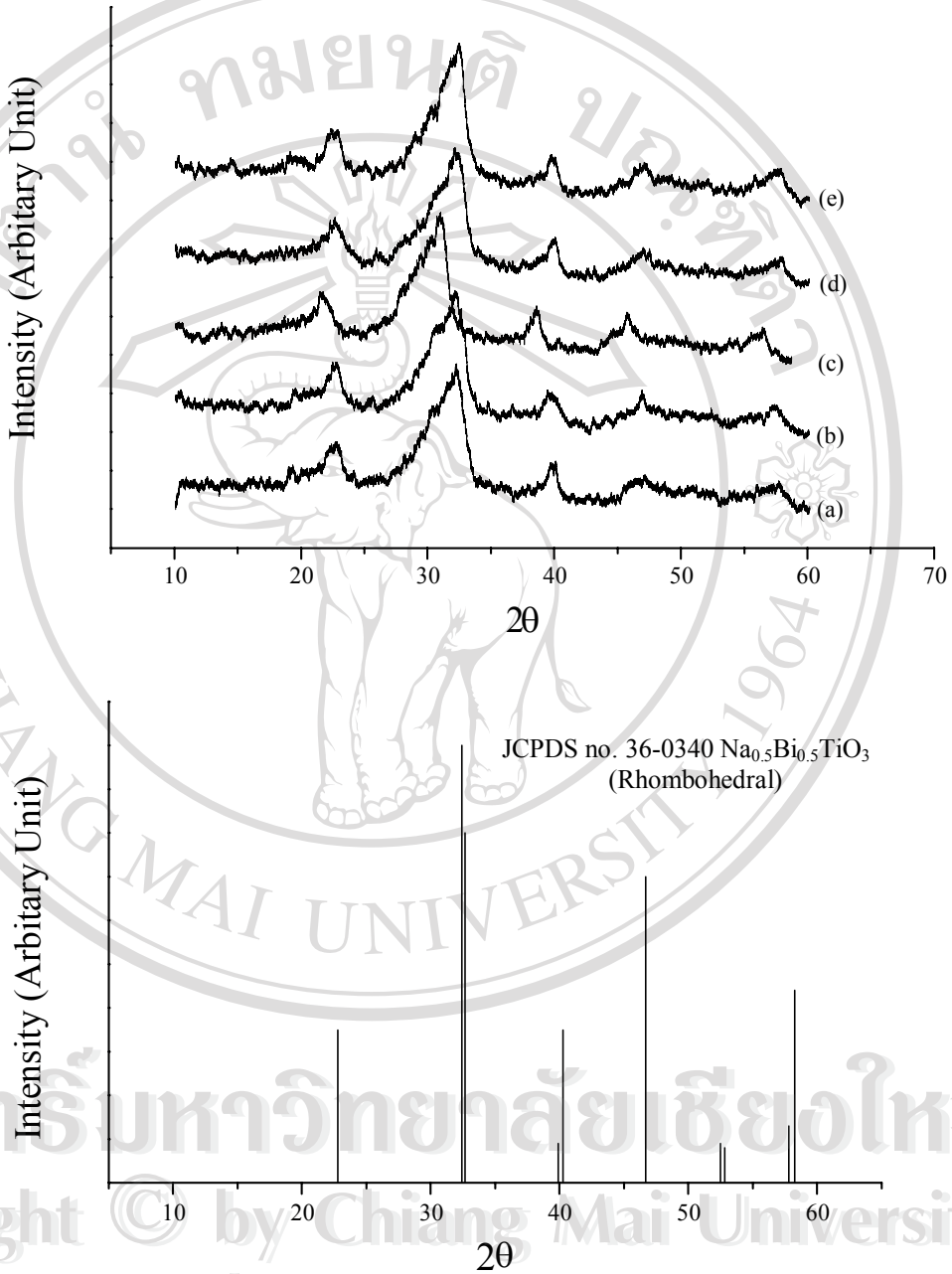
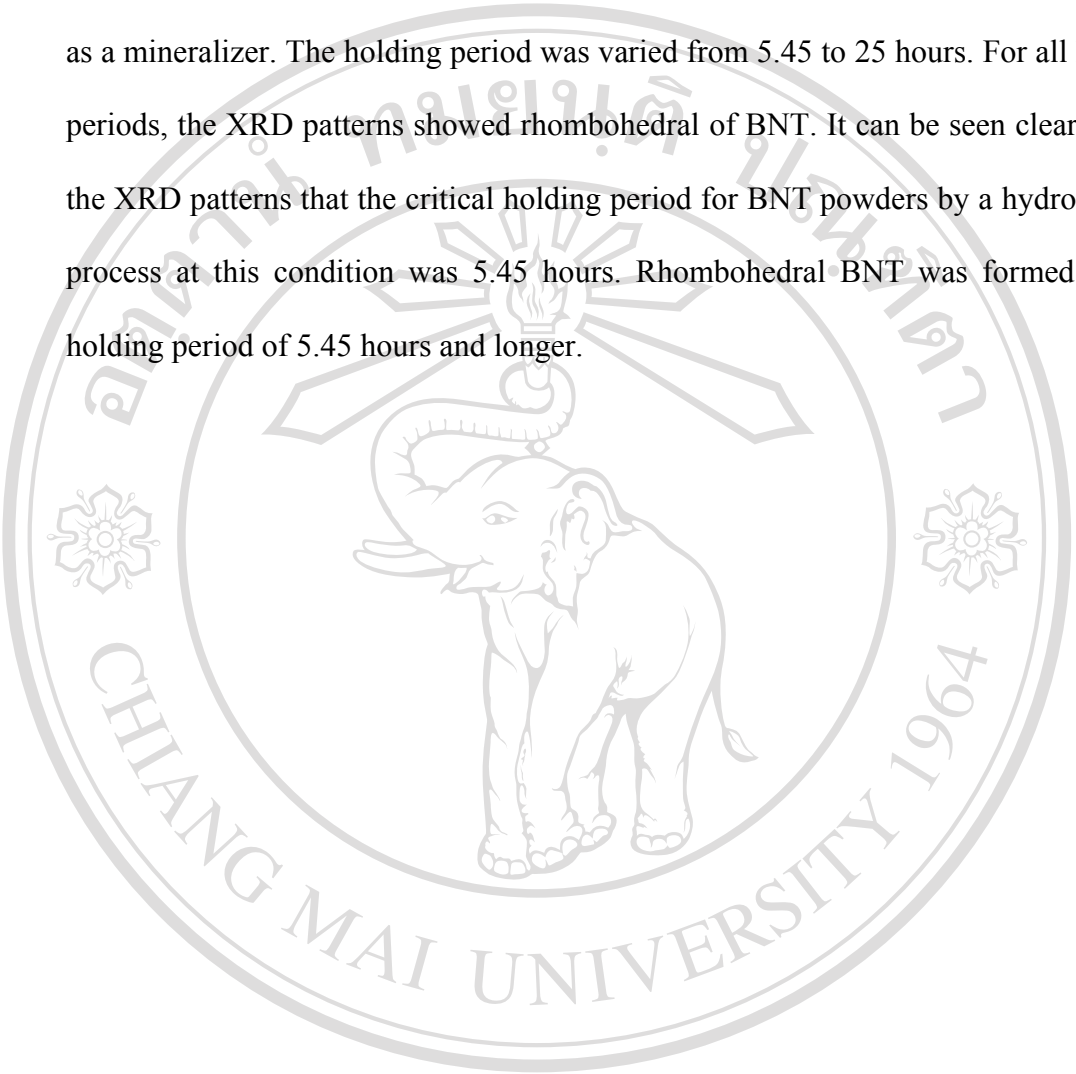


Figure 3.2 XRD patterns of BNT powders synthesized by hydrothermal process at 150 °C using 12 M NaOH as a mineralizer with different holding periods of (a) 5.45 h, (b) 10 h, (c) 15 h, (d) 20 h and (e) 25 h.

The effect of synthesis time on perovskite BNT formation is shown in Figure 3.2. The synthesis temperature was fixed at 150 °C and 12 M NaOH was used as a mineralizer. The holding period was varied from 5.45 to 25 hours. For all holding periods, the XRD patterns showed rhombohedral of BNT. It can be seen clearly from the XRD patterns that the critical holding period for BNT powders by a hydrothermal process at this condition was 5.45 hours. Rhombohedral BNT was formed with a holding period of 5.45 hours and longer.



3.1.1.1.3 Effect of Holding Period at 175 ^{o}C

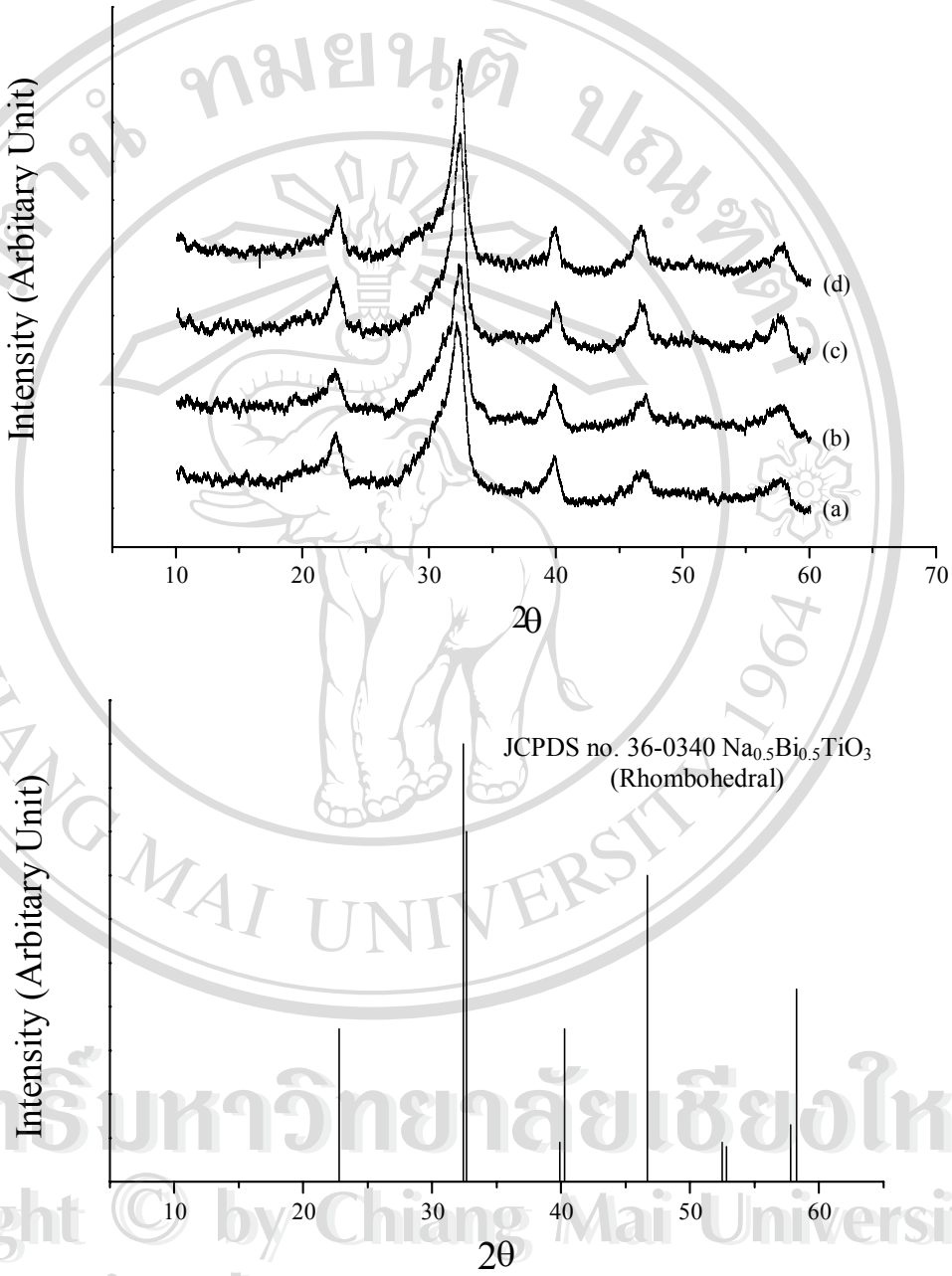
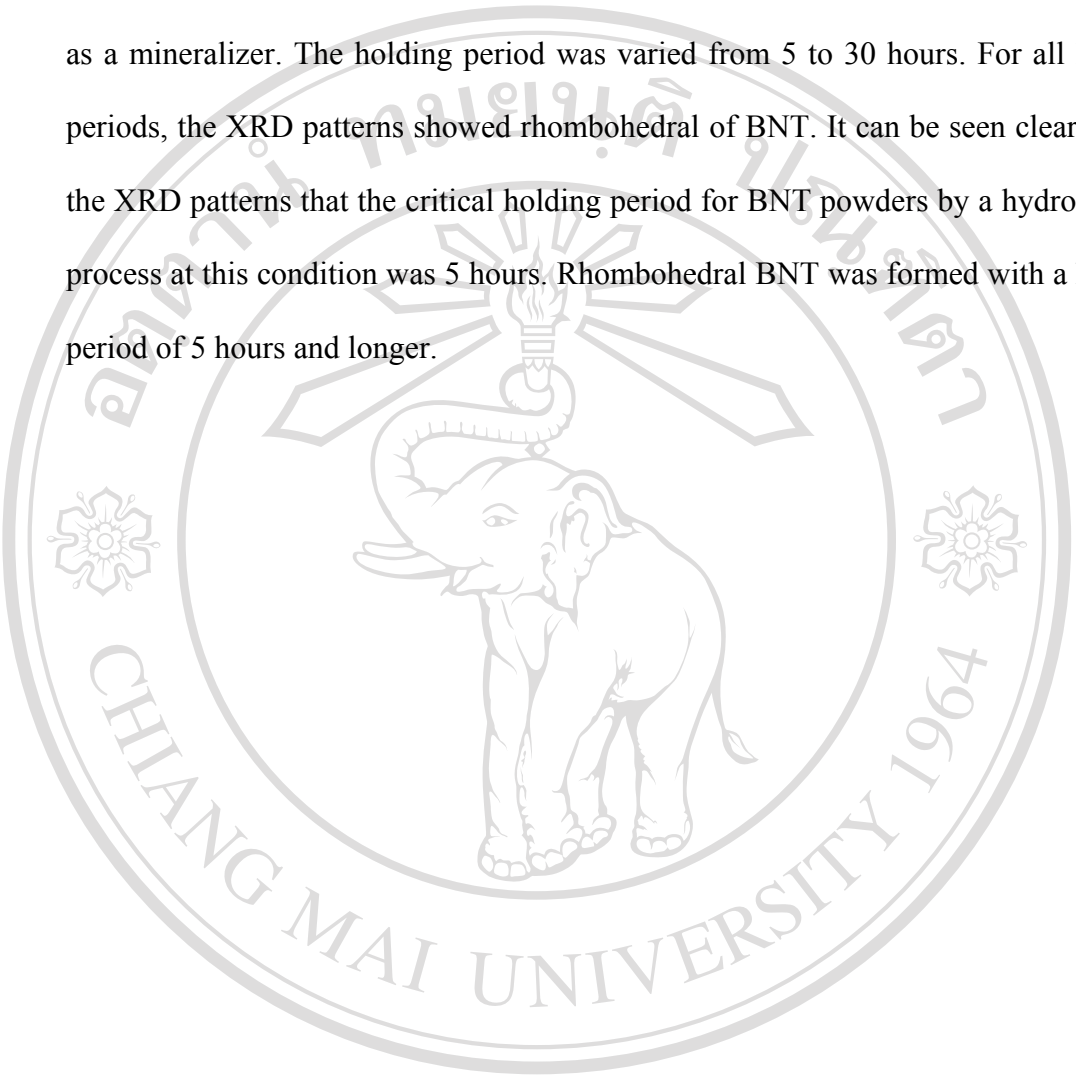


Figure 3.3 XRD patterns of BNT powders synthesized by hydrothermal process at 175 °C using 12 M NaOH as a mineralizer with different holding periods of (a) 5 h, (b) 10 h, (c) 20 h, and (d) 30 h.

The effect of synthesis time on perovskite BNT formation is shown in Figure 3.3. The synthesis temperature was fixed at 175 °C and 12 M NaOH was used as a mineralizer. The holding period was varied from 5 to 30 hours. For all holding periods, the XRD patterns showed rhombohedral of BNT. It can be seen clearly from the XRD patterns that the critical holding period for BNT powders by a hydrothermal process at this condition was 5 hours. Rhombohedral BNT was formed with a holding period of 5 hours and longer.



3.1.1.1.4 Effect of Holding Period at 200 ^{o}C

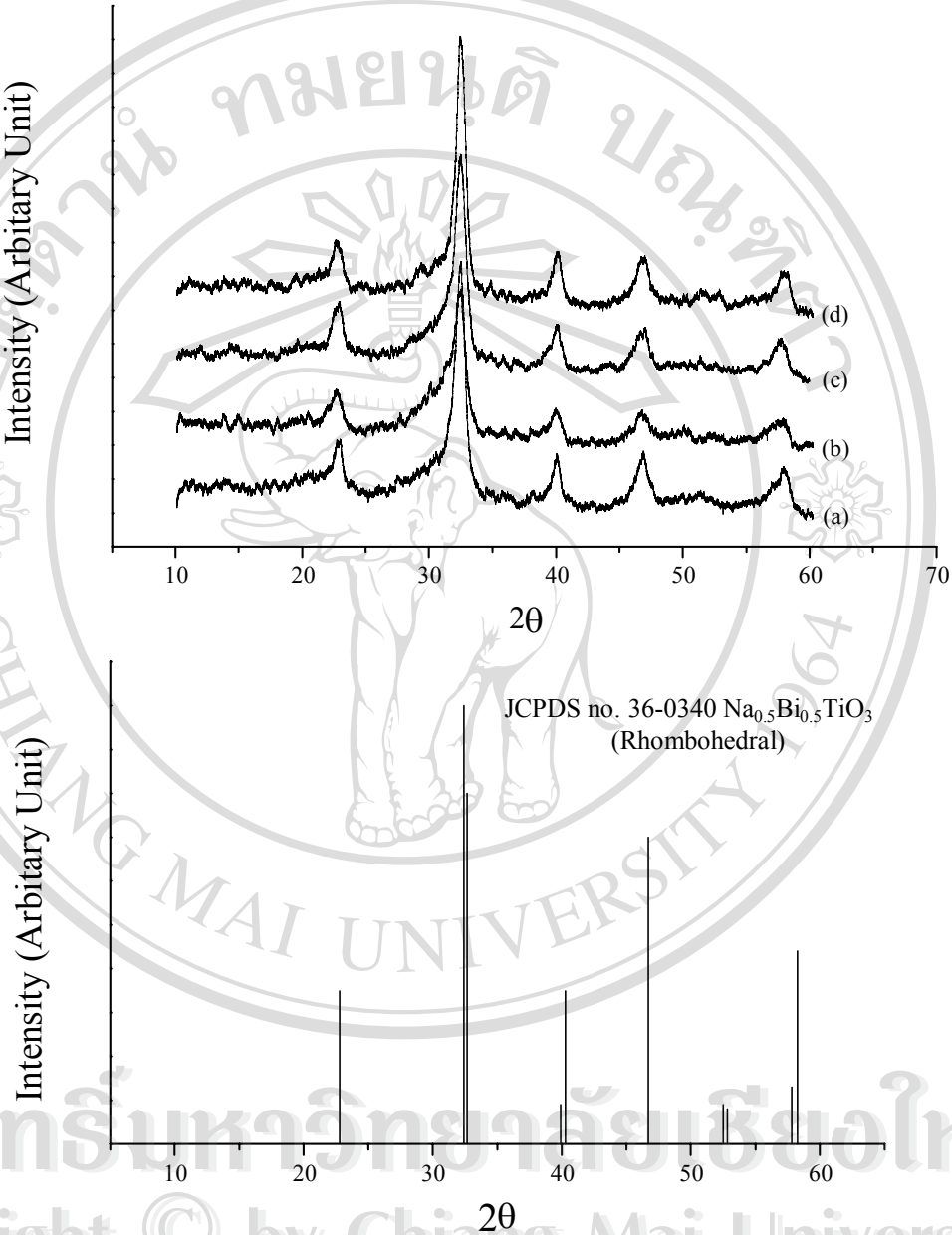
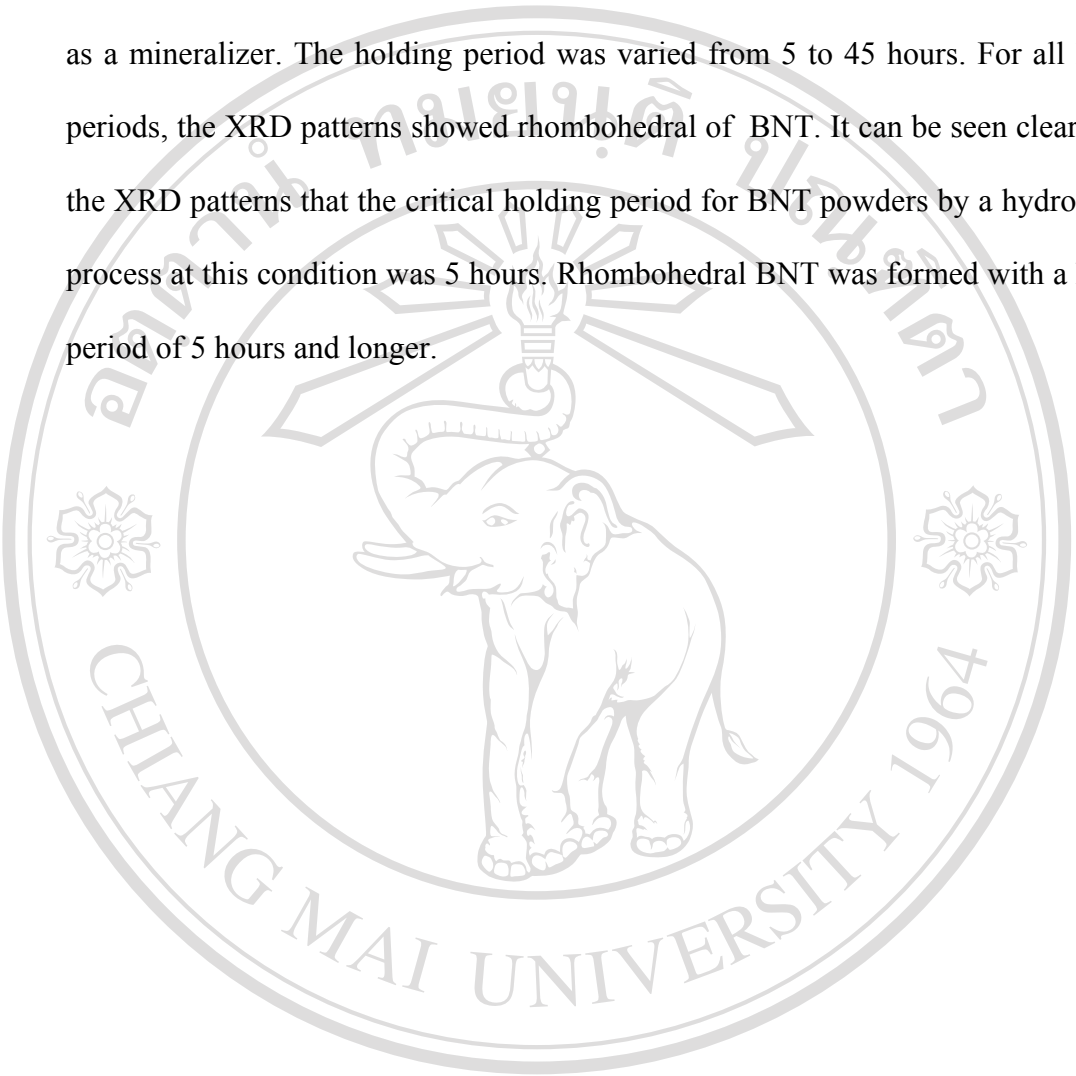


Figure 3.4 XRD patterns of BNT powders synthesized by hydrothermal process at 200 °C using 12 M NaOH as a mineralizer with different holding periods of (a) 5 h, (b) 15 h, (c) 20 h, and (d) 45 h.

The effect of synthesis time on perovskite BNT formation is shown in Figure 3.4. The synthesis temperature was fixed at 200 °C and 12 M NaOH was used as a mineralizer. The holding period was varied from 5 to 45 hours. For all holding periods, the XRD patterns showed rhombohedral of BNT. It can be seen clearly from the XRD patterns that the critical holding period for BNT powders by a hydrothermal process at this condition was 5 hours. Rhombohedral BNT was formed with a holding period of 5 hours and longer.



3.1.1.1.5 Effect of Synthesis Temperature and Mineralizer Concentration with Holding Period of 5 Hours

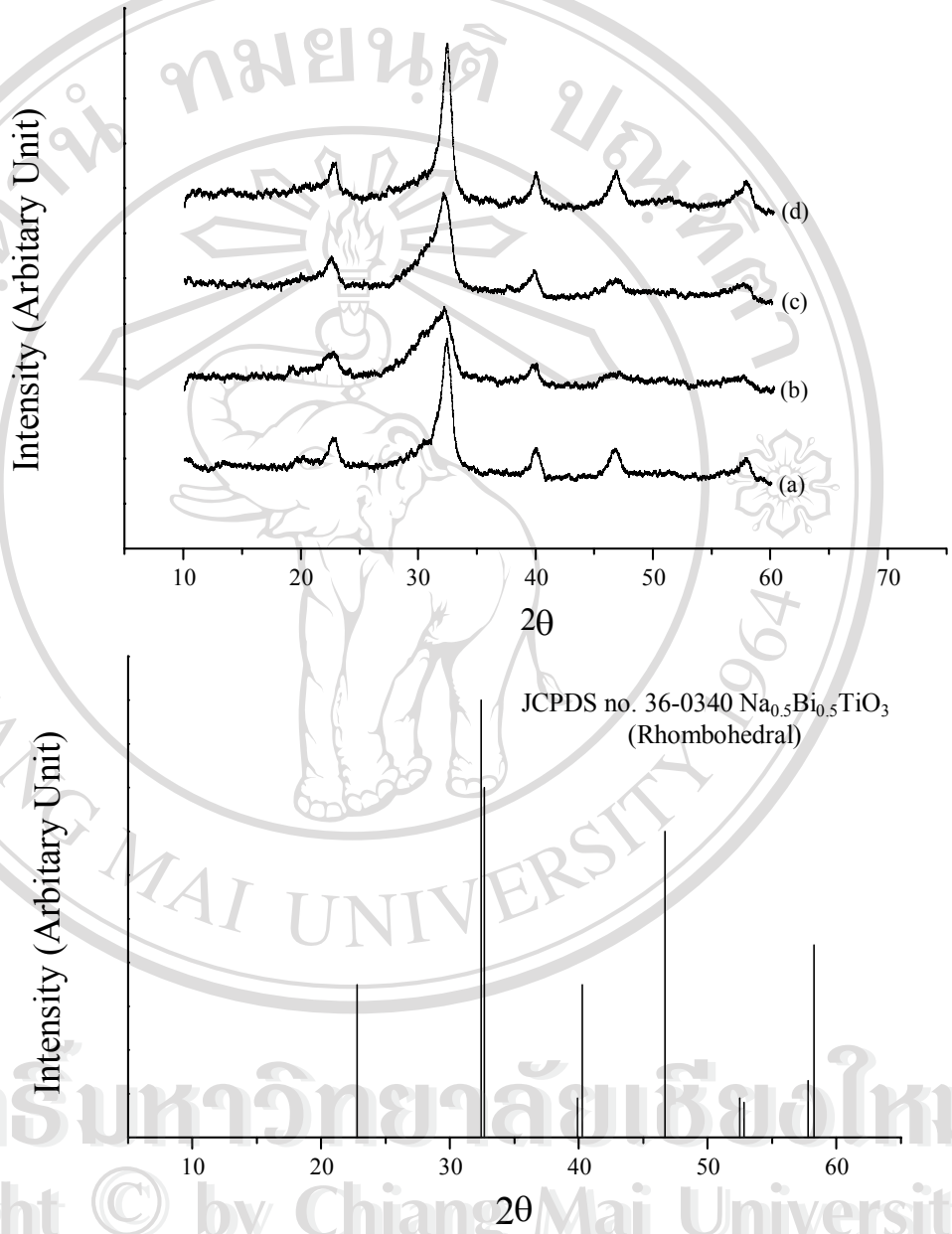


Figure 3.5 XRD patterns of BNT powders synthesized by hydrothermal process with holding period of 5 h using 10 –12 M NaOH as a mineralizer at different synthesis temperatures as (a) 10 M, 200 °C, (b) 12 M, 150°C, (c) 12 M, 175 °C and (d) 12 M, 200 °C.

The effect of synthesis temperature and concentration of mineralizer on hydrothermally synthesized BNT powders are illustrated in Figure 3.5. The holding period was fixed at 5 hours using10-12 M NaOH as a mineralizer and at different synthesis temperatures. In all cases, the product was rhombohedral BNT. From this study, it can be seen clearly that the synthesis temperature and concentration of mineralizer were of the major effects in the perovskite hydrothermally synthesized BNT powders.

3.1.1.1.6 Effect of Synthesis Temperature and Mineralizer Concentration with Holding Period of 15 Hours

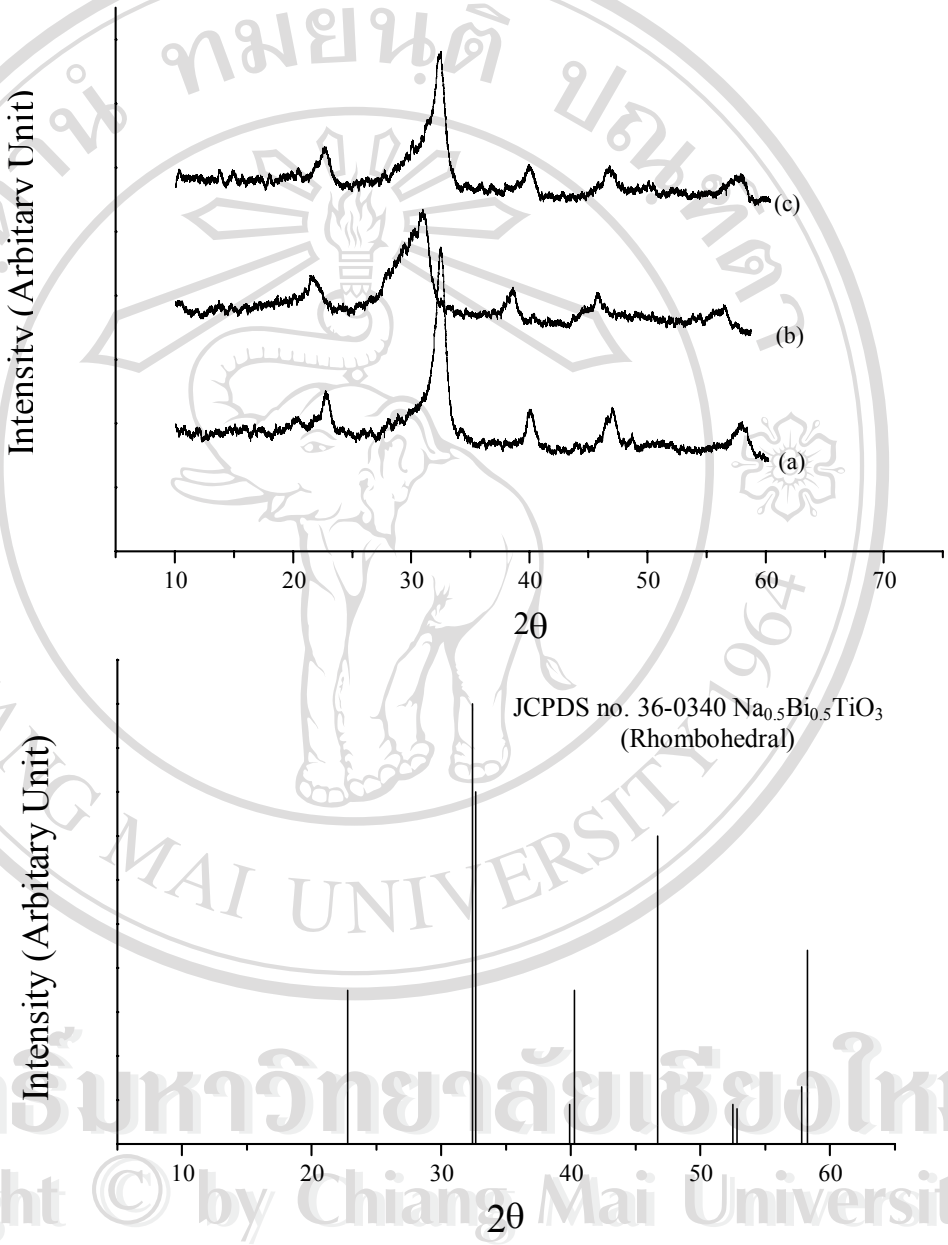


Figure 3.6 XRD patterns of BNT powders synthesized by hydrothermal process with holding period of 15 h using 10 –12 M NaOH as a mineralizer at different synthesis temperatures as (a) 10 M, 200 °C, (b) 12 M, 150 °C and (c) 12 M, 200 °C.

The effect of synthesis temperature and concentration of mineralizer on hydrothermally synthesized BNT powders are illustrated in Figure 3.6. The holding period was fixed at 15 hours using 10-12 M NaOH as a mineralizer and at different synthesis temperatures. In all cases, the product was rhombohedral BNT. From this study, it can be seen clearly that the synthesis temperature and concentration of mineralizer were the major effects in the perovskite hydrothermally synthesized BNT powders.

3.1.1.1.7 Effect of Synthesis Temperature and Mineralizer Concentration with Holding Period of 20 Hours

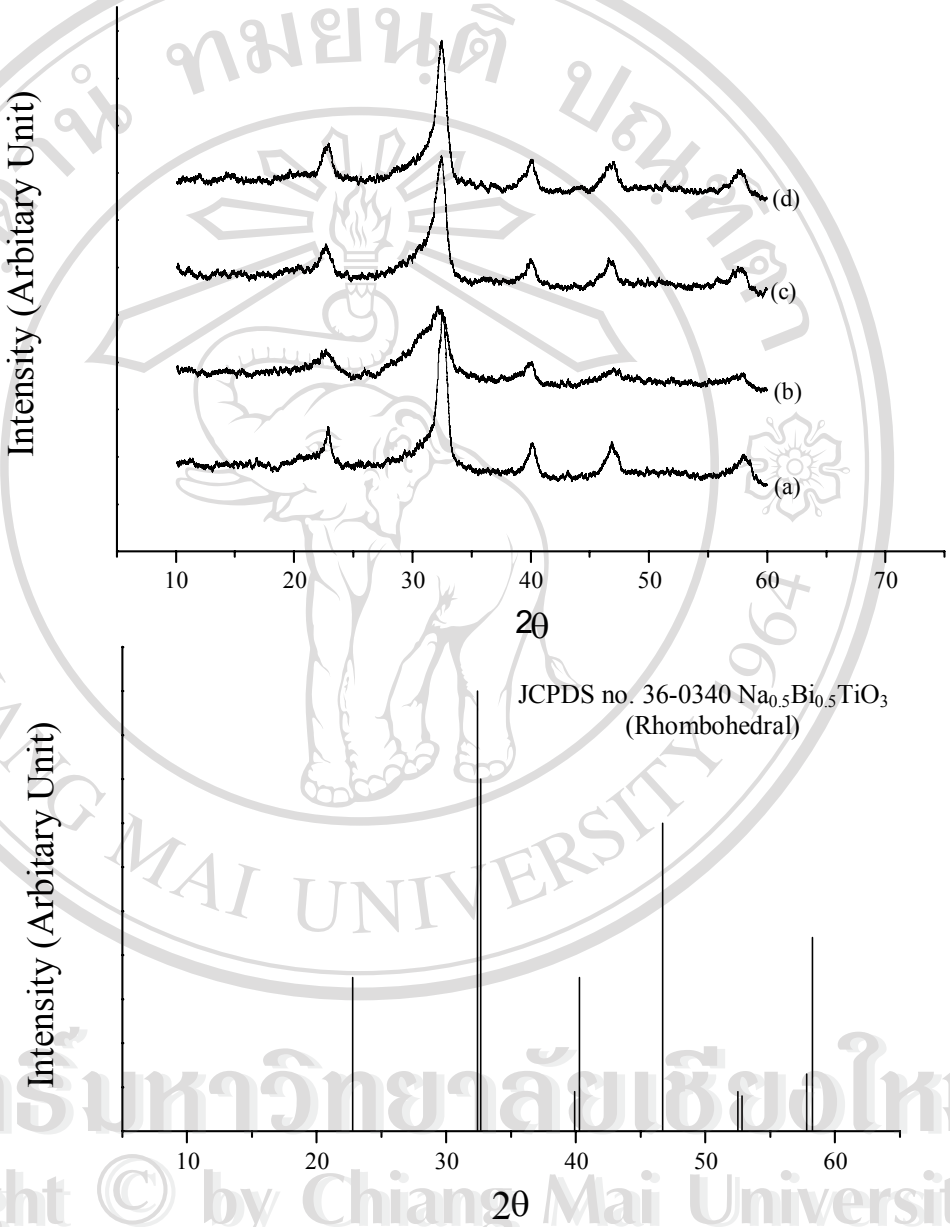
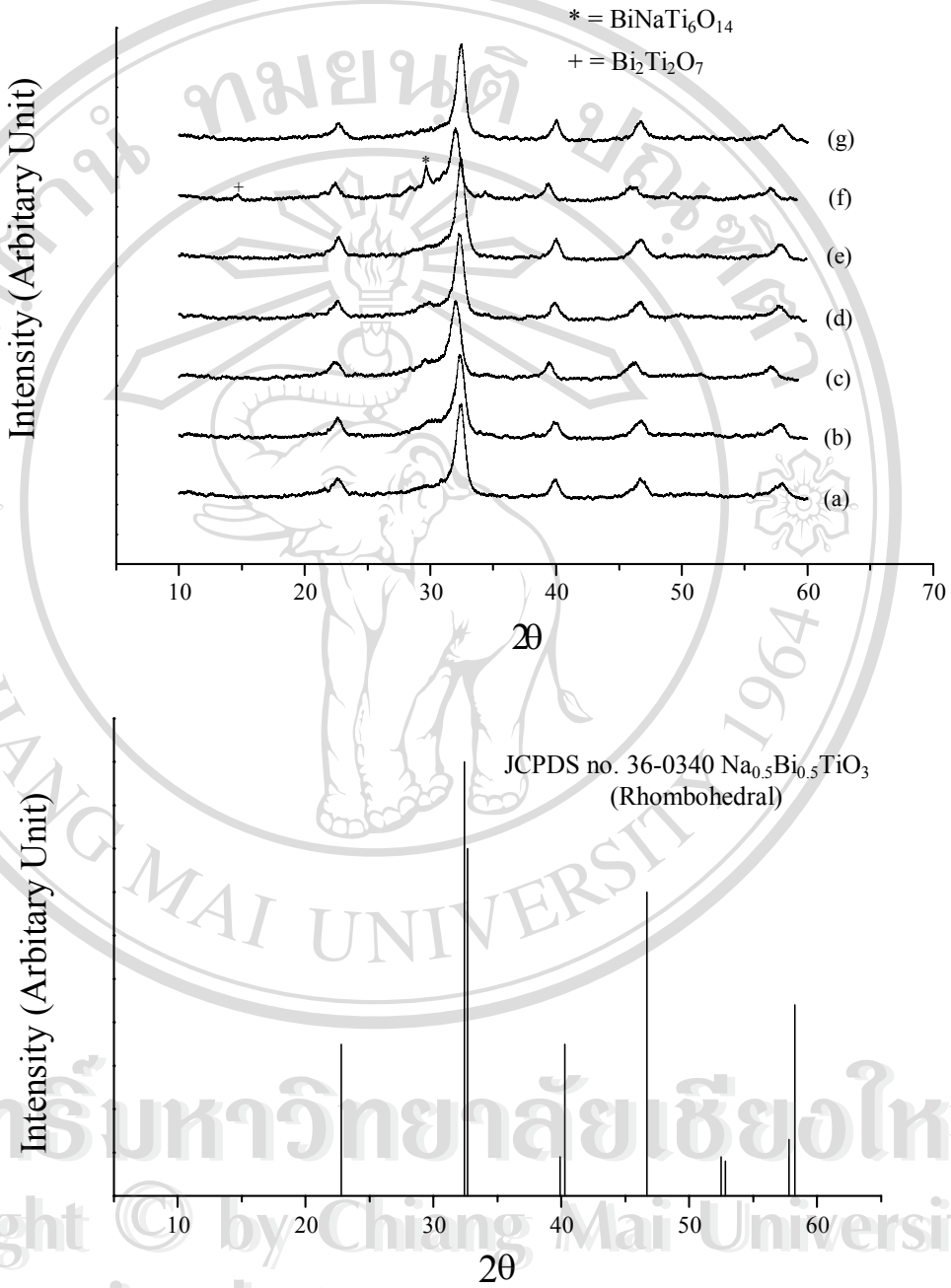


Figure 3.7 XRD patterns of BNT powders synthesized by hydrothermal process with holding period of 20 h using 10 –12 M NaOH as a mineralizer at different synthesis temperatures as (a) 10 M, 200 °C, (b) 12 M, 150 °C, (c) 12 M, 175 °C and (d) 12 M, 200 °C

The effect of synthesis temperature and concentration of mineralizer on hydrothermally synthesized BNT powders are illustrated in Figure 3.7. The holding period was fixed at 20 hours using 10-12 M NaOH as a mineralizer and different synthesis temperatures. In all cases the product was rhombohedral BNT. From this study, it can be seen clearly that the synthesis temperature and concentration of mineralizer were of the major effects in the perovskite hydrothermally synthesized BNT powders.

3.1.1.2 BLNT Powders from Hydrothermal Process



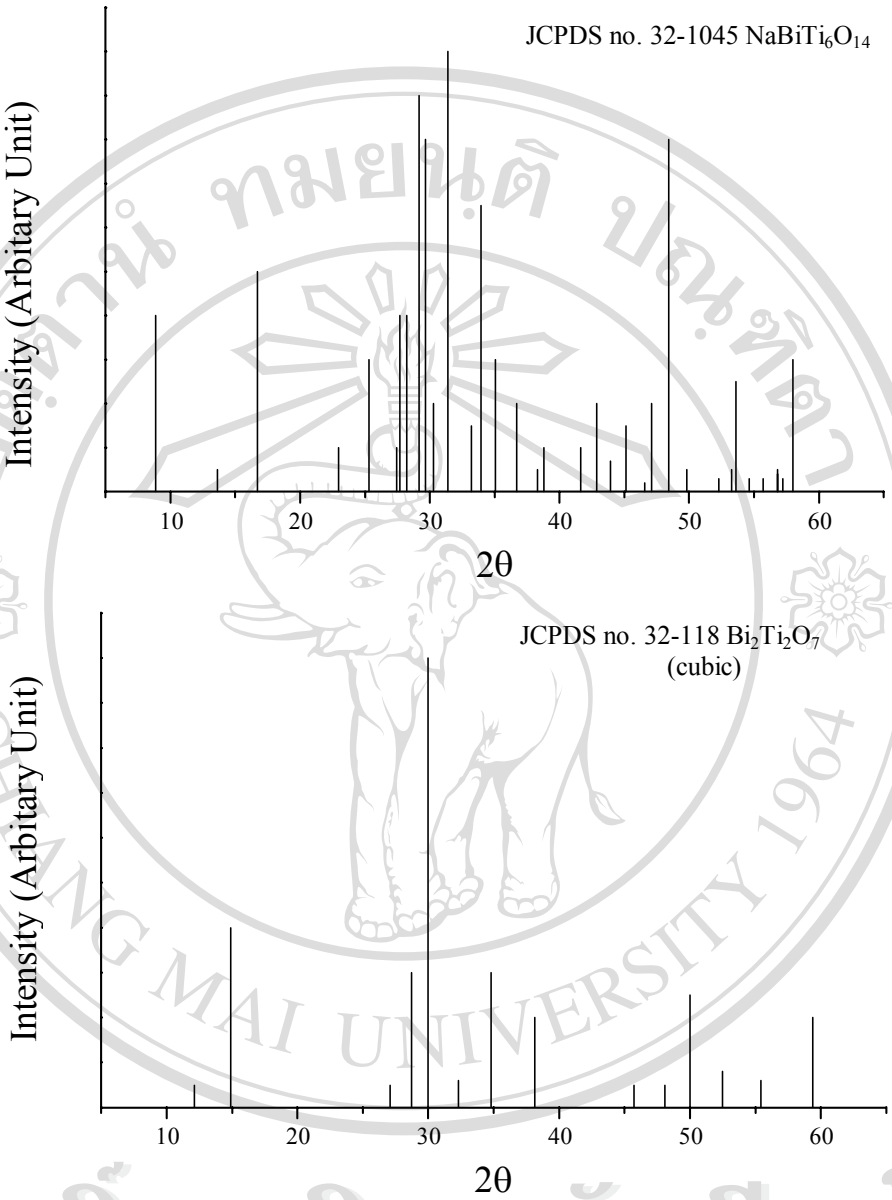


Figure 3.8 XRD patterns of BLNT powders synthesized by hydrothermal process at 200 °C with holding period of 20 h using 12 M NaOH as a mineralizer and different mole %La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La.

The XRD pattern of BLNT powders at different mole %La, synthesized by a hydrothermal process at 200 °C with a holding period of 20 hours using 12 M NaOH at pH 13 as a mineralizer, is given in Figure 3.8. From XRD patterns, the rhombohedral phase of BLNT occurred and matched with the JCPDS file number 36-0340.

From XRD investigations it can be concluded that BNT and BLNT can be hydrothermally synthesized at temperatures ranging from 150-200 ^{o}C at pH 13 with holding periods of 5-60 hours.

Table 3.1 The condition for hydrothermally synthesized of BNT and BLNT powders.

Sample	Temperature (^{o}C)	Concentration (M)	Holding periods (h)	Structure
BNT	200	10	5	Rhombohedral
	200	10	10	Rhombohedral
	200	10	15	Rhombohedral
	200	10	20	Rhombohedral
BNT	150	12	5.45	Rhombohedral
	150	12	10	Rhombohedral
	150	12	15	Rhombohedral
	150	12	20	Rhombohedral
	150	12	25	Rhombohedral

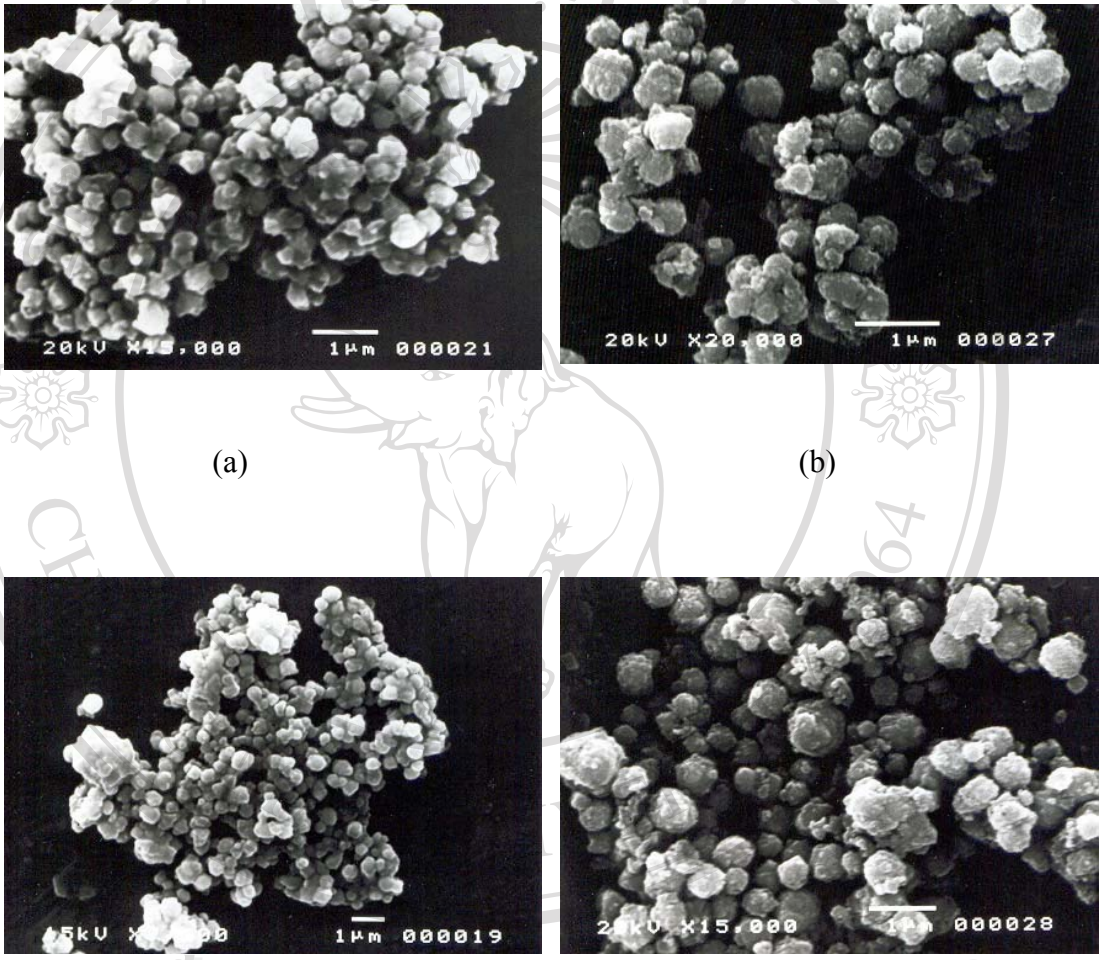
Table 3.1 (continue)

Sample	Temperature (^{o}C)	Concentration (M)	Holding periods (h)	Structure
BNT	175	12	5	Rhombohedral
	175	12	10	Rhombohedral
	175	12	20	Rhombohedral
	175	12	30	Rhombohedral
BNT	200	12	5	Rhombohedral
	200	12	15	Rhombohedral
	200	12	20	Rhombohedral
	200	12	45	Rhombohedral
BLNT (1%)	200	12	20	Rhombohedral
BLNT (2%)	200	12	20	Rhombohedral
BLNT (3%)	200	12	20	Rhombohedral
BLNT (4%)	200	12	20	Rhombohedral
BLNT (5%)	200	12	20	Rhombohedral with * $BiNaTi_6O_{14}$ and + $Bi_2Ti_2O_7$
BLNT (6%)	200	12	20	Rhombohedral

3.1.2 MICROSTRUCTURE ANALYSIS

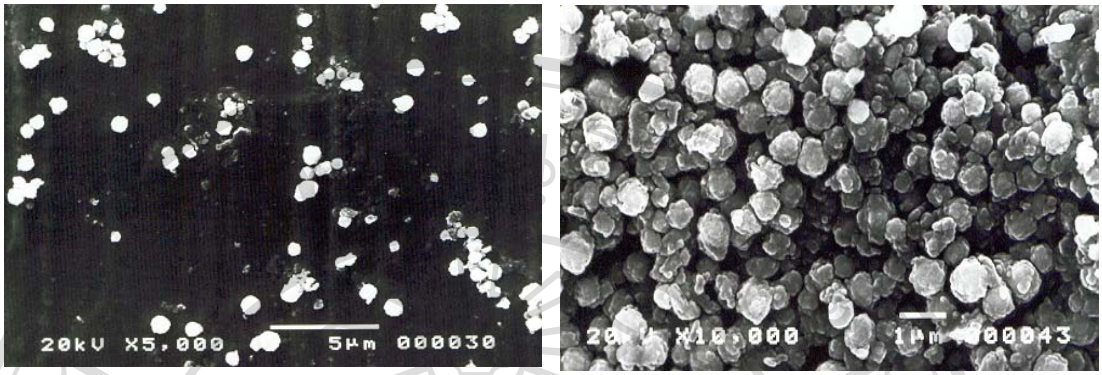
3.1.2.1 BNT Powders from Hydrothermal Process

3.1.2.1.1 Effect of Holding Period at 200 ^{o}C



(a) (b)

(c) (d)

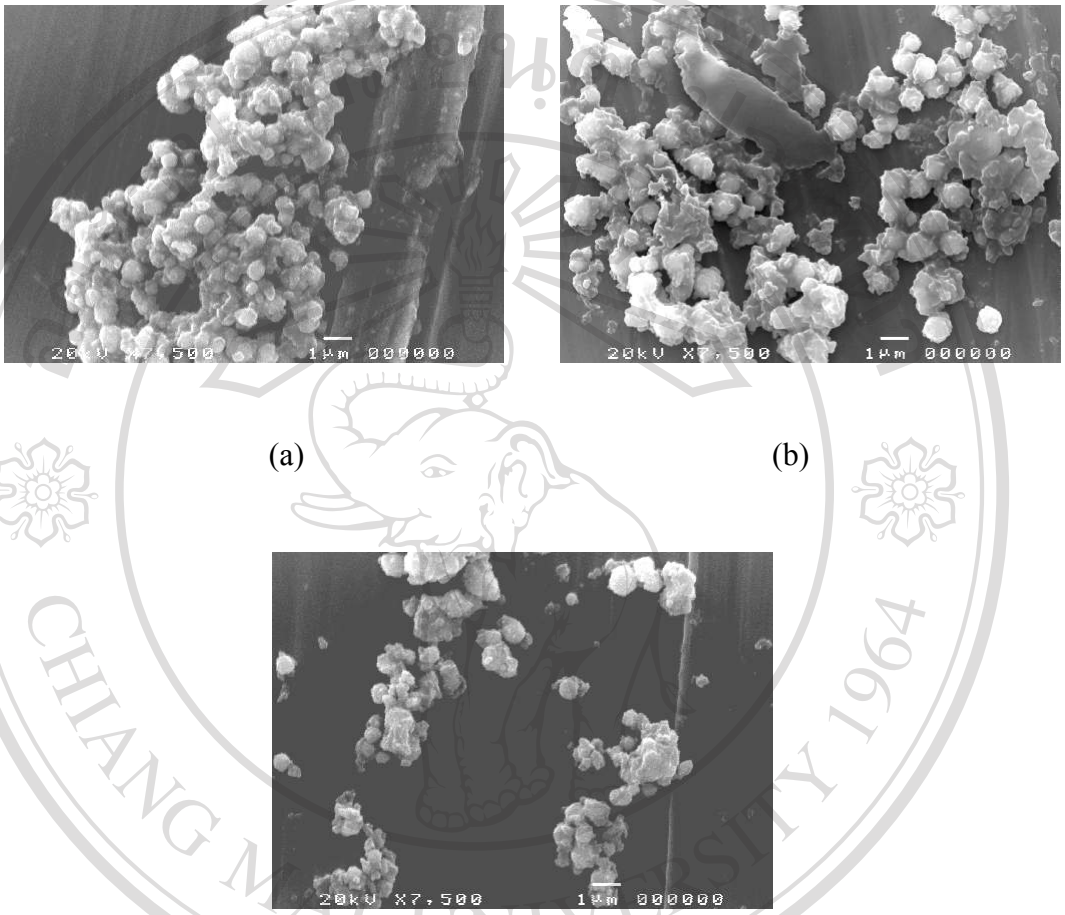


(e) (f)

Figure 3.9 SEM micrographs of BNT powders obtained from hydrothermal process at 200 °C using 10 M NaOH as a mineralizer with different holding periods of (a) 10 h, (b) 15 h, (c) 20 h, (d) 25 h, (e) 35 h and (f) 40 h.

Figure 3.9 [(a)-(f)] shows SEM micrographs of BNT powders synthesized by a hydrothermal process at 200 °C using 10 M NaOH as a mineralizer with different holding periods. As the holding period increased, the particle size and the agglomeration increased significantly. The particle size increased from 0.1 - 0.4 µm, (Figure 3.9 (a-e)), to about 0.5 µm, (Figure 3.9 (f)), when the holding period increased from 10 to 40 h.

3.1.2.1.2 Effect of Holding Period at 150 ^{o}C



(a) (b)

(c)

Figure 3.10 SEM micrographs of BNT powders obtained from hydrothermal process at 150 °C using 12 M NaOH as a mineralizer with different holding periods of (a) 5.45 h, (b) 15 h and (c) 20 h.

Figure 3.10 [(a)-(c)] shows SEM micrographs of BNT powders synthesized by hydrothermal process at 150 °C using 12 M NaOH as a mineralizer with different holding periods. As the holding period increased, the particle size and the agglomeration increased significantly. The particle size increased from 0.1 – 0.4 μm, (Figure 3.10 (a-b)), to about 0.5 μm, (Figure 3.10 (c)), when the holding period increased from 5 to 20 h.

3.1.2.1.3 Effect of Holding Period at 175 ^{o}C

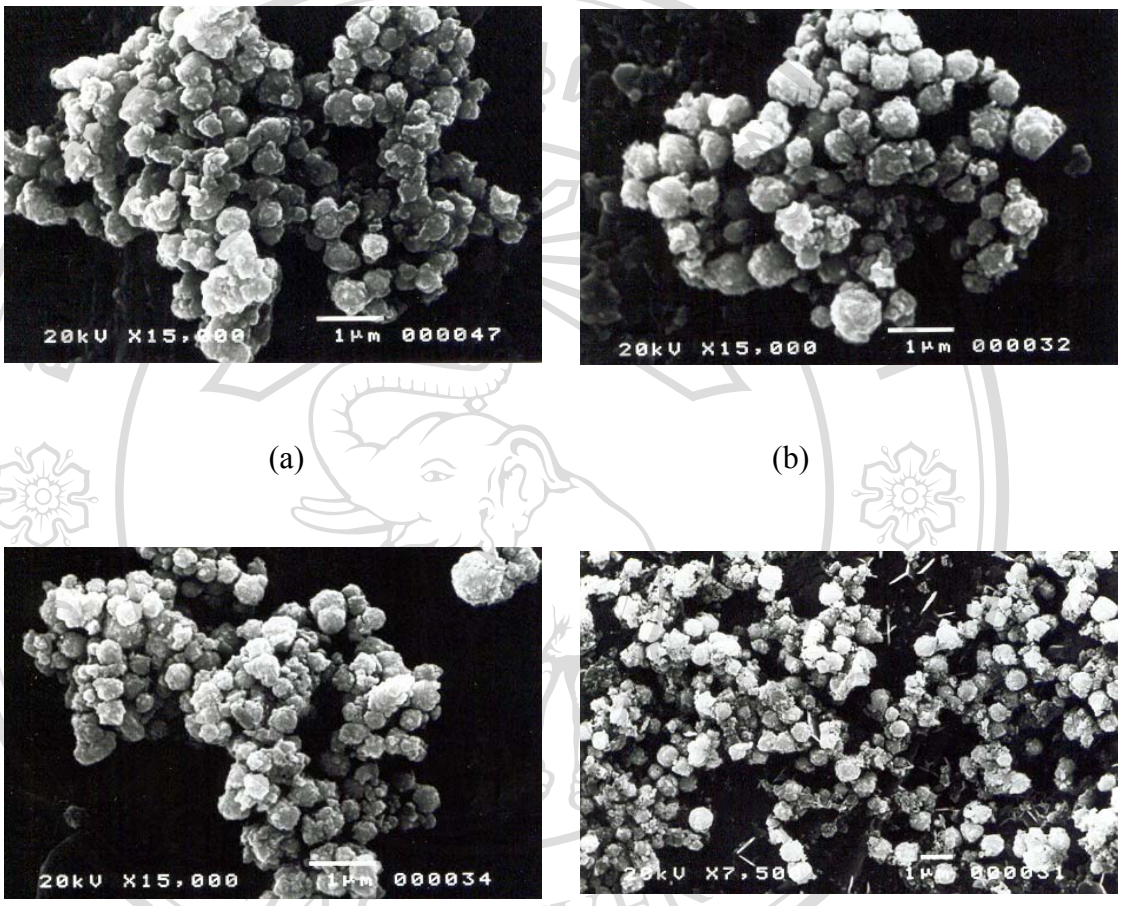
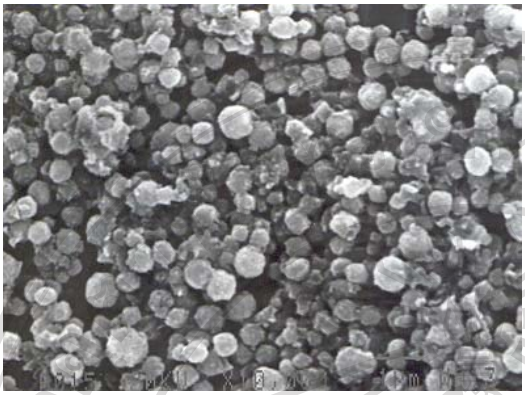


Figure 3.11 SEM micrographs of BNT powders obtained from hydrothermal process at 175 °C using 12 M NaOH as a mineralizer with different holding periods of (a) 5 h, (b) 10 h, (c) 25 h and (d) 30 h.

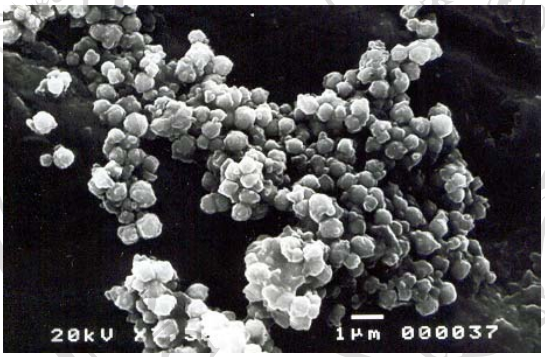
Figure 3.11 [(a)-(d)] shows SEM micrographs of BNT powders synthesized by a hydrothermal process at 175 °C using 12 M NaOH as a mineralizer with different holding periods. As the holding period increased, the particle size and the agglomeration increased significantly. The particle size increased from 0.3 – 0.4 μm, (Figure 3.10 (a-b)), to about 0.5 μm, (Figure 3.10 (c-d)), when the holding period increased from 5 to 30 h.

3.1.2.1.4 Effect of Holding Period at 200 ^{o}C



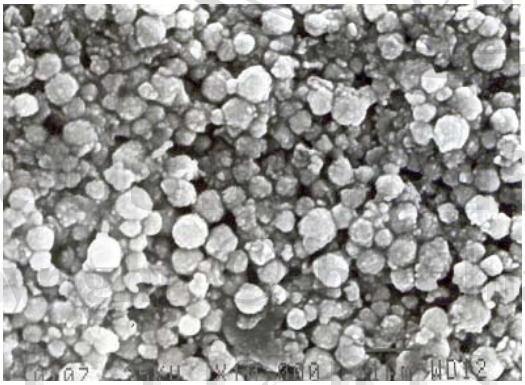
(a)

(b)

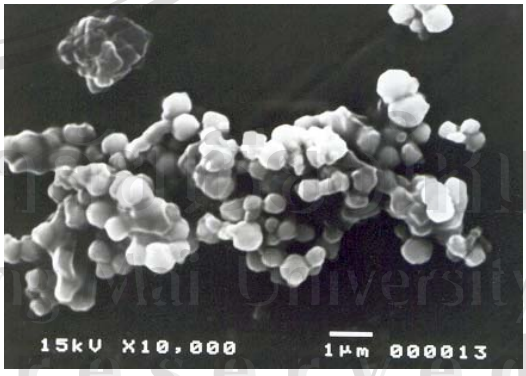


(c)

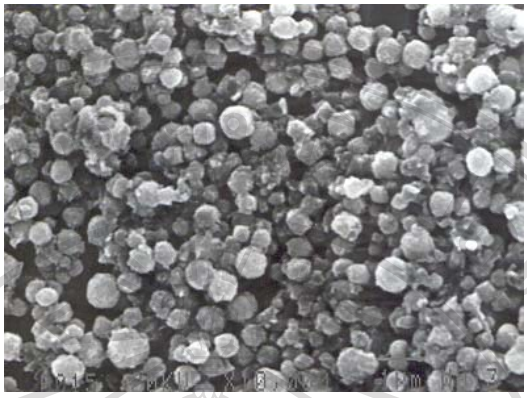
(d)



(e)



(f)



(g)

Figure 3.12 SEM micrographs of BNT powders obtained from hydrothermal process at 200 °C using 12 M NaOH as a mineralizer with different holding period of (a) 5 h, (b) 10 h, (c) 15 h, (d) 20 h, (e) 25 h, (f) 40 h and (g) 60 h.

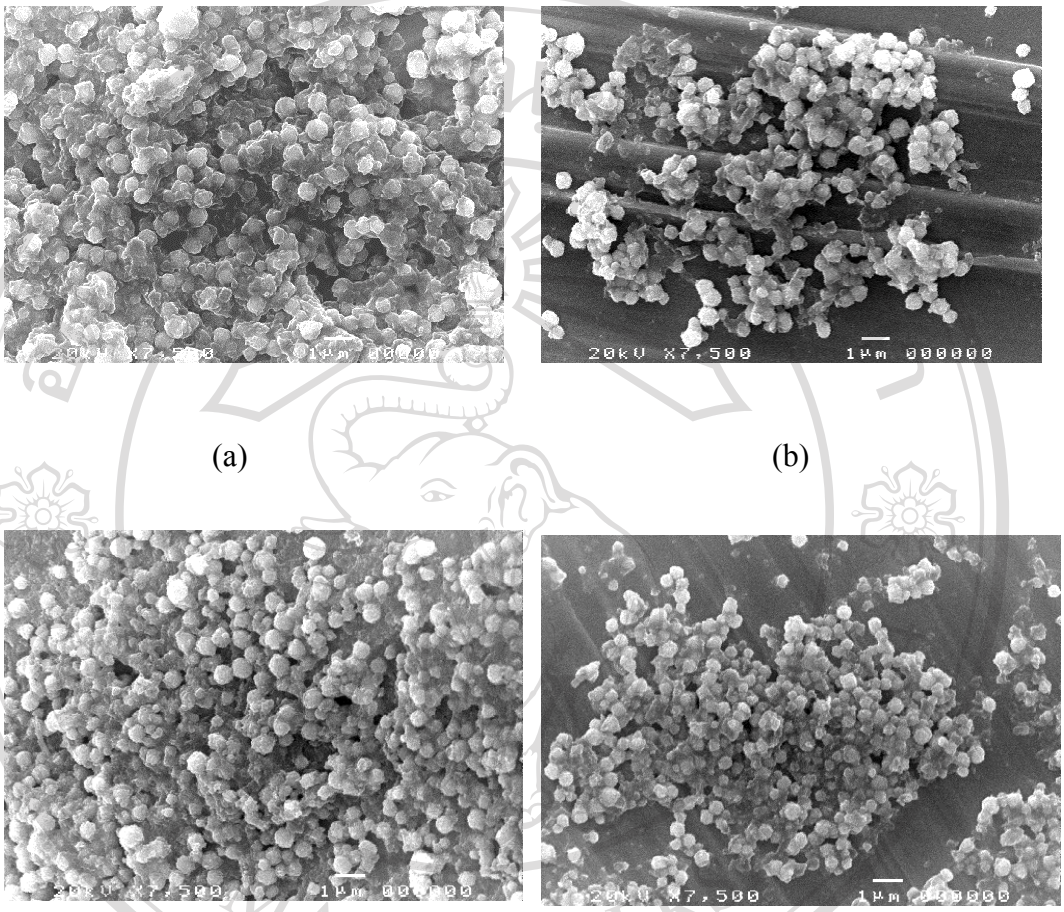
Figure 3.12 [(a)-(g)] shows SEM micrographs of BNT powders synthesized by a hydrothermal process at 200 °C using 12 M NaOH as a mineralizer with different holding periods. As the holding period increased, the particle size and the agglomeration increased significantly. The particle were spherical and particle size increased from 0.1 - 0.4 μm, (Figure 3.12 (a-c)), to about 0.5 μm, (Figure 3.12 (d-f)) and to 0.7 μm, (Figure 3.12 (g) when the holding period increased from 5 to 60 h.

From XRD and SEM investigations the optimum condition for hydrothermal synthesis of BNT powders was obtained at pH 13 and temperature of 150 ^{o}C with holding period of 5 hours to produce spherical particle with average particle size of 0.1 μm.

Table 3.2 Comparison the particle size of BNT powders from different process

Process	Sample	Calcination temperature (^{o}C)	Reaction time (h)	Average particle sizes (μm)
Conventional	-	-	-	-
Sol-gel	-	-	-	-
Co-precipitation[63]	Bismuth titanate (BT)	520	1	0.015
	BNT	520	1	0.021
	BNT	560	1	0.023
	BNT	630	1	0.080
Hydrothermal[66]	BNT	160-220	2-24	3.5-5.5
This work	BNT	150-200	5-20	0.1-0.5

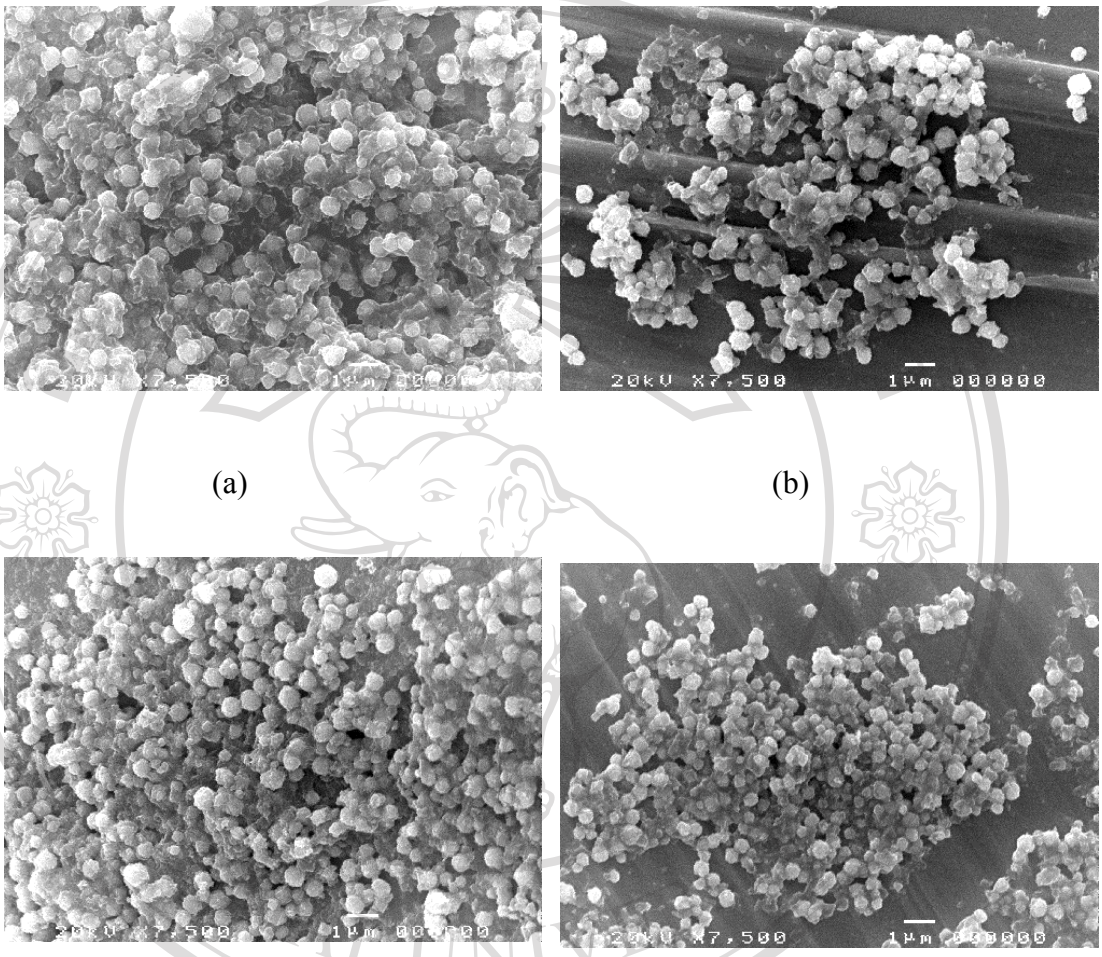
3.1.2.2 BLNT Powders from Hydrothermal Process



(a) (b)

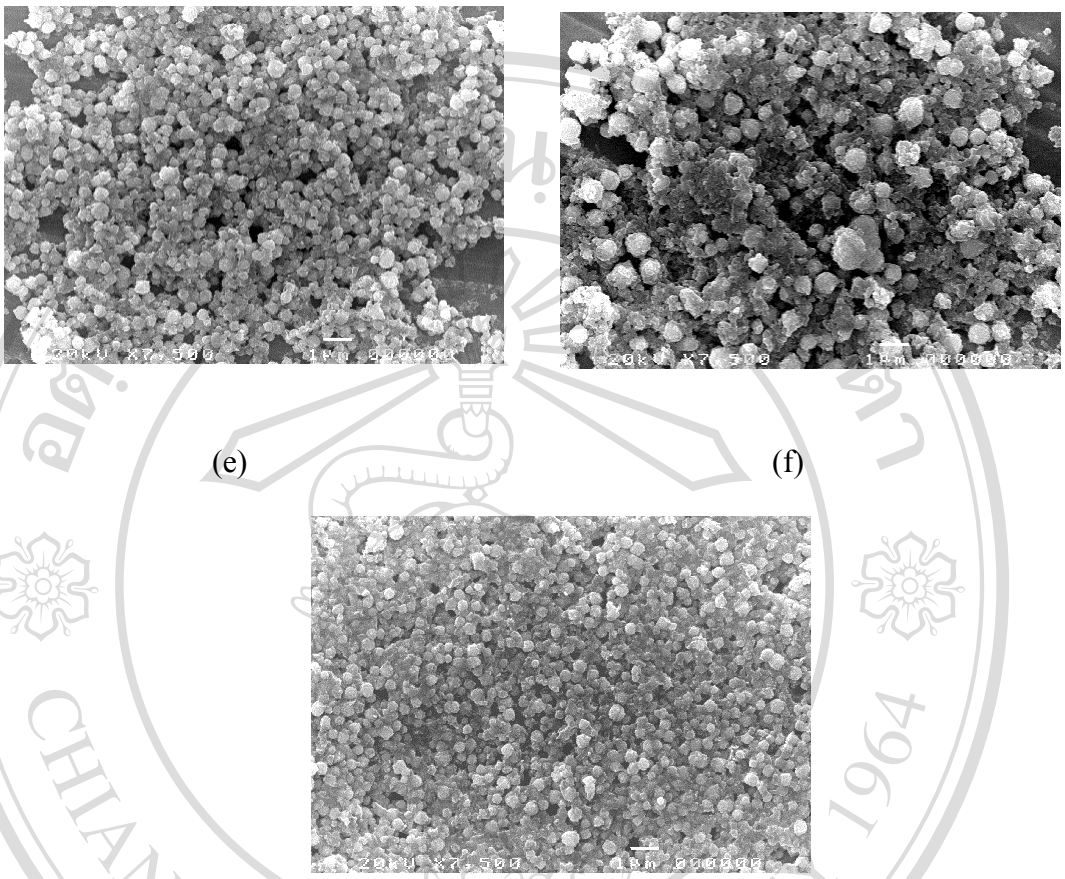
(c) (d)

3.1.2.2 BLNT Powders from Hydrothermal Process



(a) (b)

(c) (d)



(e) (f)

(g)

Figure 3.13 SEM micrographs of BLNT powders obtained from hydrothermal process at 200 °C with holding period of 20 h using 12 M NaOH as a mineralizer and different mole% La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La.

Figure 3.13 [(a)-(g)] shows SEM micrographs of BLNT powders synthesized by a hydrothermal process at 200 °C with a holding period of 20 hours using 12 M NaOH as a mineralizer and different mole %La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La. As the amount of %La increased, the particle size and the agglomeration decreased significantly. The particle were spherical and the average particle size decreased from 0.5 μm, (Figure 3.13 (a-c)), to about 0.4 μm, (Figure 3.13 (d-f)) and to 0.3 μm, (Figure 3.13 (g) when the %La increased from 1 to 6.

Table 3.3 Comparison the particle size of BLNT powders from different process

Process	Sample	Calcination temperature (^{o}C)	Reaction time (h)	Average particle sizes (μm)
Conventional	-	-	-	-
Sol-gel	-	-	-	-
Co-precipitation	-	-	-	-
Hydrothermal	-	-	-	-
This work	BLNT	200	200	0.1-0.5

3.1.3 PARTICLE SIZE DISTRIBUTION ANALYSIS

3.1.3.1 BNT and BLNT Powders from Hydrothermal Process

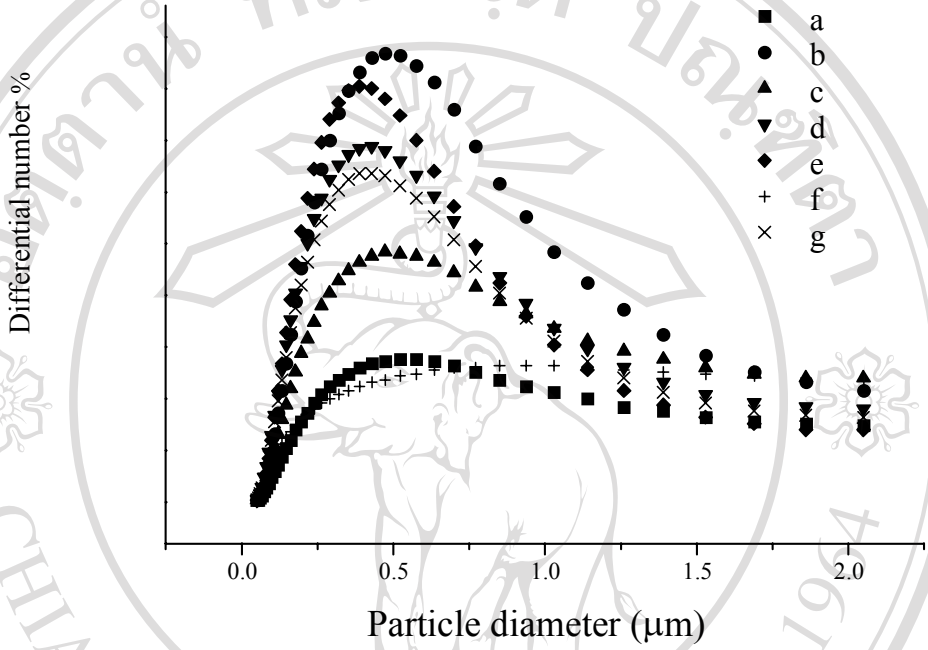


Figure 3.14 Particle size distribution analysis of BLNT powders obtained from hydrothermal process at 200 °C with holding period of 20 h using 12 M NaOH as a mineralizer and different mole % La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La.

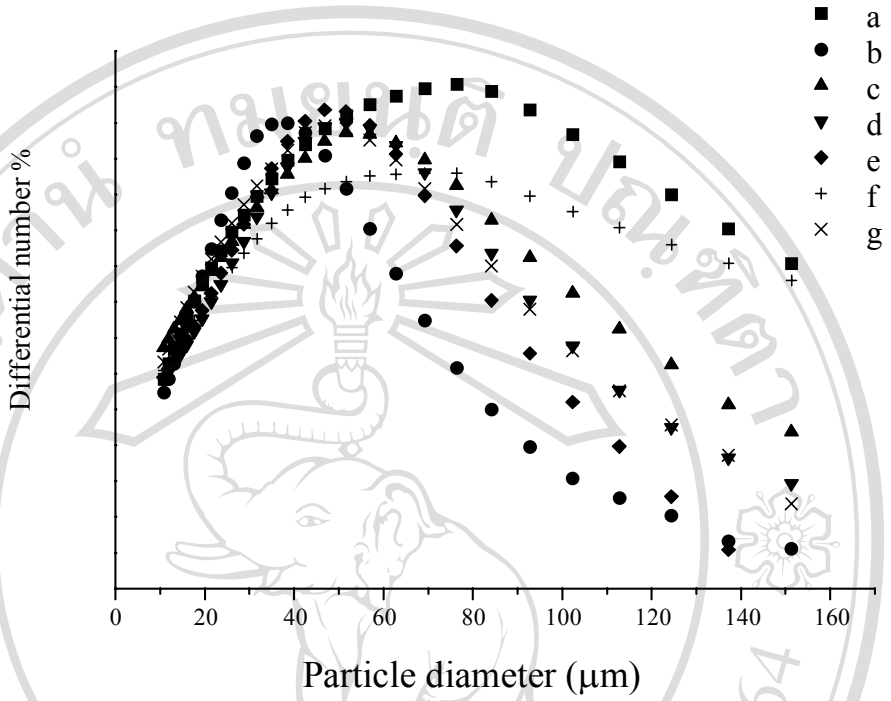


Figure 3.15 Particle size distribution analysis of BLNT powders obtained from hydrothermal process at 200 °C with holding period of 20 h using 12 M NaOH as a mineralizer and different mole % La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La.

Particle size distribution curves of BNT and BLNT powders obtaineded from a hydrothermal process at 200 °C with a holding period of 20 hours using 12 M NaOH as a mineralizer are shown in Figure 3.14 and 3.15.

Figure 3.14 (a) and 3.15 (a) show the distribution particle diameter of BNT powders. There are two regions of particle distribution. The first region consists of particles with an average size of 0.5 μm and the second region consists of agglomerated particles with an average size of 76 μm.

The distribution particle diameter of BLNT powders at different mole % La of (b) 1%La, (d) 3%La, (e) 4%La and(g) 6%La are shown in Figure 3.14 (b, d, e and g) and Figure 3.15 (b, d, e and g). There are two regions of particle distribution. The first region consists of particles with an average size of 0.4 μm which in good agreement with the SEM result (Figure 3.14 (b, d, e and g)). The second region consists of agglomerated particles with an average size of 35, 50, 46 and 46 μm (Figure 3.15 (b, d, e and g)).

The distribution particle diameter of BLNT powders at different mole % La of (c) 2%La and (f) 5%La are shown in Figure 3.14 (c and f) and Figure 3.14 (c and f). There are two regions of particle distribution. The first region consists of particles with an average size of 0.5 and 0.6 μm which is not consistent with the trend of reduction in size when mole percentage of lanthanum increases (Figure 3.14 (c and f)). The second region consists of agglomerated particles with an average size of 52 and 76 μm (Figure 3.15 (c and f)). In this case the result from particle size analysis is not in good agreement with the result from SEM.

Table 3.4 Comparison the particle size of BNT and BLNT powders from particle size distribution analysis

Sample	Average particle sizes (μm)	
	First region	Second region
BNT	0.5	76
BLNT (1%La)	0.4	35
BLNT (2%La)	0.5	52
BLNT (3%La)	0.4	50
BLNT (4%La)	0.4	46
BLNT (5%La)	0.6	76
BLNT (6%La)	0.4	46

3.2 CERAMICS CHARACTERIZATION

3.2.1 Densification of BNT and BLNT Ceramics

The BNT powders from hydrothermal process at synthesis temperature of 200 °C with holding period of 20 hours using 12.0 M NaOH as a mineralizer were selected for densification measurements because of their similar rhombohedral structure. The BNT powders from hydrothermal processes was pressed and sintered at temperatures between 800 °C and 1000 °C for 1-3 hours. The results are illustrated in Table 3.5.

The theoretical density of the rhombohedral BNT, estimated from the lattice parameters, was 5.997 g/cm^3. The sintered pellet density was measured by an immersion technique and calculated from Archimedes' principle.

From Table 3.5, the suitable sintering temperature for BNT ceramics obtain from hydrothermal process was in the range of sintering temperature of 900-1000 ^{o}C for 1-3 hours and BLNT ceramics obtained from hydrothermal process was in the range of sintering temperature of 800-1000 °C for 3 hours.

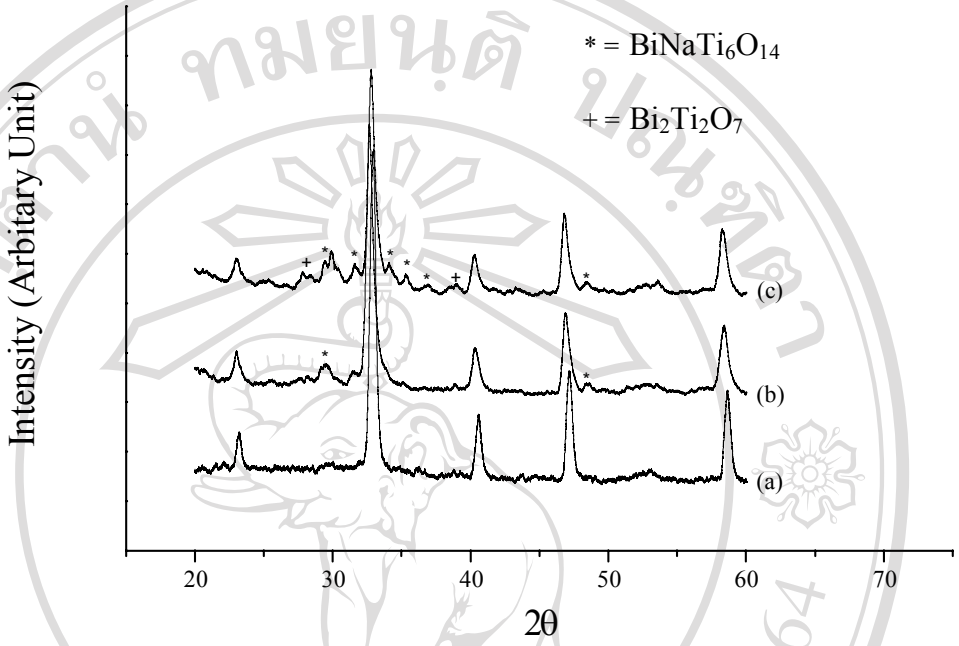
Table 3.5 The measured density, % theoretical density and % porosity of BNT and BLNT ceramics obtained from hydrothermal process.

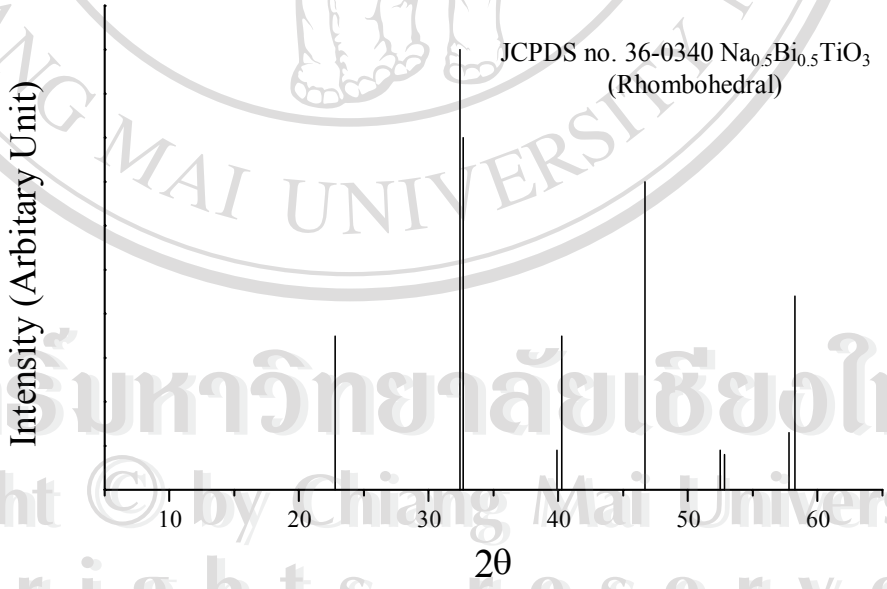
Sample	Sintering temperature (°C)	Sintering time (h)	Measured density (g/cm^3)	Theoretical density (%)	Porosity (%)
BNT	900	1	3.612	60.2	39.8
BNT	900	2	3.768	62.8	37.2
BNT	900	3	3.877	64.5	35.5
BNT	1000	1	5.334	88.9	11.1
BNT	1000	2	5.378	89.7	10.3
BNT	1000	3	5.419	90.4	9.6
BLNT (1%)	800	3	3.901	65.1	34.9
	900	3	4.292	71.6	28.4
	1000	3	5.612	93.6	6.4
BLNT (2%)	800	3	3.750	62.5	37.5
	900	3	4.168	69.5	30.5
	1000	3	5.705	95.1	4.9
BLNT (3%)	800	3	3.883	64.8	35.2
	900	3	4.241	70.7	29.3
	1000	3	5.680	94.7	5.3
BLNT (4%)	800	3	3.805	63.5	36.5
	900	3	5.340	89.0	11.0
	1000	3	5.839	97.4	2.6
BLNT (5%)	800	3	3.802	63.4	36.6
	900	3	4.252	70.9	29.1
	1000	3	5.636	94.0	6.0
BLNT (6%)	800	3	3.631	60.6	39.4
	900	3	3.929	65.5	34.5
	1000	3	5.645	94.1	5.9

Table 3.6 The comparison of average grain size and theoretical density (%) of BNT ceramics from different process

Process	Sintering temperature (°C)	Sintering time (h)	Average grain sizes (μm)	Theoretical density (%)
Conventional[16]	1070	2	4.1	94
	1050	3	3.8	95
	1050	2	3.1	94
	1050	1	3.8	94
Conventional[5]	1125	1	4	80
	1150	1	6	90
	1175	1	8	96
	1200	1	34	93
Conventional[104]	1070-1100	2	1-3	-
Conventional[105]	1130	2	10	-
Sol-gel	-	-	-	-
Co-precipitation	-	-	-	-
Hydrothermal	-	-	-	-
This work	1000	1	1.0	88.9
	1000	2	1.5	89.7
	1000	3	3.0	90.4

3.2.2 XRD patterns of BNT Ceramics





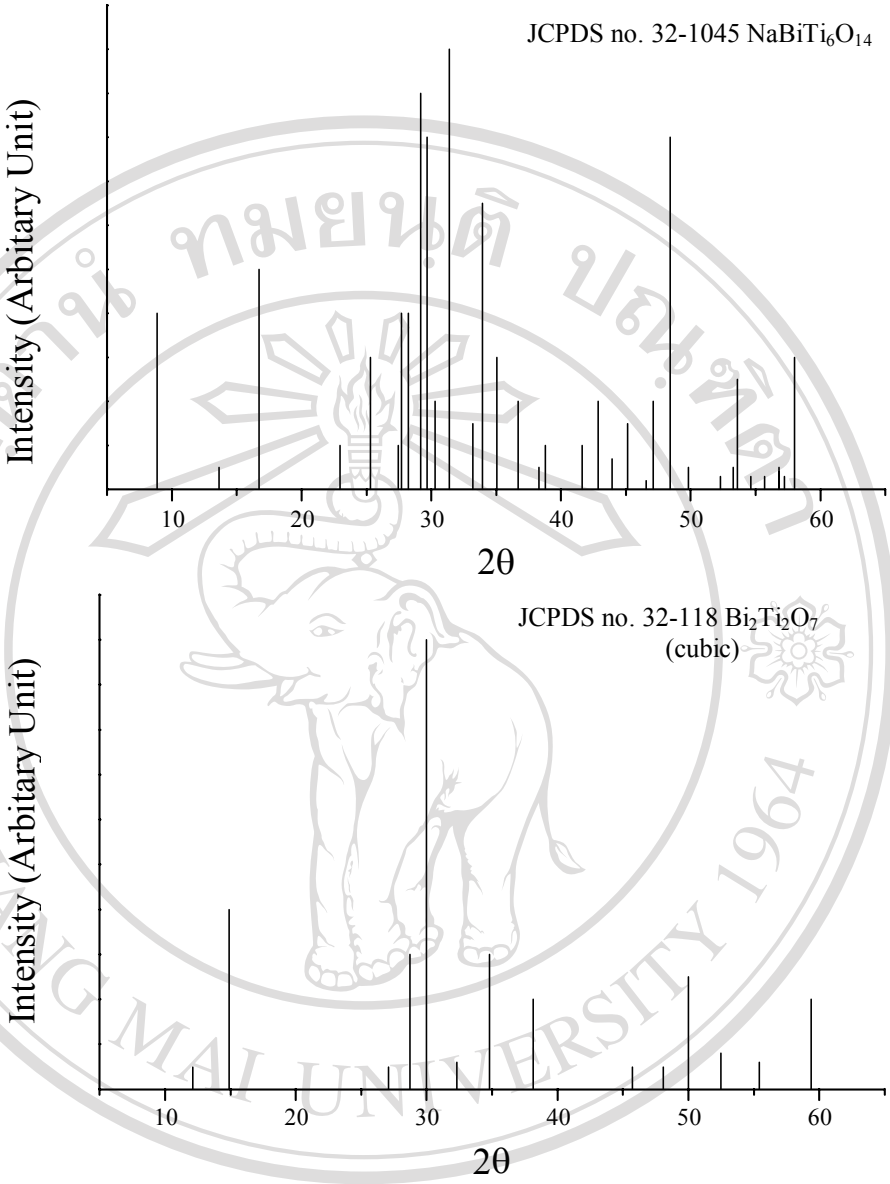
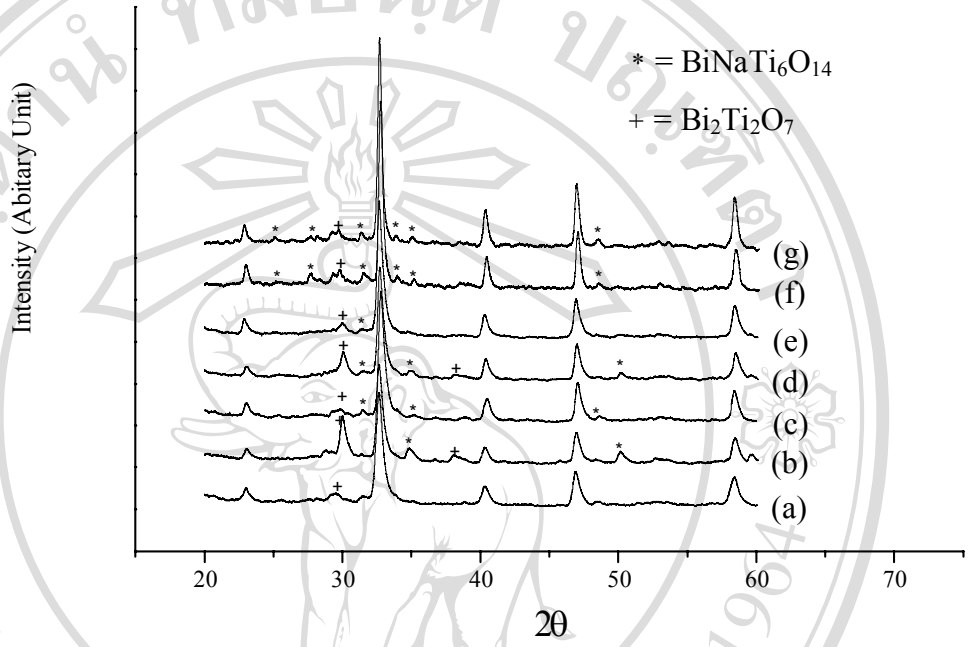
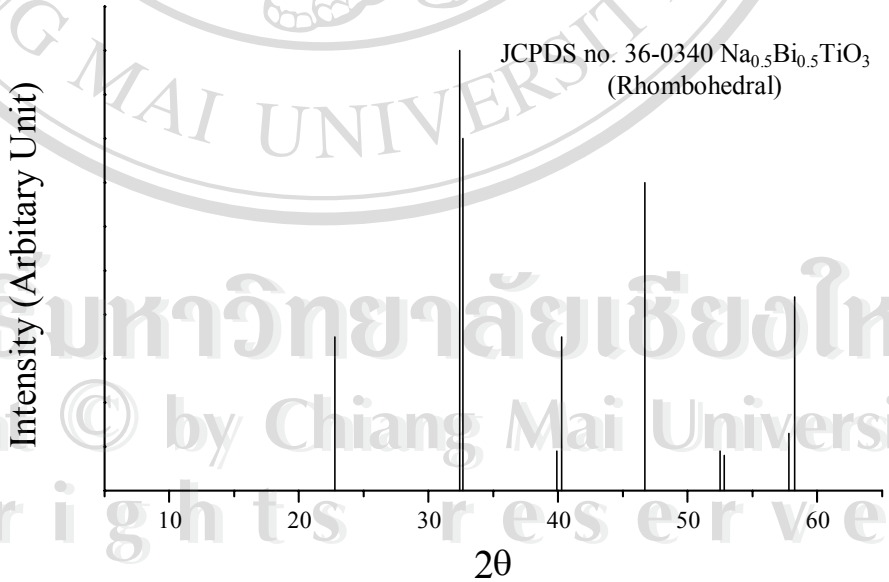


Figure 3.16 XRD patterns of sintered BNT ceramics obtained from hydrothermal process sintered at 1000 °C for (a) 1 h, (b) 2 h and (c) 3 h.

XRD patterns of hydrothermally synthesized BNT sintered at 1000 ^{o}C with different sintering times are shown in Figures 3.16. XRD results showed that BNT ceramics displayed a rhombohedral structure (JCPDS File no. 36-0340) with a sintering time of 1 hour. The second phases of $Bi_2Ti_2O_7$ and $BiNaTi_6O_{14}$ were found in the sample with a sintering time of 2 and 3 hours (Fig. 3.16 (b,c)).

3.2.3 Effect of Sintering Time on XRD Patterns of BLNT Ceramics with Different Mole %La





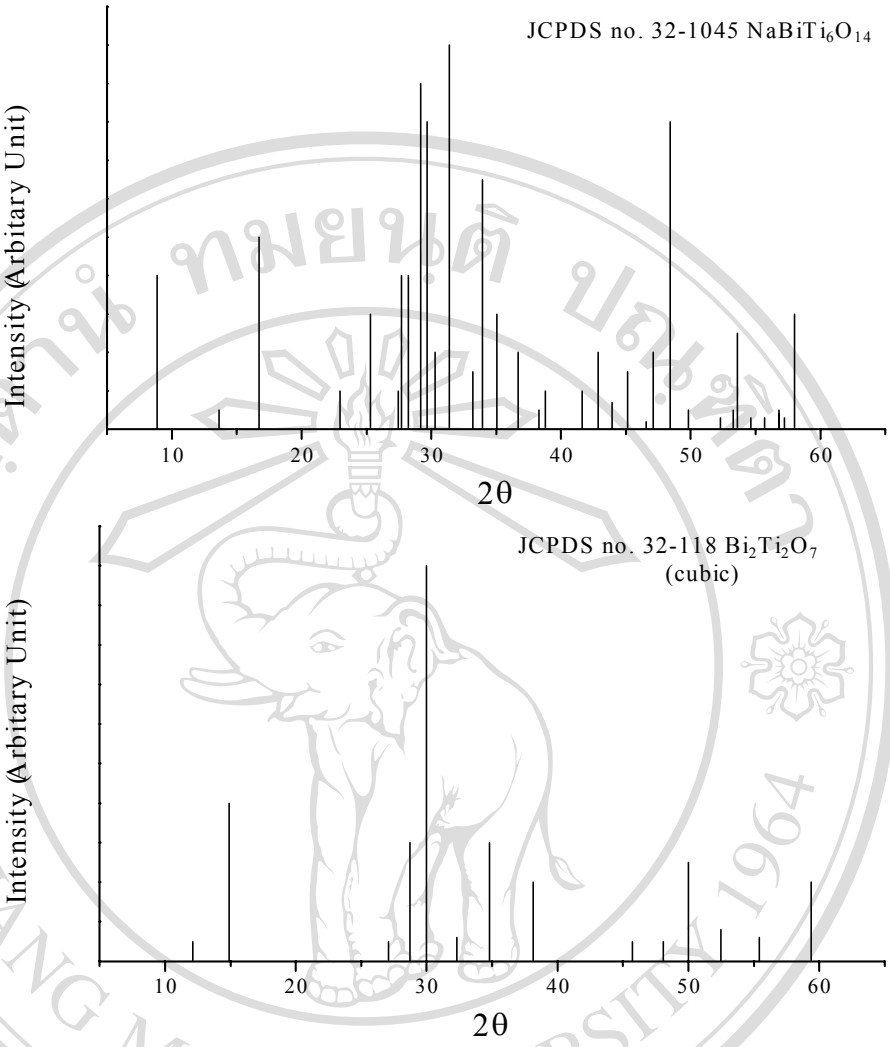
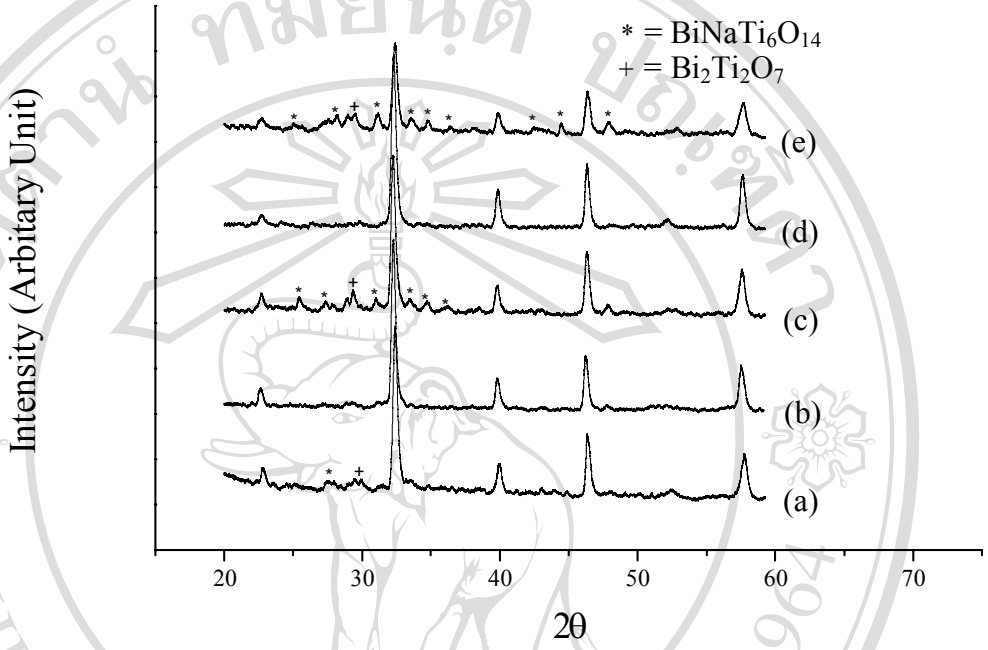
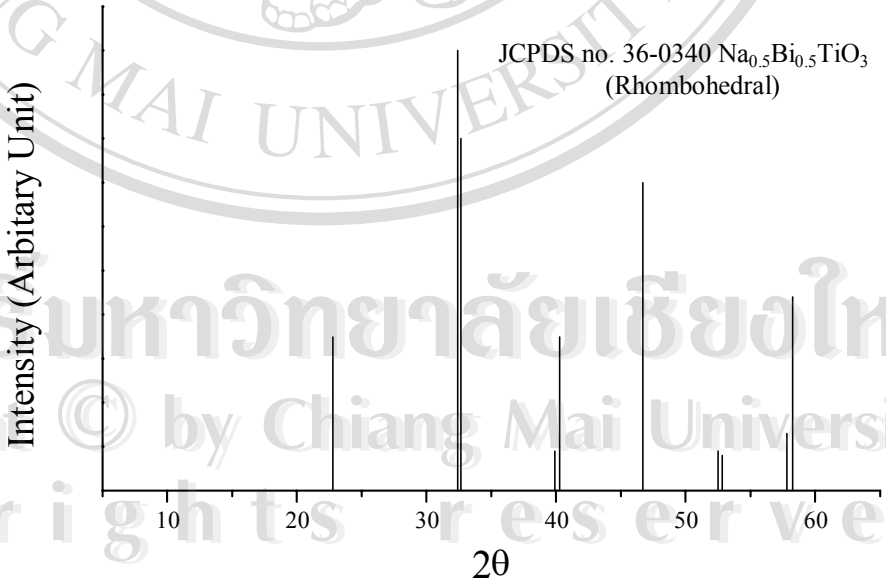


Figure 3.17 XRD patterns of sintered BLNT ceramics obtained from hydrothermal process sintered at 1000 °C for 2 h with different mole %La of (a) 0 %La, (b) 1 %La, (c) 2 %La, (d) 3 %La, (e) 4 %La, (f) 5 %La and (g) 6 %La.

XRD pattern of BLNT ceramics shows the rhombohedral structure of undoped with lanthanum sintered at 1000 ^{o}C for 2 hours in Fig. 3.17 (a). In contrast to this, doping of lanthanum 1-6 % (La) stabilizes the perovskite phase as shown in Fig. 3.17 (b-g). The second phases of $Bi_2Ti_2O_7$ and $BiNaTi_6O_{14}$ (peak at 2θ of 30)were also found in the samples.

3.2.4 Effect of Sintering Time on XRD patterns of BLNT Ceramics with Different Mole %La





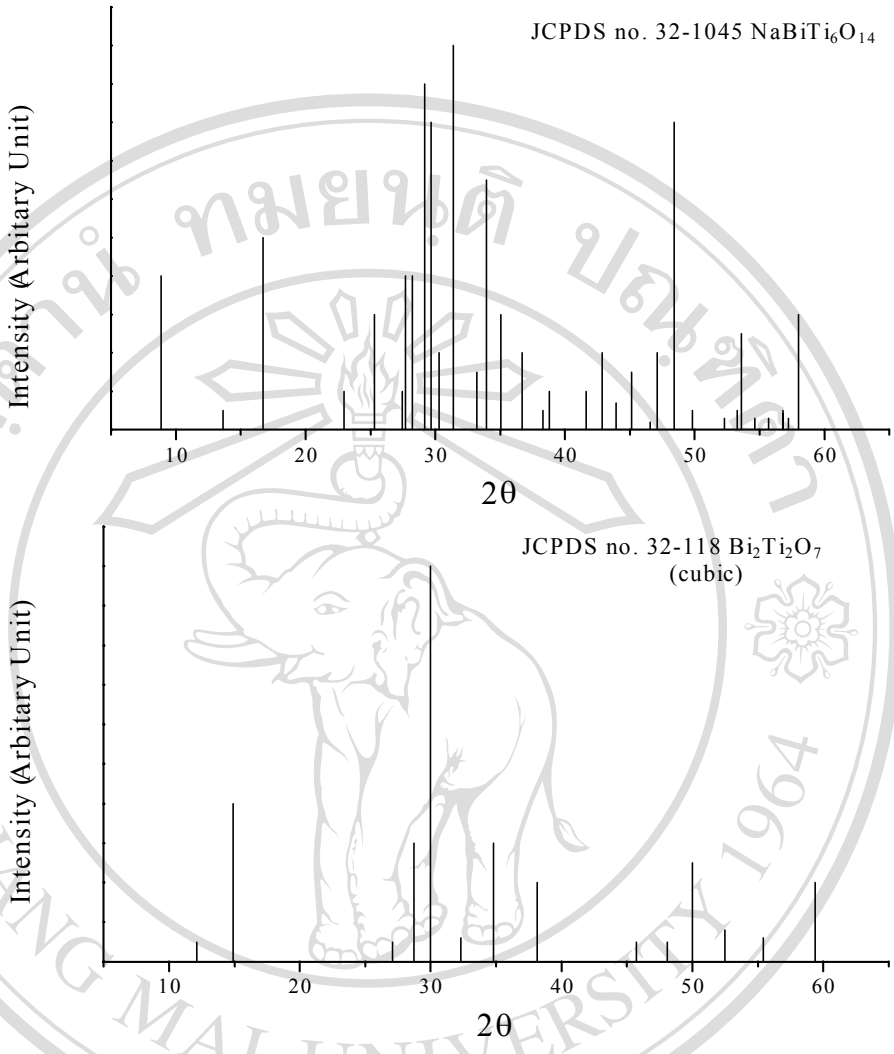


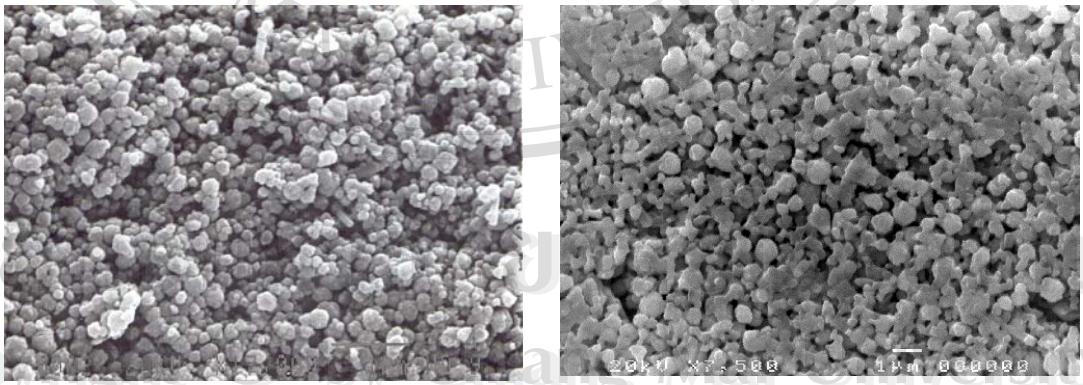
Figure 3.18 XRD patterns of sintered BLNT ceramics obtained from hydrothermal process sintered at 1000 °C for 3 h with different mole %La of (a) 1 %La, (b) 2 %La, (c) 3 %La, (d) 4 %La, and (e) 5 %La.

XRD results showed that BLNT ceramics with the rhombohedral structure when doped with lanthanum 2 %and 4 %, and sintered at 1000 ^{o}C for 3 hours (Fig. 3.18 (b,d)). For the BLNT ceramics doped with 1%, 3% and 5% lanthanum and sintered at 1000 ^{o}C for 3 hours (Fig. 3.18 (a,c,e)), showed the rhombohedral structure with second phase (peaks at 2θ of 30) in the perovskite phase.

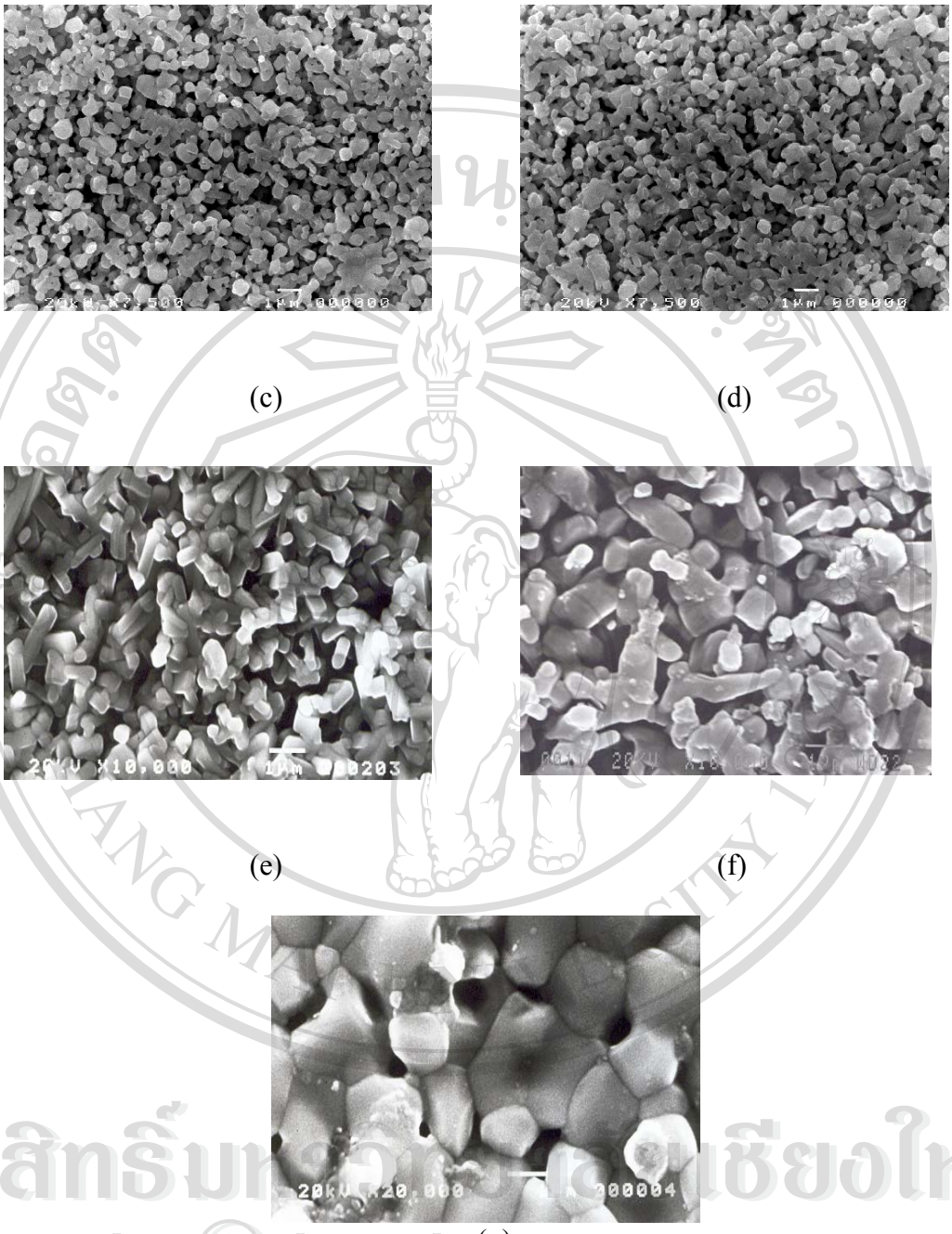
3.2.5 Microstructure of BNT Ceramics

The microstructure and grain size of the sintered samples with differnet sintering temperature and time were studied using scanning electron microscopy.

Figure 3.19 shows SEM micrographs of BNT ceramics obtained from hydrothermal process sintered at 800 °C for 3 hours and 900-1000 °C for 3 hours. The microstructure of hydrothermal BNT ceramic with sintering at 800 °C for 3 hours showed the spherical and agglomerate of particle with the average size of 0.10 μm (Fig. 3.19 (a)). At the higher sintering temperature of 900 ^{o}C for 1-3 hours, the BNT ceramics were irregular in shape and melted together with the average size in the ranging of 0.50-1.0 μm (Fig. 3.19 (b-d)). At the higher sintering temperature and longer sintering time, a higher sintered density was observed. The BNT ceramic sintering at 1000 ^{o}C for 1-3 hours are shown in Figure 3.19 (e-g). The grain size increased with the longer sintering time and the average grain size was 1.0-3.0 μm.



(a) (b)



(c) (d)

(e) (f)

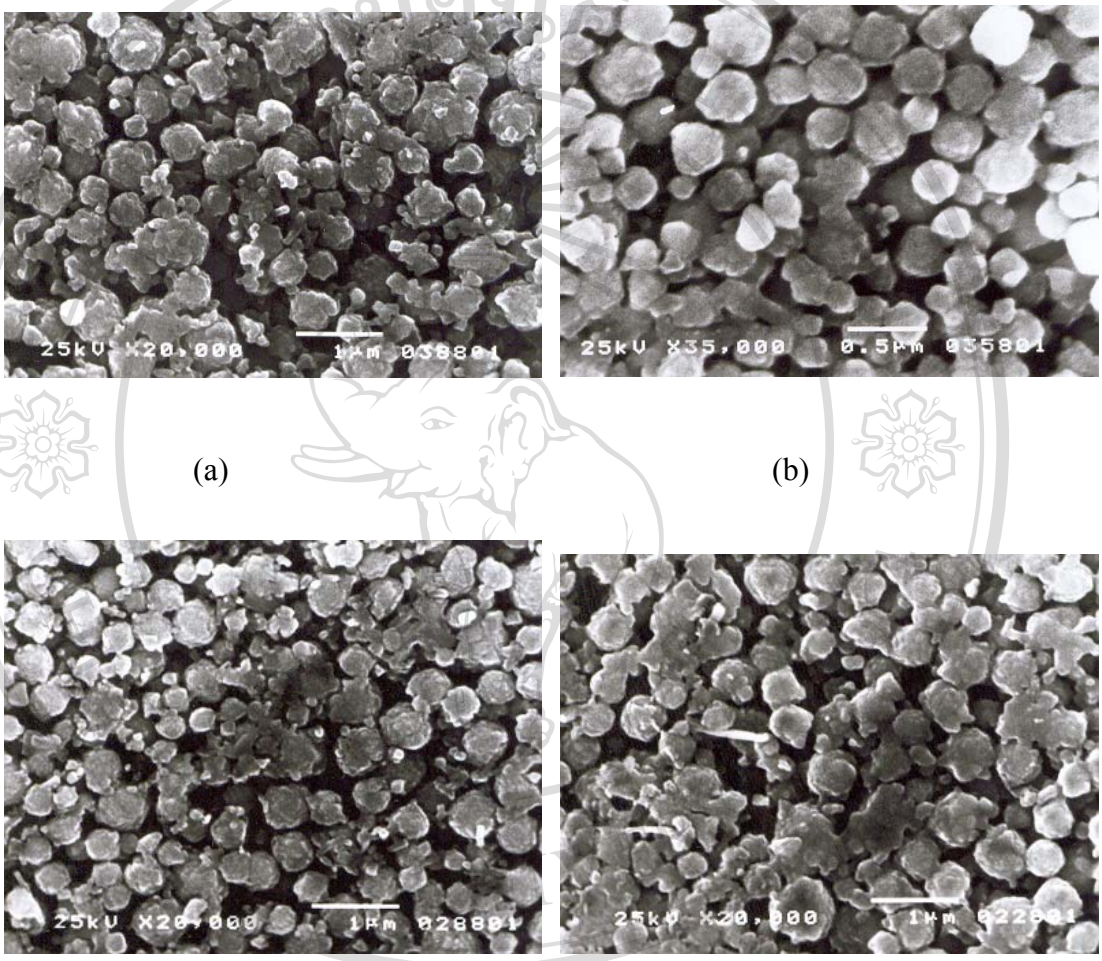
(g)

Figure 3.19 SEM micrographs of BNT ceramics obtained from hydrothermal process at 200 °C using 12 M NaOH as a mineralizer with different sintering temperature and time of (a) 800 ^{o}C, 3 h, (b) 900 ^{o}C, 1 h, (c) 900 ^{o}C, 2 h, (d) 900 ^{o}C, 3 h, (e) 1000 ^{o}C, 1 h, (f) 1000 ^{o}C, 2 h and (g) 1000 ^{o}C, 3 h.

Table 3.7 The comparison of average grain size and theoretical density (%) of BLNT ceramics from different processes

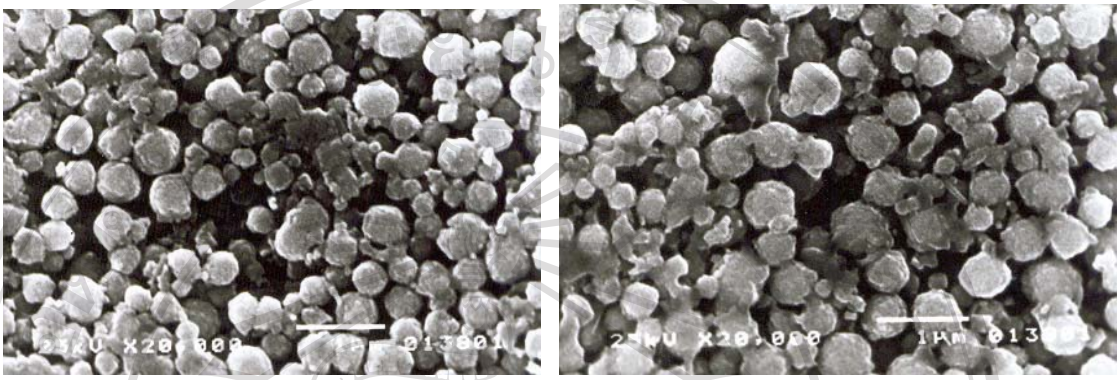
Process	Sintering temperature (°C)	Sintering time (h)	Average grain sizes (μm)	Theoretical density (%)
Conventional[16] BLNT (x=0.017)	1200 1150 1150 1150	3 4 2 1	7.3 6.1 4.6 4.9	93 94 95 95
Sol-gel	-	-	-	-
Co-precipitation	-	-	-	-
Hydrothermal	-	-	-	-
This work BLNT (x=0.020)	1000	3	0.6	95.1

3.2.6 Microstructure of BLNT Ceramics Sintered at 800 ^{o}C with Different Mole %La



(a) (b)

(c) (d)

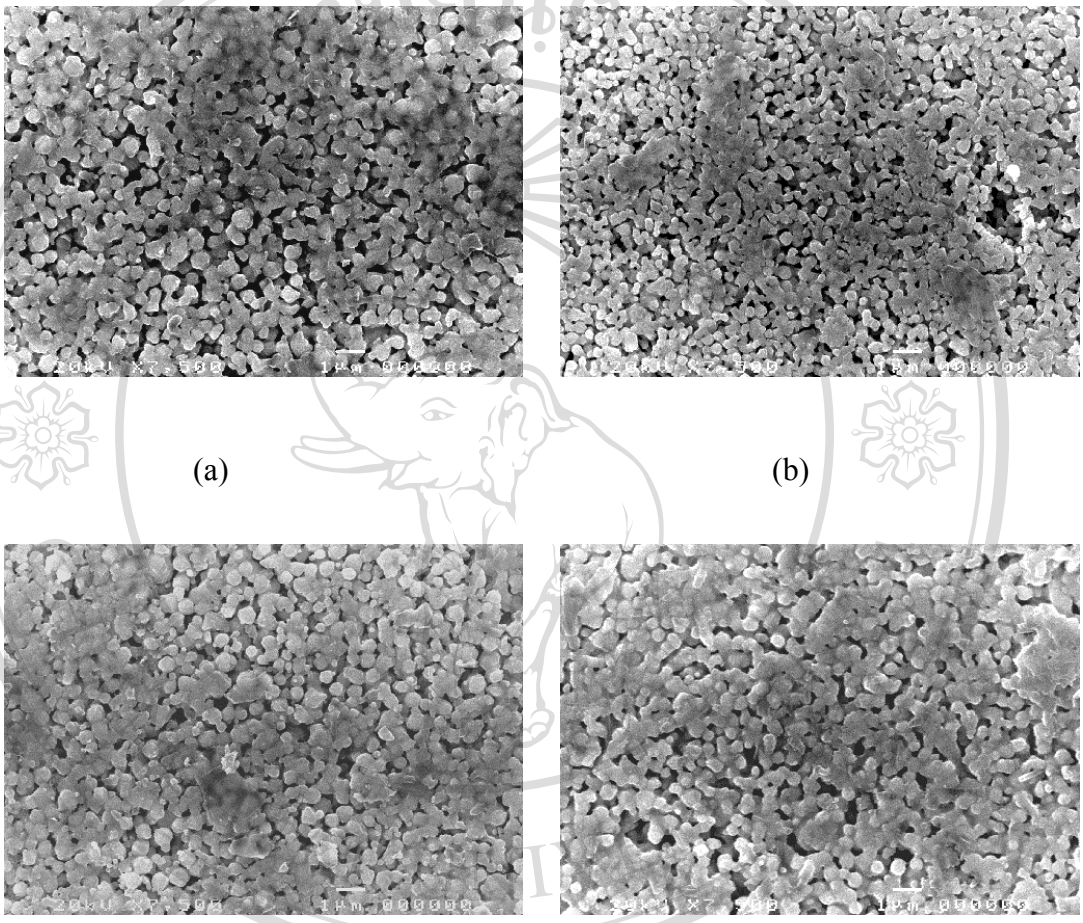


(e) (f)

Figure 3.20 SEM micrographs of sintered BLNT ceramics obtained from hydrothermal process with sintering temperature of 800 °C for 3 h and different mole %La of (a) 1 %La, (b) 2%La, (c) 3 %La, (d) 4 %La, (e) 5 %La and (f) 6 %La.

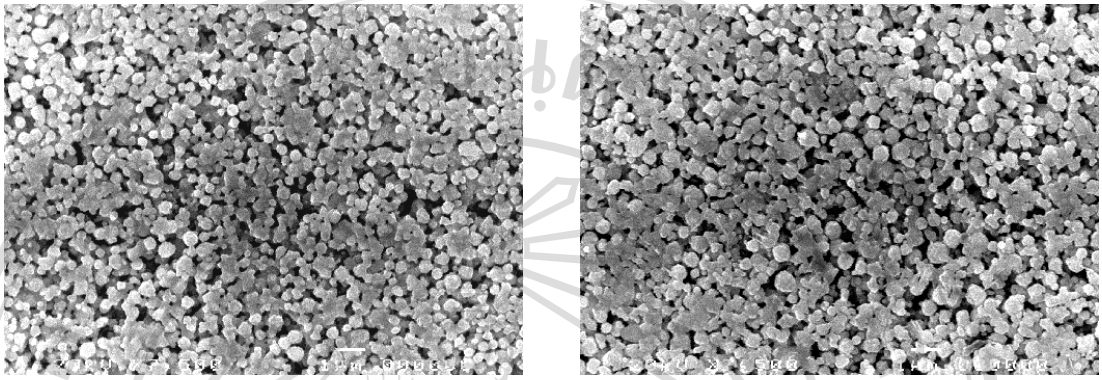
The series of SEM micrographs of BLNT ceramics doped with different mole% of lanthanum, sintered at 800^{o}C for 3 hours are shown in Figure 3.20. In Figure 3.20 (a-f), the micrograph showed the irregular particles with the average grain size of 0.10-0.50 μm and the particles were agglomerated together and the pores were found inside the structure.

3.2.7 Microstructure of BLNT Ceramics Sintered at 900 ^{o}C with Different Mole %La



(a) (b)

(c) (d)

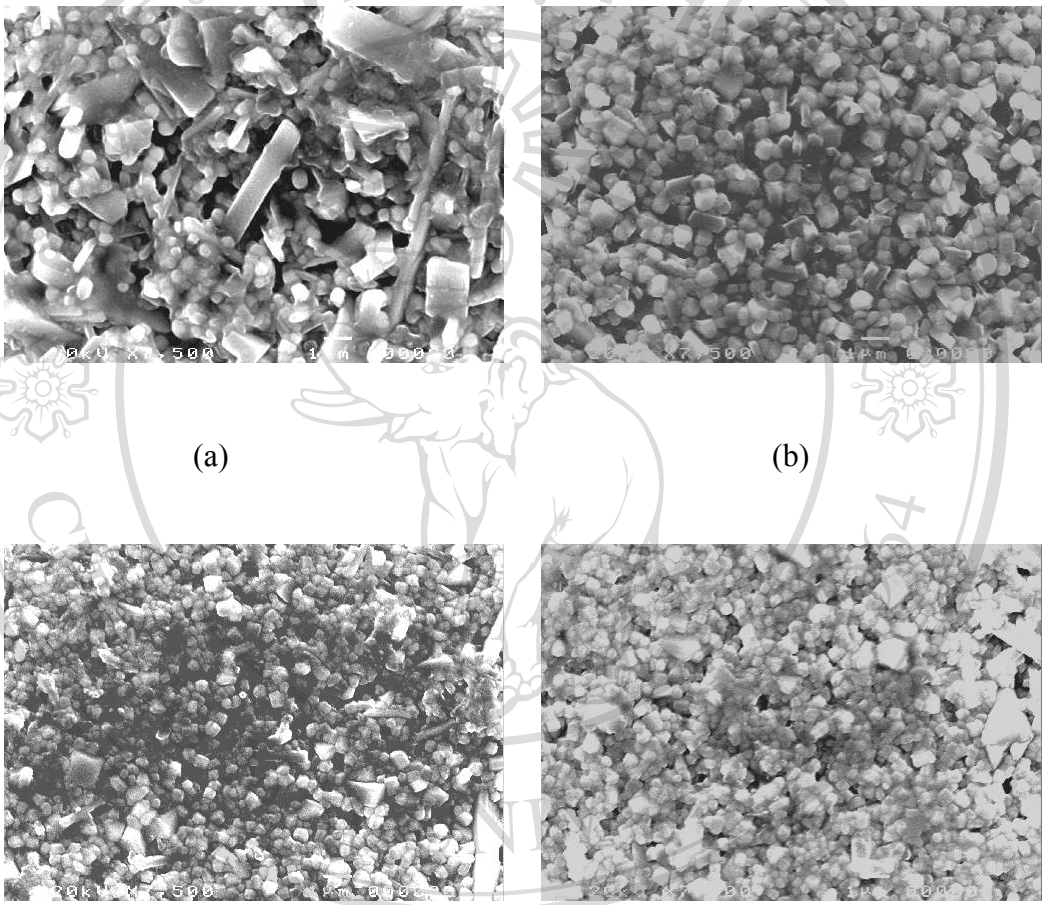


(e) (f)

Figure 3.21 SEM micrographs of sintered BLNT ceramics obtained from hydrothermal process with sintering temperature of 900 °C for 3 h and different mole %La of (a) 1 %La, (b) 2%La, (c) 3 %La, (d) 4 %La, (e) 5 %La and.(f) 6 %La.

Figure 3.21 shows micrographs of BLNT ceramics containing 1-6 mole% lanthanum after cold pressing process and sintering at 900 ^{o}C for 3 hours. Figure 3.21 (a-f), the particles were fused together between inter-small particle and the more pores occurred inside the structure. The average grain size was 1.0-2.0 μm.

3.2.8 Microstructure of BLNT Ceramics Sintered at 1000 ^{o}C for 2 Hours with Different Mole %La



(a) (b)

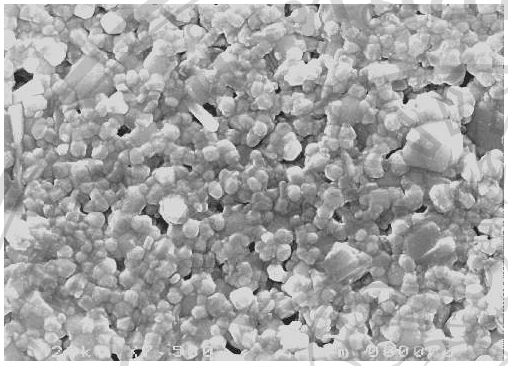
(c) (d)

(e) (f)

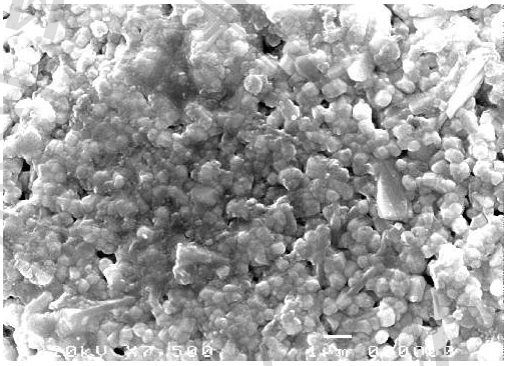
Figure 3.22 SEM micrographs of sintered BLNT ceramics obtained from hydrothermal process with sintering temperatures of 1000 °C for 2 h and different mole %La of (a) 1 %La, (b) 2%La, (c) 3 %La, (d) 4 %La and (e) 5 %La and (f) 6 %La.

Figure 3.22 shows SEM micrograph of BLNT ceramicscontaining 1-6 mole% lanthanum after cold pressing process and sintering at 1000 ^{o}C for 2 hours. Figure 3.22(a) shows the grain size of BLNT ceramicswith the form of platelets and irregular small particle mixed together. The average grain size was 5.0 μm in the long shape of platelets structure and irregular inshape was 0.20 μm. Figure 3.22(b, c, d and f) show the BLNT ceramicswith the average grain size 0.50 μm, the pores occurred inside the structure. In Figure 3.22(e), the average grain size was 1.0 μm.

3.2.9 Microstructure of BLNT Ceramics Sintered at 1000 ^{o}C for 3 Hours with Different Mole %La

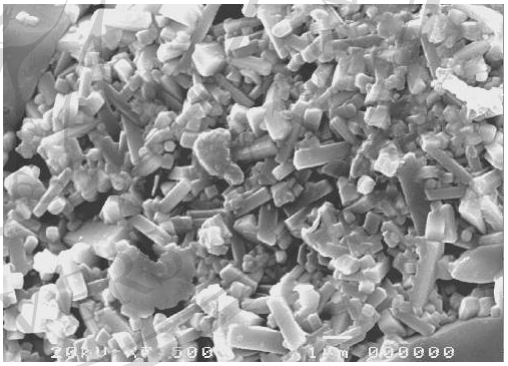


(a)

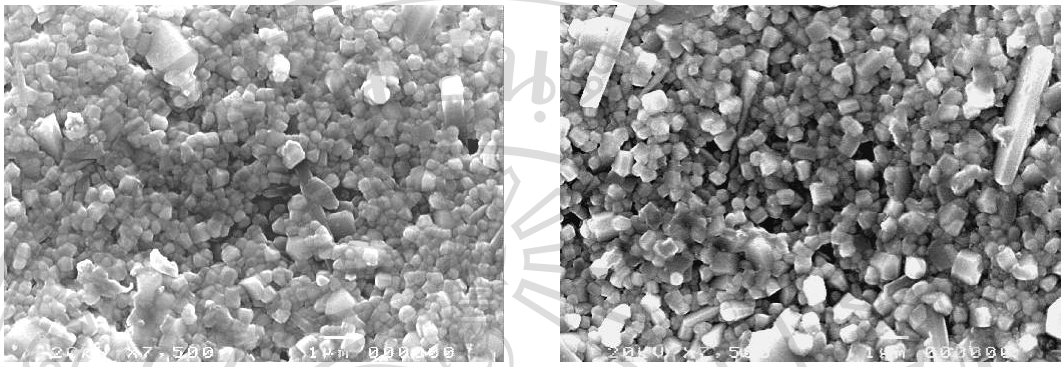


(b)

(c)



(d)



(e) (f)

Figure 3.23 SEM micrographs of sintered BLNT ceramics obtained from hydrothermal process with sintering temperatures of 1000 °C for 3 h and different mole %La of (a) 1 %La, (b) 2%La, (c) 3 %La, (d) 4 %La (e) 5 %La and (f) 6 % La.

Figure 3.23 shows SEM micrographs of BLNT ceramics containing 1-6 mole % lanthanum after cold pressing process and sintering at 1000 ^{o}C for 3 hours. Figure 3.23 (a, b, e and f) show the average grain size of 0.60 μm, however, the pores were observed inside the structure. For Figure 3.23 (c, d), SEM micrographs show the mixed small particle and platelets like structure form together. The average small irregular particle size was 0.40 μm and the longitudinal of platelets like structure was 5.0 μm.