# The Characterization of Natural Zeolite For Water Filtration System

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#### Abstract

The characterization of Indonesian's natural zeolite for water purification using X-Ray Diffractometer has been done. The objective is to obtain a general guidance for development of natural zeolite in molecular sieve, ion exchange and catalyst applications. It was found that the XRD spectra show two zeolite types. The zeolites originated from Lampung (ZL) was belong to clinoptilolite group and that originated from Banten (ZB) was belong to mordenite group. The crystal system for ZL was monoclinic with end-centered lattice and space group of Cm/2 (12). The crystal system for ZB was orthorhombic with end-centered lattice and space group of CmC21 (36). The character of both zeolites were confirm by ICDD standard library. It was also found that the particle size of zeolites did not affect the XRD spectra where zeolite of 40-50 mesh was similar to that XRD spectra of zeolite of 170-200 mesh. The chemical analyses show that both zeolites contain almost similar chemical elements. The contents of Fe, Ca, and K were found higher in ZL as it is compared to ZB. Reciprocally, Na was found higher in ZB. The water filtration ability shows that ZB was better than ZL for filtering of Pb and Fe elements. These ability were improved by chemical activation of zeolites. These two zeolites, howover, behave similar less ability for the filtering of Ca and Mg elements. These characterization is necessary to designing a further development of natural zeolite applications.

Keywords: natural zeolite, clinoptilolite, mordenite, x-ray diffraction, water filter

#### Abstrak

Telah dilakukan karakterisasi zeolit alam Indonesia untuk pembuatan sistem penjernihan air. Zeolit alam yang didapat dari Lampung (ZL) dan Banten (ZB) dikarakterisasi dengan X-Ray Diffractometer (XRD) dan diuji kemampuannya untuk penjernihan air. Tujuan penelitian ini adalah untuk mengembangkan pemanfaatan zeolit alam sebagai penyaring (molecular sieve), penukar ion maupun sebagai katalis. Pengukuran XRD menunjukkan bahwa zeolit alam yang berasal dari Lampung termasuk jenis klinoptilolit dengan sistem kristal monoklinik, jenis kisi end-centered, dan groupCm/2 (12). Sedangkan yang berasal dari Banten termasuk jenis mordenit dengan sistem kristal orthorhombic, jenis kisi end-centered, dan group Cmc21 (36). Dari penelitian in,i diketahui juga bahwa ukuran zeolit alam tidak mempengaruhi bentuk pola spektrum XRD. Analisa kimia menunjukkan bahwa kedua jenis zeolit tersebut mengandung unsur kimia yang sama. Kadar unsur Fe, Ca, dan K didapatkan lebih tinggi terkandung dalam ZL. Sebaliknya kadar unsur Na lebih tinggi dalam ZB. Kemampuan penyaringan Pb dan Fe didapatkan lebih baik pada ZB. Kemampuan ini dapat ditingkatkan bila zeolit terlebih dahulu diaktivasi secara kimia. Namun demikian baik ZL maupun ZB mempunyai kemampuan penyaringan Ca dan Mg yang sangat rendah. Karakteristik ini sangat berguna untuk perancangan dan pengembangan aplikasi ZL dan ZB ke depan.

Kata kunci: zeolit alam, klinoptilolit, mordenit, difraksi sinar-x, filter air

#### 1. INTRODUCTION

Indonesia is carrying out different research work in order to improve the natural resources utilization. It was understood that the higher utilization of natural resources make higher contribution to natural development. In this work, natural zeolites which are abundance available in Indonesian wish to be developed to achieve a more added value materials such as water purification/filtration system.

In fact, the natural zeolites have been used in agriculture as soil conditioner, as component of fertilizer, animal feed as well as a neutralization agent for bad smell waste [1,2]. The applications of zeolite in engineering are still very few. Some research were done to develop zeolites for its application as water filtration or as membrane system [3,4,5,6].

According to various researches, zeolite mainly a synthetic ores may have a great potential as membrane system such as micro filtration (MF), ultra filtration (UF), ad reverse osmosis (RO) as well as a powerful catalyst, ion exchanger, and gas separation systems [7,8,9,10,11,12,13].

In fact, however the researches for the utilization of natural zeolites are still low due to some reasons. Among of that reasons were the variety of character of natural zeolites. Two sources of natural zeolite, i.e. originated from Lampung (ZL) and originated from Bayah, Banten (ZB) are used to be characterization in the present work. The characterizations of two zeolites were carried out using x-ray diffractometer (XRD) instrument.

The objectives of this work is to obtain a general orientation of zeolite's crystal system, its properties as well as to determine a selective elements that probably affecting in the character of natural zeolite for its application as water filtration system.

## 2. MATERIALS AND METHOD

#### **Materials**

Natural zeolite were supplied by PT Minamata Mineral Perdana, i.e. originated from Lampung (ZL), and by volunteer zeolite mining agency, i.e. originated from Bayah, Banten (ZB). The zeolites were treated by ball milling and sieved to obtain a homogeneous powder size of 40-200 mesh, before used. Some necessary chemicals were used as a zeolite binder or as chemical activation agent.

#### **Instruments**

Shimadzu X-Ray Diffractometer instrument type 7000 made in Japan was used to

characterization of crystal system. The instrument was first calibrating using silicon standard.

Perkin Elmer Atomic Absorption
Spectrometer (AAS) type AAnalyst 700, made
in USA was used to analyze chemical elements.
Other equipments such Memmert Oven
(Germany) and Vulcon Kiln type 550 (Germany)
were used for drying and sintering work. Various,
tools, beaker glass and plastic equipments were
used as necessary.

## **Zeolite Activation**

The chemical activation of zeolite were done as follows:

50 gr of zeolites was kept in HCl 2M solution for one hour, washed and decanted by aquadest until neutral (pH=7.0). It was, they kept in NaNO<sub>3</sub> 2M solution for 1 hour, washed and decanted adequately by aquadest until neutral, and then dried in oven at 105°C until a constant weight.

#### XRD Characterization

Natural zeolite powder sample was put into a sample holder appropriately, kept homogeneous surface and placed it at measurement position. The measurement was done according to computer operating system and the obtained XRD spectrum was recorded. It took about 45 minute for each sample measurement. The obtained XRD spectrum was evaluated by standard diffraction data of x-ray from ICDD (International Centre for Diffraction Data).

#### **AAS Measurement**

0.5 gr of zeolite sample was destructed by aqua regia (HCl + HNO<sub>3</sub>), diluted to 100 ml by distilled water and arrange the concentration to be appropriate for AAS measurement. The measurement was done by flame detector technique and the results were computerized recorded and print-out as necessary.

# Filter Preparation

The zeolite sample was poured into a glass column (dia.3cm) for 5 cm thickness for media zeolite bed. The zeolite sample may be 40-200 mesh zeolite powder or a chemical

activation zeolite. The media zeolite bed ability in filtering of different solution of Pb acetate (0.2 M), FeCl<sub>3</sub> (0.1 M), MgSO<sub>4</sub> (1.0 M), and CaCl<sub>2</sub> (0.2 M) were observed. The filtrate was measured by AAS after a necessary dilution. The contents of Pb, Fe, Mg, and Ca either in filtrate and remain in media zeolite bed was evaluated. The filtering capability of natural zeolite that was prepared by a binder was also investigated.

### 3. RESULTS AND DISCUSSION

### XRD of Lampung Zeolite (ZL)

Figure 1 shows the XRD Spectrum of Lampung zeolite. As can be seen in Fig.1, there are 3 strongest peaks, followed by another 3 weaker peaks. The position of the six peaks can be summarized in Fig. 2, and it details can be seen in Table 1.

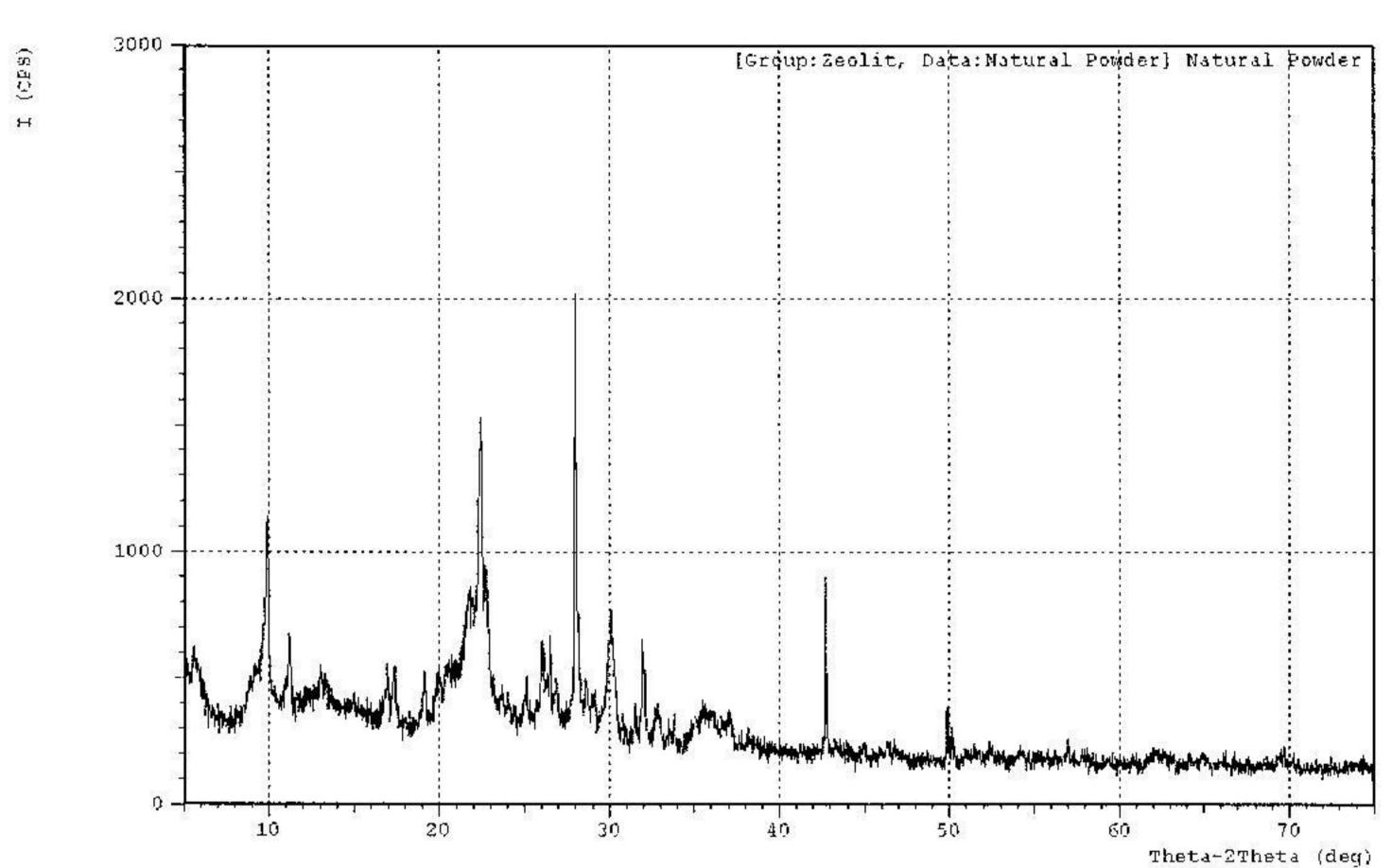


Fig.1. XRD Spectrum of Lampung Zeolit.

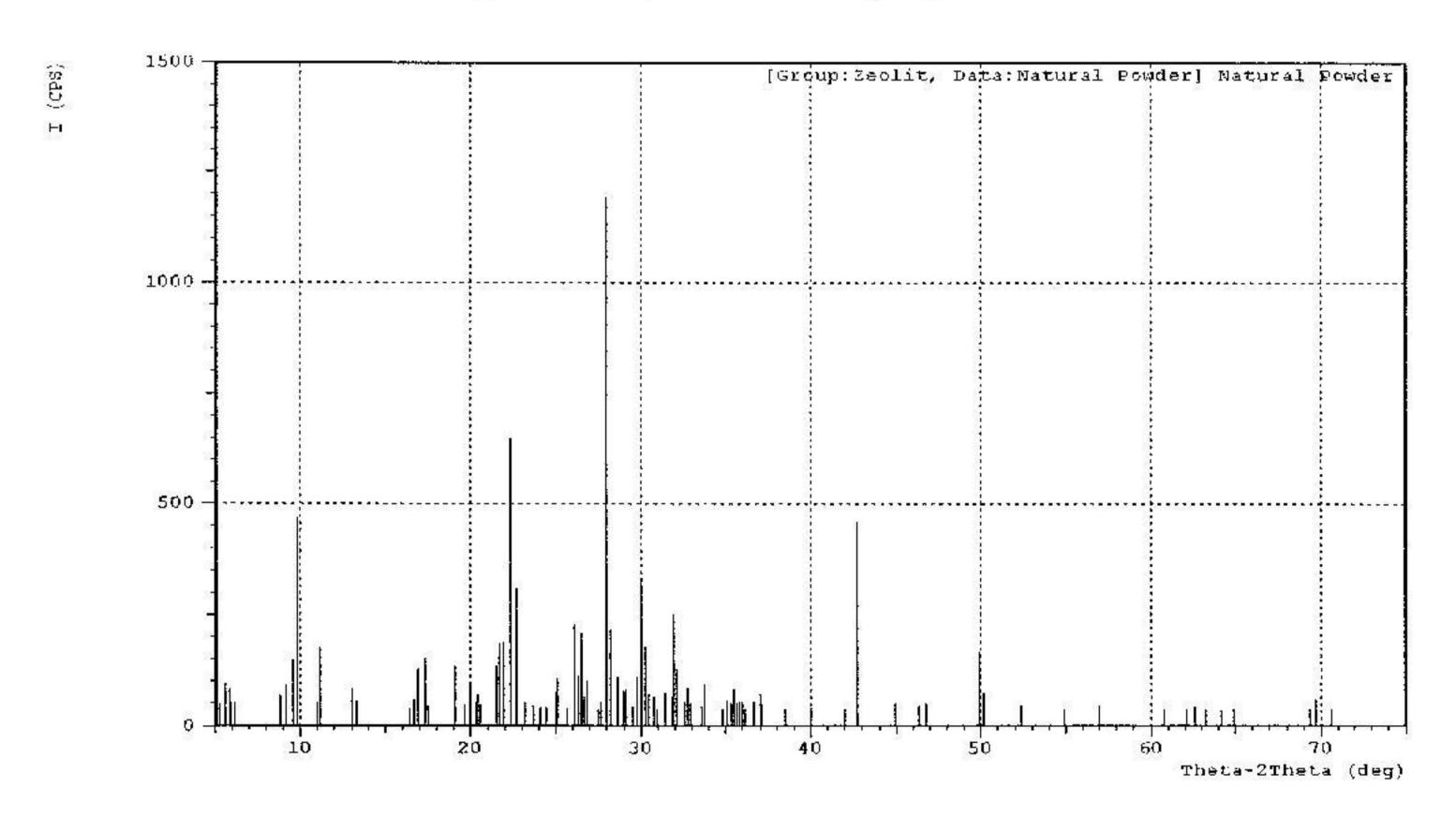


Fig.2. XRD Spectrum Line of Lampung Zeolit

Tabel 1. The Strongest Peaks of XRD Spectrum of Lampung Zeolite (Powder)

\*\*\* Basic Data Process \*\*\*

Group : Zeolit

Data : Natural Powder

# Strongeno. per no. p	ak 5. 5	peaks 2Theta (deg) 27.9583 22.3963 9.8631	d (A) 3.18874 3.96647 8.96057	1/I1 100 54 39	FWHM (deg) 0.10640 0.29840 0.21860	Intensity (Counts) 715 388 282	Integrated (Counts) 4385 6224 3110	Int	
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According to ICDD 47-1870 data library, the existence of the strongest peaks may indicate that the mineral was appropriate with potassium sodium calcium aluminum silicate hydrate of clinoptilolite-Na.

Based on Bravais classification crystal system, it was found to be a base-centered monoclinic, with a lattice of end-centered and the space group of C2/m (12). Accordingly, there are three unequal axes, one pair not at right angle,  $a \neq b \neq c$ ,  $\alpha = \gamma = 90^{\circ}$  and  $\beta \neq 90^{\circ}$  [14]. The crystal system can be illustrated as the following figure (See Fig.3).

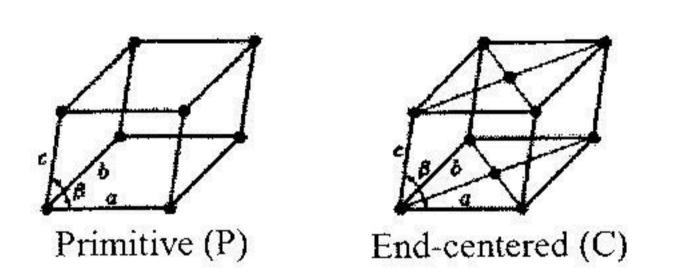


Fig.3. Bravais lattice for monoclinic crystal system.

The data indicate that the crystal structure should belong to a clinoptilolite, its density of 2.16 g/mol and a unit cell volume of 2100Å. These agreed to description that was reported by Thamzil Las (Ph.D. Thesis) [15]. The unit cell may have a dimensions of a=7.41 Å, b=17.89 Å, and c=15.85 Å. If the data are compared to that ICDD library, then it seems to be the same. In order word, the ZL could be predicted behave a crystal structure belong clinoptilolite group. The chemical formula of clinoptilolite is assumed to be Na<sub>6</sub> [(AlO<sub>2</sub>)6 (SiO<sub>2</sub>)<sub>30</sub>] 24 H<sub>2</sub>O.

In ICDD 47-1870 data, the clinoptilolite-Na may contains of [Na,K,Ca]<sub>5</sub> Ål<sub>6</sub> Si<sub>30</sub>O<sub>72</sub>.18H<sub>2</sub>O as can be seen in Table 2.

Tabel 2. ICDD Data for 47-1870

ICDD 47-1870

Mineral : C

: Clinoptilolite-Na

Potassium Sodium Calcium Aluminum Silicate Hydrate

Chemical: [Na, K, Ca]<sub>5</sub> Al<sub>6</sub> Si<sub>30</sub> O<sub>72</sub>. 18 H<sub>2</sub>O

Crystal System	Monoclinic	Space Group	C2/m(12)	a	17,647	α	90
Lattice	End-centered	Density	2.046	b	18,007	β	116.3
Lambda	1,5406	Pattern	I	c	7,396	γ	90

It can be concluded therefore, that the observed zeolite (ZL) should be a clinoptilolite with a monoclinic crystal system.

Further XRD measurement of ZL with

different mesh has shown the same pattern of XRD spectrum. The strongest peaks was given at 2θ of 28.0520° followed by the second strongest at 2θ of 22.3963°. (See Table 3)

Tabel 3. The Strongest Peaks of XRD Spectrum of Lampung Zeolite (60-100 mesh)

Group : Zeolit
Data : 60\_100 Mesh

# 5	Strongest	3	peaks
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no.	peak	2Theta	d	I/I1	FWHM	Intensity	Integrated I	nt
	no.	(deg)	(A)		(deg)	(Counts)	(Counts)	
1	55	28.0520	3.17830	100	0.10620	677	4102	
2	39	22.3934	3.96698	35	0.32680	234	3638	
3	38	22.0178	4.03380	29	0.15410	197	1279	

From Table 3, the peaks of XRD spectrum at 20 of 28.0520° and 22.3963° may be estimated as peaks character for natural zeolite of ZL. In fact, the XRD spectrum of ZL do not affected by the particle size, since the two XRD spectrum of ZL show the similar pattern.

### XRD of Activated Lampung Zeolite (ZL)

Fig. 4 shows the XRD spectrum of activated zeolite ZL. As can be seen in Fig. 4, there are three other strong peaks i.e. at  $2\theta$  of  $42.2126^{\circ}$ ,  $31.4545^{\circ}$ , and  $51.4774^{\circ}$  beside of three peaks that were assumed to be

characterization's peaks of clinoptilolite. Actually, the three other strong peaks have also available in XRD spectrum pattern of previously ZL, but in the form of weaker peaks.

Perhaps a higher peak at 20 of 42.2126° as its compared to that previously ZL was caused by sodium cation that coming from activation process. If it's true, it can be predicted that activated ZL may containing a single cation of Na. Therefore, the activation of zeolite can be favorable for molecular siever as well as for cation exchanger.

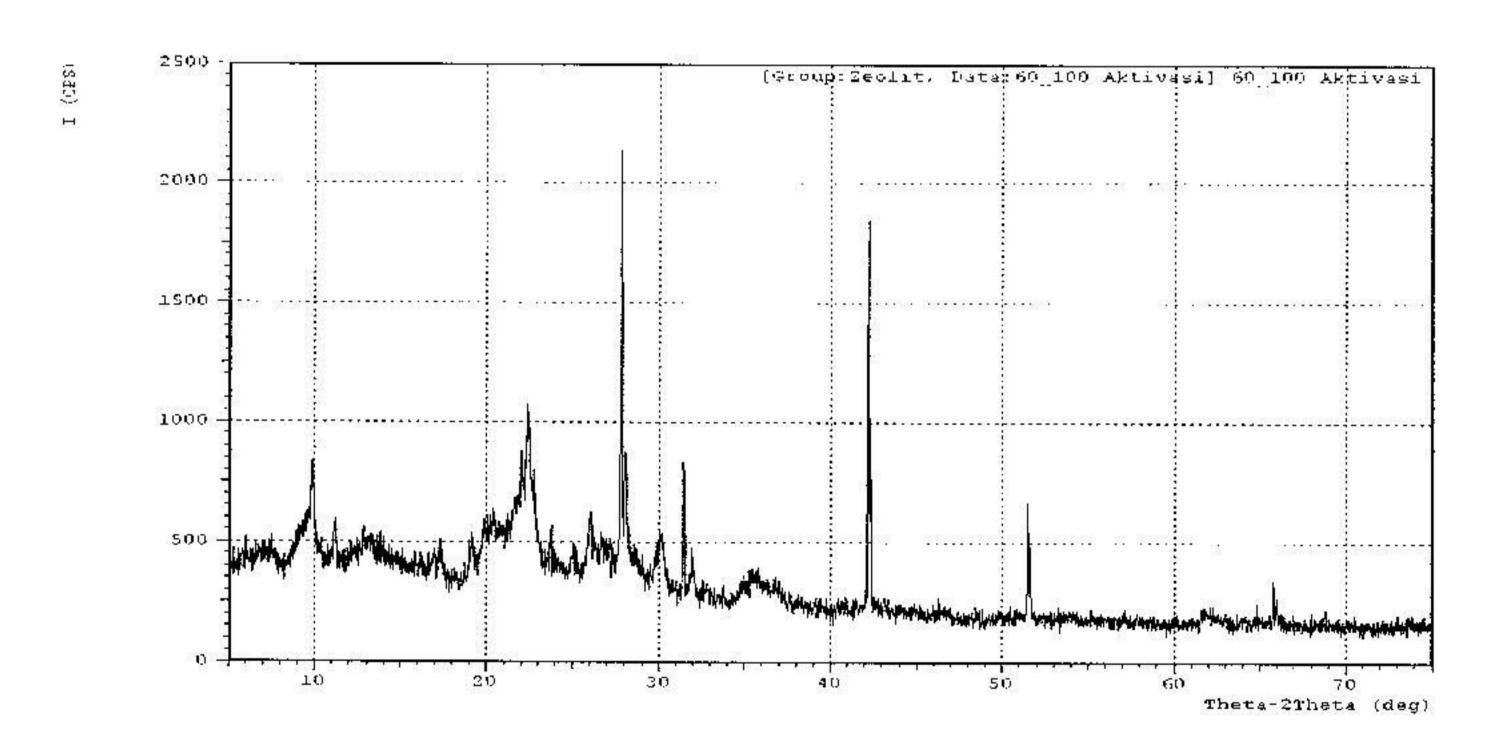


Fig.4. XRD spectrum of activated Lampung zeolit.

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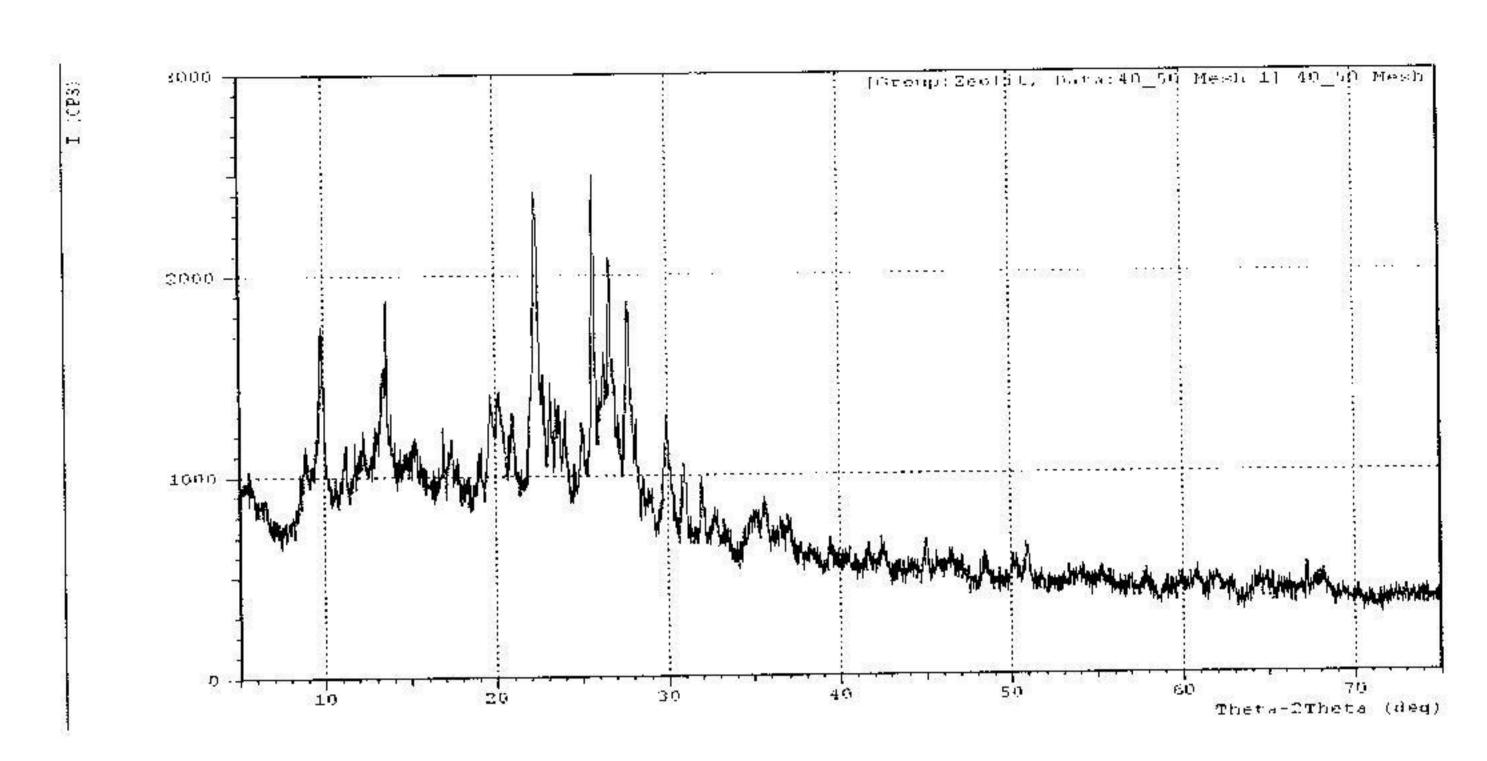


Fig.5. XRD Spectrum of Bayah, Banten Zeolit (40-50 mesh)

Fig. 5 shows the XRD spectrum of Bayah, Banten zeolite (ZB) of particle size of 40-50 mesh. As can be seen in Fig.5, there are three strongest peaks available i.e. peak at 2θ of 25.6897° (100), 27.3619° (95), and 26.6704°

(77). Other strong peaks were shown at 2θ of 27.7185° (68), 9.8023° (62), and 13.6000° (51). All the peaks may be assumed as a characteristic XRD spectrum of ZB. Detail data of XRD spectrum of ZB are presented in Table 4.

Tabel 4. The Strongest Peaks of XRD Spectrum of Bayah, Banten Zeolite (40-50 mesh)

-	* Basic Dat	a Process	* * *			
Group Date	Zeolit 40_50 Mesh					
# Strongest no. peak no. 1 46 2 39 3 49	3 peaks 2Theta (deg) 25.6897 22.3619 26.6784	d (A) 3.46495 3.97250 3.33874	1/11 100 95 77	FWHM (deg) 0.24540 0.41990 0.37920	Intensity (Counts) 598 567 462	Integrated Int (Counts) 9606 10712 9189
# Peak - 1234567890123444444444444444444444444444444444444	Lister (	d) 7753384481536036838797215228848822894630079514589 67731338481536036838797215228848822894630079514589 68,173893848153603683879721522888488228894630079514589 68,173893848153801819999999999999999999999999999999999	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	FWHM (0.24000 0000 0000 0000 0000 0000 0000 00	Y)  en  en  100  113  147827210177051388467993322627598072  en  110  113  1 1231  1 231  1 151111 152244  15224  15224  15224  15224  15224  15224  15224  15224	Integrated Integrates) 455 47183 4216901904 5602 3 900 4 56000000000000000000000000000000000000

peak	2Theta	d	1/11	FWHM	Intensity	Integrated Int
no.	(deg)	(A)	1/11	(deg)	(Counts)	Integrated Int (Counts)
50	26.9000	3.31174	37	0.00000	223	0
51	27.1400	3.28300	26	0.29660	158	3356
52	27.7185	3.21578	68	0.39370	407	7308
53	28.1200	3.17077	25	0.29840	150	2474
54	28.5771	3.12108	11	0.21580	67	699
55	28.9218	3.08466	7	0.36360	44	740
56 53	29.5800	3.01751	4	0.12000	23	212
57 <b>58</b>	29.9533 <b>30.360</b> 0	2.98074	33	0.36670	199	3591
5 <b>9</b>	30.3000	2.94173 2.88907	22	0.16000 0.27580	43 131	585 1869
60	31.9783	2.79646	19	0.23420	113	1387
61	32.3622	2.76416	_3	0.07560	20	93
62	32.7636	2.73120	9	0.31270	54	960
63	33.2928	2.68899	4	0.34570	25	469
64	34.4414	2.60190	6	0.15710	33	298
65	34.7200	2.58165	11	0.16000	63	1343
66	35.0600	2.55739	13	0.00000	75	0
67	35.2600	2.54334	10	0.00000	59	О
68	35.6290	2.51784	18	0.33800	106	2145
69 70	36_1000	2.48607	6	0.26800	38	499
70 71	36.5800 37.0000	2.45454	10	0.52000	61	1272
72	37.6340	2.42764 2.38818	11	0.28660 0.172 <b>00</b>	64 21	972 231
73	38.2353	2.35200	<b>4</b> 3	0.26070	18	290
74	39.4493	2.28237	6	0.20530	37	427
75	40.3950	2.23109	5	0.29000	27	469
76	41.6551	2.16646	8	0.20170	46	592
77	41.9512	2.15185	5	0.13750	27	207
78	42.2400	2.13781	4	0.13600	26	180
79	42.4650	2.12700	9	0.31000	51	650
80	42.7200	2.11489	4	0.12000	22	157
81	44.0766	2.05289	4	0.27330	21	411
82 83	<b>44.9573</b> 45.6725	2.01470	10	0.29740	58	947
84	45.9000	$1.98480 \\ 1.97549$	5 5	0.21500	30	357
85	46.2000	1.96336	5	0.26000 0.22 <b>660</b>	27 32	281 2 <b>90</b>
86	46.4200	1.95457	8	0.42400	48	950
87	46.8000	1.93958	5	0.00000	29	0
88	47.0483	1.92992	6	0.20330	36	595
89	48.4315	1.87798	9	0.27190	55	881
90	50.0600	1.82064	8	0.21140	50	530
91	50.3200	1.81184	б	0.21500	38	471
92	50.8984	1.79260	12	0.25190	74	1148
93	53.4125	1-71400	5	0.20500	32	414
94 95	53.8000	1.70257	3 4	0.32000	19	374
96	54.0000 54.1950	1.69673 $1.69109$	6	0.00000	23	0
97	55.2571	1.66107	6	0.23000 0.23 <b>43</b> 0	34 36	528 643
98	55.6566	1.65009	4	0.23330	23	347
99	56.9650	1.61526	5	0.17000	28	311
100	57.8200	1.59339	6	0.22000	37	536
101	59.8000	1.54527	5	0.28000	31	461
102	59.9800	1.54107	4	0.28000	25	369
103	60.2400	1.53503	3	0.0000	20	О
104	60.5200	1.52860	4	0.00000	26	O
105	60.7800	1.52268	8	0.36000	47	631
106 107	60.9400	1.51907	5 3	0.29720	32	325
107	61.3672 61.6318	1.50951	3	0.08780	18	101
109	61.9400	1.50367 1.49692	8	0.20640 0.28000	44	422
110	62.2800	1.48956	4	0.28000	50 24	825
111	62.5200	1.48442	4	0.00000	21	0
SMC35WED (File/A)			978 <del>-11</del>		(عاف المشواء	<b>→</b>

The measurement of XRD spectrum of ZB for different particle size of 170-200 mesh results the same XRD spectrum pattern. The XRD spectrum of ZB which has particle size of 170-200 mesh shows three strongest peaks at 2θ of 25.780° (100), 22.382° (78), and 9.761° (54) and

these similar to that XRD spectrum of other particle size of ZB. Therefore, the particle size of zeolite may concluded do not affect the pattern of XRD spectrum. Table 5 shows ICDD data for 49-0924

Tabel 5. ICDD Data for 49-0924

ICDD 49-0924

Mineral : Mordenite, syn

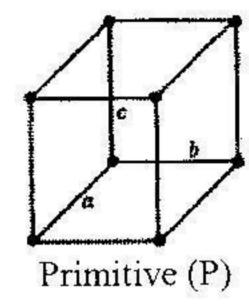
Sodium Aluminum Silicate – Zeolite Al-modenite

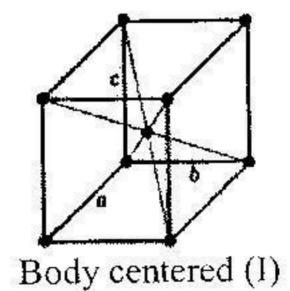
Chemical :  $Na_2 Al_2 Si_{13.3} O_{29.6} + x$ 

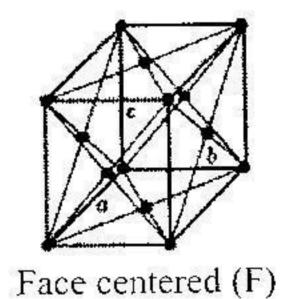
Crystal System	Orthorhombic	Space Group	Cmc21 (36)	a	18,067	α	90
Lattice	End-centered	Density	_	b	20,284	β	90
Lambda	1,5406	Pattern	I	c	7,491	_ γ	90

If the XRD peaks of ZB is compared to that ICDD 49-0924, then it can assumed that ZB belong to be a mineral mordenite group. The crystal system should be orthorhombic, lattice of end-centered, and space group of Cmc21 (36). In this case, there are 4 possible Bravais lattice of orthorhombic crystal system i.e. simple or

primitive (P), body centered (I), end centered (C), and face centered (F) as shown in Fig. 6 [14]. There are also exist three unequal axis at right angles,  $a \neq b \neq c$ ,  $\alpha = \beta = \gamma = 90^{\circ}$ .







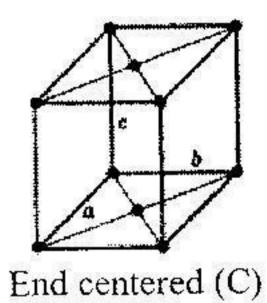


Fig.6. Bravais lattice of orthorhombic.

Supandi [2] reports that crystal structure of Bayah zeolite may contains two crystal systems, there are crystal monoclinic with space group C2/m and crystal orthorhombic with space group of CmCm. Supandi conclusion based on a calculation by a refinement Rietan method.

Perhaps, the conclusion was correct, because the XRD spectrum of our sample ZB didn't merely follows the ICDD orthorhombic crystal system. Therefore, it can be assumed that our ZB sample may comprises a mixture of mineral mordenite and clinoptilolite.

# Chemical Analysis

Table 6 show some metal oxide that containing in ZL and ZB as it was compared to others results.

Table 6. Metal Oxide in Zeolite

55.55 48 8.55 0	Percentage of Metal Oxide (%)							
Metal Oxide	Curren	t Result	*PT Minamata	*Supandi				
	ZL	t Result *PT Minam	Clinoptilolite	Mordenite				
Fe <sub>2</sub> O <sub>3</sub>	0,94	0,80	1,29	1,46				
Na <sub>2</sub> O	0,63	0,98	0,75	0,20				
K <sub>2</sub> O	0,70	0,67	1,54	2,81				
CaO	0,52	0,42	1,31	2,36				

<sup>\*</sup>The comparison is assumed that the zeolite comes from the same area, but not exactly the same sample.

As can be seen in Table 6, the metal oxides that containing in ZL were almost nearly similar that containing in ZB. But it was not in case of the results that reported by others. The different may caused by the different of analysis method or different sample itself.

The result of Croatian zeolite of clinoptilolite was also different [16]. Therefore, the different origin of zeolite may provide different characteristics and chemical containment. For the reason, the characterization of natural zeolite became very important, before it's planned to be utilized for specific purpose.

#### **Zeolite Filter Performance**

Table 7 shows the zeolite filter performance. It can be seen in Table 7, that

activated ZL was found to be the best for filtering heavy metal of Pb. In this case, original solutions that contain 19.07 gr could be reduced to be 8.86 (almost 50%). On the other hand, ZL and ZB could be able to reduce only smaller i.e. it is reduces to be 15.33 and 10.33, respectively. In case of Fe, ZB shows the best as compared to that ZL and activated ZL i.e. it can reducing Fe from 0.49 gr to 0.18 gr, whereas ZL and activated ZL could reduce only 0.43 gr and 0.26 gr, respectively. All kind of zeolite (ZL, ZB, and activated ZL) was found to be worse to reduce Mg and Ca. Even more Ca became higher in case of ZL and ZB performance. The results indicate that activated zeolite became very important to design an appropriate application of natural zeolite.

Table 7. Zeolit Filter Performance

	Heavy Metal Contents (gram)									
Filter	Pb		Fe		Mg		Ca			
	X	Y	X	Y	X	Y	X	Y		
Zeolit Lampung	19,07	15,33	0,49	0,43	0,28	0,24	0,94	1,02		
Zeolit Banten	19,07	10,33	0,49	0,18	0,28	0,21	0,94	0,97		
Activated ZL	19,07	8,86	0,49	0,26	0,28	0,22	0,94	0,84		

Caution: X = before filtering, Y = after filtering.

## 4. CONCLUSION

Two types of natural zeolites i.e. originally from Lampung (ZL) and Bayah, Banten (ZB) have been characterized by XRD method. It may be concluded that ZL was belong to clinoptilolite mineral with monoclinic crystal structure and ZB was belong to mordenite mineral with orthorhombic crystal structure.

The ZB however may have a mixture composition of mordenite and clinoptilolite mineral. The chemical activation of natural zeolite was important to improve the performance of zeolite.

Different origin and different sample of zeolite provide different zeolite character. Therefore the caharacterization of natural zeolite became very important for further design application of zeolite.

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