



Innovative Synthesis of Lynde Type-A (LTA) Zeolite: A Comparative Study of Microwave-Assisted Hydrothermal Route

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Abstract. Lynde Type-A (LTA) zeolite is one type of zeolite that has various uses including as an adsorbent, catalyst, ion exchanger, and molecular sieve. Generally, LTA zeolites are produced from commercial chemicals and natural materials such as natural zeolites using the hydrothermal method which takes up to 24 hours to synthesize. The use of the hydrothermal method in the synthesis process is proven to produce zeolites with good quality but takes a long synthesis time, therefore it is necessary to innovate by comparing the hydrothermal synthesis method and the Microwave-assisted Hydrothermal Synthetic Route through various tests. The use of microwave as a synthesis method was carried out with a power of 200 watts and variations in synthesis time of 10, 20 and 30 minutes. After being analyzed using XRF (X-ray Fluorescence) the synthesis product has a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 1.69 then through FTIR (Fourier Transmission Infra Red) testing found the characteristic of LTA zeolite in the form of double rings at wave numbers $550\text{-}600\text{ cm}^{-1}$, through XRD (X-Ray Diffraction) testing obtained the highest percent relative crystallinity of LTA of 83,61%, through BET analysis (Brunaur, Emmet And Teller) found the highest surface area of $11.055\text{ m}^2/\text{gr}$ in the 30-minute synthesis product, and through SEM analysis (Scanning Electron Microscopy) displayed zeolites in the form of rhombic crystals similar to previous LTA zeolites.

Keywords: Lynde Type-A Zeolite, Zeolite Synthesis, Hydrothermal Method, Microwave-assisted synthesis.

1 Introduction

LTA zeolite is one type of synthetic zeolite that is widely applied as an adsorbent in the industrial sector. To synthesize LTA zeolites, materials containing silica and alumina are needed, which can be found in organic materials such as bagasse, rice husks, coal fly ash, coal bottom ash and natural zeolites [1-4]. Previously, Lampung natural zeolite has been successfully used as raw material for the synthesis of LTA zeolite by step

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change temperature of hydrothermal method using water bath [5]. The synthetic process with the hydrothermal method produces LTA zeolite with a relative crystallinity percent of 71.39%, a surface area of 15.7 m²/g and a total pore volume of 0.048 cc/g [5]. To achieve these results requires a synthesis time of 4 hours, therefore it is necessary to make innovations that can shorten the synthesis time, one of which is by changing the synthesis method used.

The most widely used synthesis method besides the hydrothermal method is the microwave radiation method. Various studies have reported the use of microwave in the synthesis process is able to shorten the synthesis time because the heat is generated by the sample molecules that interact with each other, in contrast to the hydrothermal method which uses an oven or water bath that conducts heat through an intermediary to the destination sample. The use of microwave is able to reduce the activation energy of the reaction so as to make the reaction run faster [7]. In [4] successfully synthesized LTA zeolite with coal fly ash using microwave with power variations of 100, 200 and 300 watts for 10-30 minutes then obtained a percent crystallinity of zeolite of 67.24% with the use of 300 watts of power for 30 minutes. Previously, similar experiments were also carried out by [8] using commercial silica and alumina with microwave power variations ranging from 180-900 watts. Zeolite NaA crystallinity of 99% was achieved by using 900 watts of power for 10 minutes followed by 360 watts of power for 50 minutes. Based on this, research is needed to compare the hydrothermal method and microwave irradiation in the synthesis of LTA zeolite using Lampung natural zeolite. The comparison is based on surface area, relative LTA criticality and zeolite morphology.

2 Material and Methods

The raw materials used in this study were Lampung Natural Zeolite (LNZ) from CV. Minatama Bandar Lampung with SiO₂ /Al₂O₃ content of 5.51% and supporting materials such as NaOH (pro analysis UNI-CHEM CNA 43/500), Al₂O₃ (pro analysis UNI-CHEM CAS 169/1000) and distilled water from the Laboratory of Agricultural Product Technology, University of Lampung. The tools used in this research are mortar, 200 mesh sieve, thermometer, desiccator, digital balance, PTFE reactor with a volume of 300 cm³ (65 mm ID and 90 mm height) and microwave

2.1 Research procedure.

The research procedures used include:

1. LNZ Pretreatment

A certain amount of LNZ was pulverized with a mortar until smooth and continued with sieving using a 200 mesh sieve to obtain a uniform zeolite size.

2. Preparation of Sodium Hydroxide (NaOH) Solution

Sodium hydroxide solution is made by dissolving 10 grams of NaOH into 182.28 grams of H₂O which is then divided into two solutions of equal volume (solution A and solution B).

3. Preparation of Sodium Silica Solution

Sodium silica solution was prepared by dissolving 10.80 grams of LNZ into solution A, then heated at 60° C at atmospheric pressure with stirring for 1 hour.

4. Preparation of Sodium Alumina Solution

Sodium alumina solution was prepared by dissolving 6.79 grams of Al_2O_3 into solution B, then heated at 60°C at atmospheric pressure with stirring for 1 hour.

5. Preparation of Alumina Silica Solution

Sodium silica solution and sodium alumina solution are mixed until homogeneous into silica alumina solution by stirring under environmental conditions.

6. Ageing Zeolite

The silica alumina solution was then aged for 45 hours at room temperature and atmospheric pressure without stirring.

7. Zeolite Synthesis

The silica alumina solution was put into the PTFE reactor and heated in a microwave with a power of 200 watts at various synthesis times for 10, 20 and 30 minutes.

8. Synthesis Product Purification

The synthesis product was filtered with filter paper to separate the solid (zeolite) and the filtrate. The solid synthesis product was washed with distilled water until a pH of ± 7 was reached.

9. Drying of Synthesized Products

The synthesized solid was dried using an oven at 100° C for two hours. The solid was then stored in a desiccator to be characterized

2.2 Characterization of Synthesis Products

The characterization of the synthesis product is carried out to determine the character of the synthesis product carried out using a tool:

1. X-ray Fluorescence (XRF) Panalytical Epsilon 3
2. Fourier Transmission Infra Red (FTIR) Nicolet™ is 50 FTIR Spectrometer with NIR Module with 400-4000 cm^{-1} wave spectrum.
3. X-Ray Diffracton (XRD) X'pert Propanalytical.
4. BET (Brunaur, Emmet And Teller) Quantachrome Instrument.
5. Scanning Electron Microscopy (SEM) ZEISS Electron Microscopy using 5,000-50,000x magnification.

3 Results and Discussion

3.1 Fourier Transmission Infra Red (FTIR) Analysis

The FTIR spectra of the products synthesized for 10, 20 and 30 minutes show double rings vibrations at wave numbers 574.7; 579.5 and 578.5 cm^{-1} [6]. O-Si-O or O-Al-O asymmetry stretching vibrations at wave numbers 1008.6; 1005.7 and 1008.6 cm^{-1} [10]. O-H bending vibrations are found at wave numbers 1630.5; 1633.4 and 1633.4 cm^{-1} [11]. H-O-H vibrations were found at wave numbers 3432.7; 3417.2 and 3415.3 cm^{-1}

[9]. Then the vibrations of the Si-OH-Al group were found at wave numbers 3636.8; 3617.8 and 3616.8 cm^{-1} [6].

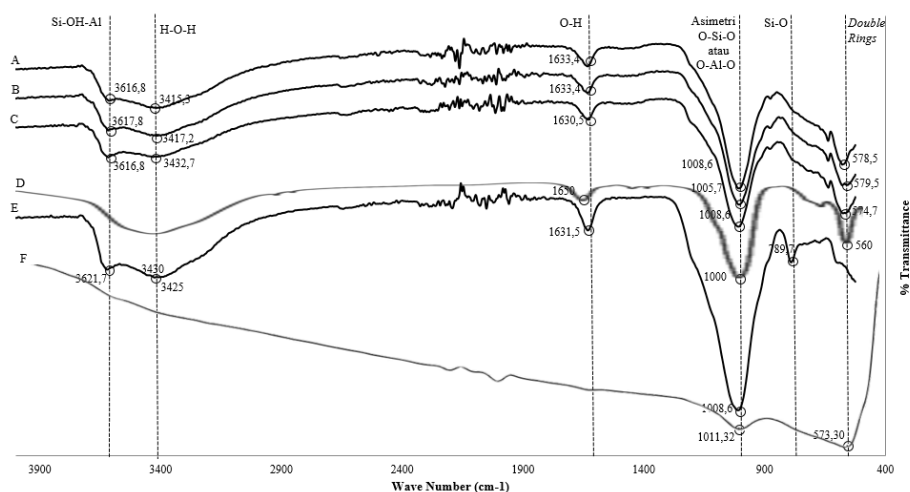


Fig. 1. FTIR spectral data of (A) 30 min synthesis product, (B) 20 min synthesis product, (C) 10 min synthesis product, (D) Zeolite LTA Jacas et al. [11], (E) LNZ and (F) Zeolite LTA [5]

Table 1. Area and Absorbance Intensity of LNZ and Synthesis Product (SP).

Category	Absorbance Area				Absorbance Intensity			
	LNZ	SP 10 minutes	SP20 minutes	SP 30 minutes	LNZ	SP 10 minutes	SP20 minutes	SP 30 minutes
Vibration Si-OH-Al	7.384	6.918	3.078	1.380	216	210	122	72
Vibration H-O-H	49.590	19.980	10.947	8.263	450	152	214	364
O-H Vibration	58.482	21.175	22.544	24.037	1028	612	637	665
O-Si-O or O-Al-O vibrations	1.294.290	344.598	316.350	403.073	6573	2852	2910	3347
Si-O vibrations	14.146	-	-	-	643	-	-	-
Double rings	-	14.272	24.864	30.573	-	393	593	712

In addition to showing the functional groups, FTIR testing also shows the area of the absorbance peak between the height of the vibrational peak and the vibrational baseline and the intensity of the functional groups of the % transmittance of each sample. The area of the adsorbance peak and the intensity of the functional group are proportional to the number of related functional groups [6,12]. In this case, the height and baseline of the vibration can be seen using the OriginPro application so that the area and intensity of the absorbance peak are obtained as in Table 1.

When the synthesized product is compared with LNZ, many differences are found such as the Si-O functional group which is only found in LNZ and double rings vibrations which are only found in the synthesized product. The Si-O group is only found in natural zeolites because in the synthesis process there is a stage of adding an alkaline solution which causes desilication or breaking of the Si-O bond from the zeolite framework [8]. Then double rings vibrations were only found in the synthesized product because double rings are characteristic of LTA zeolites [6,9,11,13]. However, LNZ and synthesized products still have similarities in several groups such as Si-OH-Al groups, H-O-H groups, O-H groups, and O-Si-O groups or O-Al-O groups. The Si-OH-Al, H-O-H, O-H, and O-Si-O or O-Al-O groups in Table 1 appear to have decreased after LNZ turned into a synthesis product, the decrease in H-O-H groups is due to the heating and drying process which evaporates some of the water in the synthesis product [12]. The decrease in O-H groups, O-Si-O groups and Si-OH-Al groups is due to the desilication process, where O-H will break the O-Si-O bond and form Si-OH which will eventually be removed in the process of purifying the synthesis product in order to reach pH ± 7 [8].

The synthesis products were made with variations in microwave radiation time ranging from 10, 20 and 30 minutes so that there are differences between one product and another. This difference can be seen in Table 1 where there is a decrease in the number of Si-OH-Al groups, H-O-H groups, O-H groups and an increase in the number of double rings during the addition of synthesis time. The decrease in H-O-H groups is a result of the increase in synthesis temperature as the synthesis time increases, this is in line with the research of Chen et al. [12] in the FTIR analysis of geological materials with temperature variations. In addition, the decrease in O-H groups occurs due to the desilication process that forms Si-OH-Al and will later be discarded in the purification process of the synthesis product, so that the longer the synthesis time the number of H-O-H, O-H and Si-OH-Al groups will be less. On the other hand, the addition of synthesis time can increase the number of *double rings* in the synthesis product, this happens because the addition of synthesis time increases the time of zeolite crystallization and makes the number of double rings continue to grow because double rings are the framework in LTA zeolite [6,9].

When all synthesis products are compared with LTA zeolites belonging to Jacas et al. [11] all samples appear to have similar vibrational patterns, this can be seen from the many vibrational similarities between LTA zeolites and three synthesis products such as the presence of O-H-O groups, H-O groups, O-Si-O or O-Al-O groups, and double rings vibrations, while when compared to LTA zeolites belonging to [5] the synthesized product appears to have a similar vibrational pattern in O-Si-O or O-Al-O group vibrations and double rings vibrations. Jacas et al's LTA zeolite [11] is an LTA zeolite synthesized from commercial chemicals by hydrothermal method using an oven and [5] LTA zeolite. [5] is LTA zeolite synthesized from natural zeolite Lampung by hydrothermal method using water bath. When the synthesis product has a vibrational pattern similar to the previous LTA zeolite, it can be confirmed that the synthesis product is an LTA zeolite [14].

3.2 X-Ray Diffraction (XRD) Analysis

XRD characterization of synthesis products and LNZ can provide information on the type of minerals and the relative crystallinity of zeolite constituent components. The type of mineral constituent of zeolite is shown by the peaks at post 2θ and the relative crystallinity level of the components is shown by the peak intensity displayed in Fig. 2. and Table 2. From Fig. 2., it can be seen that the gravic LNZ displays several clinoptilolite peaks at post 2θ 9.817; 12.99; 16.82; 18.97; 22.37; 25.92 and 32.75 in accordance with Treacy and Higgins [15] and other minerals such as corundum, tridymite and quartz found through the Match!3 application. Then the relative crystallinity of each component in Table 2 shows the relative crystallinity of LNZ is dominated by clinoptilolite minerals as much as 47.11% which indicates LNZ is a clinoptilolite zeolite and the relative crystallinity of various other minerals of 52.89% indicates impurities in the zeolite framework that can affect the properties and abilities of zeolites as adsorbents or catalysts [13].

Fig. 2. and Table 2 show the difference between LNZ and the synthesized product. LNZ has a relative crystallinity of 47.11% clinoptilolite and 52.89% various other minerals, while the synthesis product has a relative crystallinity of 83.61% LTA zeolite; 3.38% clinoptilolite and 13.05% other minerals. The decrease in the relative crystallinity of clinoptilolite and the increase in the relative crystallinity of LTA zeolite in the synthesis product are the result of a series of synthesis processes to convert LNZ into LTA zeolite and indicate the success of the synthesis process.

After being synthesized with variations in synthesis time of 10, 20 and 30 minutes, the synthesis product experienced an increase in the relative crystallinity of LTA and a decrease in the relative crystallinity of other minerals. The relative crystallinity of LTA increased from 63.28% for 10 minutes of synthesis; 67.51% for 20 minutes of synthesis; and 83.61% for 30 minutes of synthesis, this is in line with the research of [1] in their efforts to synthesize zeolite A from rice husk ash and the research of [4] on the synthesis of LTA zeolites from coal fly ash using microwave radiation. The increase in relative crystallinity during the addition of synthesis time shows that the synthesis time can affect the crystallinity of zeolite minerals because the synthesis time is proportional to the time of zeolite to crystallize or called the crystallization stage [4]. The crystallization stage is one of the stages of zeolite formation which starts from the primary amorphous phase or the process of mixing amorphous reactants containing ions forming the primary structure of zeolites (SiO_4 , PO_4 , AlO_4 , etc.) in acidic or alkaline media. Furthermore, the secondary amorphous phase or the cation structuring stage in solution forms the zeolite secondary structure unit, then the nucleation phase or the zeolite nucleus formation process and continued with the zeolite growth phase. The rate of nucleation and zeolite growth or crystallization in the system is referred to as a function of synthesis time [6].

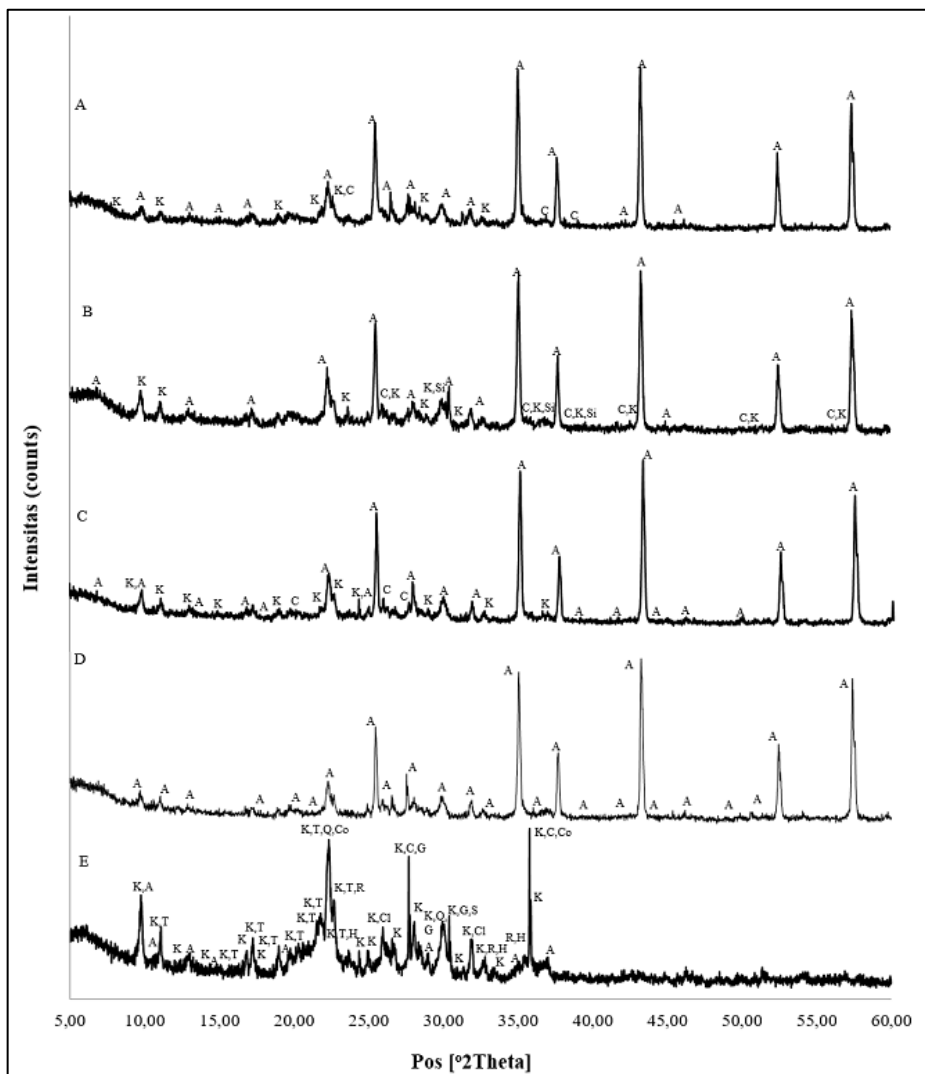


Fig. 2. XRD of (A) 30 min synthesis product, (B) 20 min synthesis product, (C) 10 min synthesis product, (D) Previous LTA zeolite synthesized by [5], and (E) LNZ.

Image caption :

A = Zeolite LTA

K = Clinoptilolite

T = Tridymite

C = Corundum

Q = Quartz

Si = Siderite

R = Ribidium

H = H₂O

Co = (CoZn)Mg

G = Germanium Antimony Telluride

S = Siderazot

Cl = Clausthalite

Table 2. Relative crystallinity of zeolite.

Minerals	Relative Crystallinity Percentage			
	LNZ	Synthesized Products 10 min	Synthesized Products 20 min	Synthesized Products 30 min
LTA	11,75%	63,28%	67,51%	83,61%
Klinoptilolit	47,11%	5,77%	5,29%	3,38%
Tridymite	13,12%	-	-	-
Corundum	8,13%	9,87%	9,15%	8,05%
Quartz	7,05%	-	-	-
Rubidium	3,53%	-	-	-
H ₂ O	2,45%	-	-	-
(CoZn)Mg	2,21%	-	-	-
Germanium	1,96%	-	-	-
Siderite	-	-	6,02%	-
Other Minerals	-	21,08%	12,03%	5,00%

Fig. 2. shows the graph of synthesis products (gravic A, B and C) which are LTA zeolites, this is evidenced by the appearance of typical LTA zeolite peaks at post 2θ which is in accordance with [15] and has a graphical shape match with previous LTA zeolites synthesized by [5]. This statement is reinforced by the relative crystallinity of the synthesized product which is dominated by LTA zeolite, where Table 2 shows that the synthesized product is dominated by LTA zeolite ranging from 63.28% to 83.61%. When compared with LTA zeolite belonging to Ginting et al. [5] with a relative crystallinity of 71.38%, the microwave-synthesized zeolite looks superior because with a synthesis time of 30 minutes it is able to achieve a relative LTA crystallinity of 83.61%. The relative crystallinity of zeolite synthesized by microwave is greater than the hydrothermal method because the microwave method uses microwave radiation to heat the reactants quickly and uniformly. This process can produce higher temperatures in a shorter time, accelerating the formation of zeolite crystals with larger and more regular sizes [16]. Then too long synthesis time also tends to reduce zeolite crystallinity so that zeolites synthesized by hydrothermal for 4 hours [5] tend to have lower crystallinity than zeolites synthesized by microwave for 30 minutes [17].

3.3 Scanning Electron Microscopy (SEM) Analysis

SEM was used to determine the surface morphology and particle size uniformity of the samples, in this case LNZ and the product synthesized for 30 minutes, and the results are shown in Fig. 4.

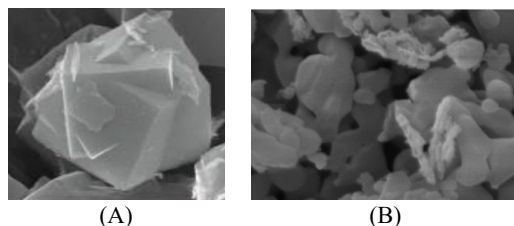


Fig. 5. Zeolite LTA crystals synthesized by (A) [20] and (B) [5]

From Fig. 6. it can be seen that the LTA zeolite has a varied shape, Fig. (A) is LTA zeolite synthesized from kaolin [20] while Fig. (B) is LTA zeolite synthesized from LNZ by hydrothermal method [5]. When juxtaposed with the product synthesized by microwave irradiation, Fig. 6. (A) has a similar shape, but when compared to Fig. 6. (B) both have a very different shape, Fig. 6. (B) shows zeolites that have the potential to undergo sintering while Fig. 4. (C) and 4 (D) are in the form of rhombic crystal aggregates. Fig. 4. or the synthesis product image shows zeolites with high crystallinity which proves the previous XRD testing, while image (B) shows lower crystallinity due to the long heating time in the LTA zeolite synthesis process [17].

3.4 Brunaur, Emmet And Teller (BET) Analysis

After BET analysis, the surface area of LNZ and synthesized products for each synthesis time is shown in Table 3.

Table 3. Comparison of Zeolite Surface Area.

Sample	Surface Area (m ² /g)
Lampung Natural Zeolite	3,525
Synthesized Product 10 Minutes	7,798
Synthesized Products 20 Minutes	8,556
Synthesized Products 30 Minutes	11,055

From Table 3, it can be seen that the surface area of the synthesis product is larger than LNZ because the synthesis process with microwave irradiation is able to clean the zeolite framework from organic and inorganic impurities so that the surface area is larger. This is in accordance with the research of [4] in the synthesis of zeolite A from coal fly ash with microwave irradiation where the surface area of zeolite A became 47.14 m²/g from the original coal fly ash surface area of 15.47 m²/g. Table 3 also displays an increase in the surface area of the synthesized product as the synthesis time increases, which is shown by the surface area of the synthesized product which reaches 11.055 m²/g with a 30-minute synthesis process. The length of synthesis time shows the length of time of microwave radiation which acts as a heater in the crystallization process of zeolite. The longer the zeolite is exposed to radiation means the longer the crystallization process takes place so that more LTA zeolite will be formed and the more zeolite pores are read as zeolite surface area.

3.5 X-ray Fluorencence (XRF) Analysis

XRF characterization was carried out on Lampung natural zeolite (LNZ) and 30-minute synthesis products to see the composition of zeolites before and after synthesis, the results are shown in Table 4 below.

Table 4. Elemental Composition of Zeolite.

Elements	% Mole Component	
	LNZ	Synthesized Product 30 Minutes
MgO	1,162	0,004
Al O ₂₃	12,788	31,618
SiO ₂	70,521	53,475
P O ₂₅	3,364	3,348
SO ₃	1,296	1,179
Cl	0,024	0,444
K O ₂	2,777	4,594
CaO	4,462	1,415
Ti	0,229	0,015
V	0,003	0,001
Mn	0,058	0,085
Fe O ₂₃	3,311	0,598
Co	0,008	0,194
Cu	0,001	0,002
Zn	0,007	0,012

Table 4 shows that there is a change in the number of constituent elements of zeolite before and after synthesizing. Before being synthesized LNZ was dominated by silica in the form of SiO₂ as much as 70.52%, other constituents of 16.69% and alumina in Al₂O₃ of 12.79%. After being synthesized for 30 minutes using microwave irradiation zeolite is dominated by silica as much as 53.48% then alumina by 31.62% and other elements by 14.90%. From this it can be seen that the synthesis process that occurs affects the constituent elements of zeolites where after being synthesized the amount of silica and other minerals in zeolites decreases so that the percentage of alumina in zeolites increases. This synthesis process makes the SiO₂/Al₂O₃ ratio of zeolites drop to 1.69 mol/mol which initially amounted to 5.51 mol/mol. Based on the value of the ratio of silica and alumina zeolites can be classified into zeolites with low silica content (SiO₂/Al₂O₃ = 1.0-2.0), medium silica content (SiO₂/Al₂O₃ = 2.0-5.0) and zeolites with high silica content (SiO₂/Al₂O₃ = 10-100) [20]. Zeolite made by microwave irradiation method this time is LTA zeolite which is included in the low silica content zeolite group and this is evidenced by XRF testing with a SiO₂/Al₂O₃ ratio of 1.69 mol/mol.

The process of synthesizing LTA zeolite has been widely done before, such as research conducted by [21] which produced LTA zeolites with SiO₂/Al₂O₃ mol/mol ratios of 1.34; 1.72 and 1.92 and research conducted by [14] which produced LTA zeolite with SiO₂/Al₂O₃ ratio of 1.62 mol/mol. When compared with the previous LTA zeolite [21,14] synthetic zeolite with SiO₂/Al₂O₃ ratio of 1.69 mol/mol in this study falls into the range of SiO₂/Al₂O₃ ratio of the previous LTA zeolite [21,14] so that it shows the

synthesis process of LTA zeolite using microwave irradiation has been successfully carried out with a power of 200 watts and synthesis time for 30 minutes.

3.6 Comparison of Hydrothermal and Microwave Irradiation Methods

Based on the results of analysis using FTIR, XRD, BET, XRF and SEM, it can be seen that the use of microwave is proven to be able to be used in the synthesis of LTA zeolite with 200 watts of power and shorter operating time for 10, 20 and 30 minutes. This synthesis process can run faster due to the homogeneous heat generated from microwave radiation in zeolite samples which is commonly called the thermal effect [16].

Generally, to be able to synthesize zeolite by hydrothermal method, it takes 24-72 hours at 90°C, then [22] varied the synthesis temperature gradually 90°C and 95°C or the so-called step-change temperature method aimed at increasing the nucleation process and making zeolite crystallinity greater in a shorter time [22]. The same also applies to the microwave irradiation method where the reaction temperature will continue to rise until the microwave radiation ends, thus making the rate of nucleation and crystallization increase and zeolites can be synthesized in a short time. This is in accordance with research by [24]. From the study it can be seen when the sample is heated by microwave high temperatures can be achieved in a short time and vice versa for conventional methods, this can be seen in Fig. 7. below.

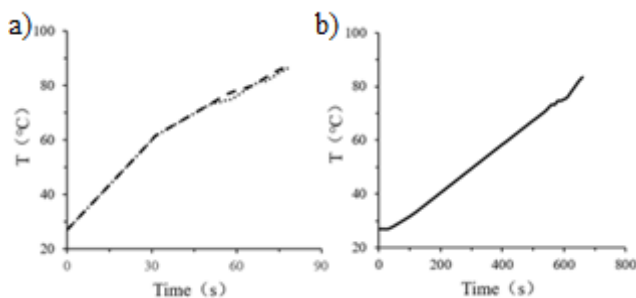


Fig 6. Comparison of (a) microwave radiation and (b) conventional methods [25].

The difference between conventional methods and microwave irradiation lies in the heat transfer method used. Conventionally, heat propagates to the sample by conduction and convection through solid and liquid intermediaries, while with microwave heat can propagate without intermediaries (radiation) and make only a small portion of heat wasted to the environment so that in the same time the heat generated by radiation is greater than the conduction and convection methods [25].

4 Conclusions

From the results of the research that has been carried out, it can be concluded that:

- 1) The microwave irradiation method is proven to be applicable in the synthesis of LTA zeolite from LNZ for 10, 20 and 30 minutes as seen from FTIR, XRD, XRF and SEM testing.
- 2) LTA zeolite synthesized with 30 minutes of microwave is proven to be able to compete with LTA zeolite synthesized by conventional hydrothermal method.
- 3) The best condition for the synthesis process of LTA zeolite using microwave irradiation with 200 watts of power is 30 minutes which is seen in the most double rings vibrations characterized by the highest intensity and area of 711 cm and 30,573 cm², the largest relative crystallinity of LTA zeolite 83.61% and the largest surface area of 11.055 m² /g.

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