



Preparation of Synthetic Zeolites From Myanmar Clay Mineral

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By

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ABSTRACT

Faujasite type zeolite X was successfully synthesized from Myanmar clay mineral, kaolinite, by treating with sodium hydroxide at 820°C followed by dissolution in water and hydrothermal treatment. It was found that the solution of fused clay powder can be crystallized at 90 °C under ambient pressure to synthesize faujasite type zeolite X. The effects of aging time and the amount of water on the formation of the product phase and Si/ Al ratios of the resulting products were investigated. Most of the Si and Al components in kaolinite might be dissolved into an alkaline solution and reacted to form ring-like structures. Then it was effectively transformed into zeolite materials. The maximum relative crystallinity of faujasite zeolite obtained was found to be 100%. Zeolite P was found to be a competitive phase present in some resulting products during hydrothermal treatment. The cation exchange capacity of kaolinite is very low, but increased after a proper treatment. It was found that the prepared faujasite type zeolite X, zeolite P and hydrogen zeolite (HZ) can reduce the hardness, the alkalinity, the total dissolved solid and the dissolved iron of raw water in the batch wise operation of water treatment. Therefore, it can be used as the cation exchanged resin for water treatment.

Key words: kaolinite, zeolite P, faujasite, zeolite X.

ACKNOWLEDGMENT

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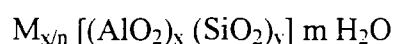
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CHAPTER (1)

INTRODUCTION

1.1. Zeolites

Zeolites have been potentially developed into key materials in the chemical industry for a variety of applications ranging from ion exchange to the catalysis of petrochemical processes. Zeolites are microporous, crystalline hydrated aluminosilicates symmetrically stacked (AlO₄) and (SiO₄) tetrahedra which result in an open and stable three dimensional honey comb structure with a negative charge. The negative within the pores is neutralized by positively charged ions (cations) such as sodium. Molecular sieves zeolites are crystalline hydrated aluminosilicates of group 1A and group 2A elements, in particular sodium, potassium, calcium, strontium and barium. Chemically, the structural formula of a crystallographic unit cell of a zeolite may be expressed in terms of the oxides,



where M is a cation of valence 'n'. 'm' is the number of water molecules, the sum of 'x' and 'y' is the total number of tetrahedra in the unit cell, and y/x usually has values of 1.5. However, recently, high silica zeolites have been prepared in which y/x is 10 to 100 or even higher.

There are two kinds of zeolites: natural zeolites and synthetic zeolites. About fifty natural zeolites are now known and more than one hundred and fifty have been synthesized for specific applications such as molecular sieves, cation exchangers, and industrial catalysts or as detergent builders. Typical chemical compositions of kaolin and some synthetic sodium zeolites are shown in **Table 1.1**.

1.2. Natural Zeolites

Zeolites are a family of crystalline aluminosilicate minerals. The first zeolite was described in 1756 by Cronstedt, a Swedish mineralogist who coined the name from two Greek words: zeo, to boil, and lithos, stones; meaning 'boiling stones', referring to the evolution of steam when the rock is heated. Relatively pure zeolite deposits have been discovered in over 50 countries, most of the major deposits being in or close to volcanic areas.

Table 1.1. Typical chemical compositions of kaolin and some synthetic sodium zeolites

Sr. No.	Material	Chemical composition	Structural formula (Unit cell structure)	Si/Al	SiO ₂ /Al ₂ O ₃
1	Kaolin	Al ₂ O ₃ 2SiO ₂ 2 H ₂ O	Al ₂ Si ₂ O ₅ (OH) ₄	1	2
2	Zeolite A	Na ₂ O Al ₂ O ₃ 2SiO ₂ 4.5 H ₂ O	Na ₁₂ Al ₁₂ Si ₁₂ O ₄₈ 27H ₂ O	1	2
3	Zeolite X	Na ₂ O Al ₂ O ₃ 2.5 SiO ₂ 6 H ₂ O	Na ₈₆ Al ₈₆ Si ₁₀₆ O ₄₂₄ 264H ₂ O	1.2	2-3
4	Zeolite Y	Na ₂ O Al ₂ O ₃ 5SiO ₂ 8-9 H ₂ O	Na ₅₆ Al ₅₆ Si ₁₃₆ O ₃₈₄ 250H ₂ O	2.5	5
5	Zeolite P (NaP1)	Na ₂ O Al ₂ O ₃ 3.3 SiO ₂ 12 H ₂ O	Na ₆ Al ₆ Si ₁₀ O ₃₂ 12H ₂ O	1.6	3.3
6	Faujasite	Na ₂ O Al ₂ O ₃ 2.4SiO ₂ 6.7 H ₂ O	Na ₂ Al ₂ Si _{2.4} O _{8.8} 6.7H ₂ O	1.2	2.4

There are about fifty different species of natural zeolites, each having a different chemical composition and structure but only eight zeolite minerals make up the major part of volcano-sedimentary deposits: analcime, chabazite, clinoptilolite-heulandite, erionite, ferrierite, laumontite, mordenite and phillipsite. Some help to assist plant growth while others make excellent filtration media, but the same zeolite will not necessarily do both well.

1.3. Synthetic Zeolites

The synthesis of zeolites from low-cost silica-alumina sources has been the aim of many experiments. Zeolites are usually synthesized in alkaline phase under hydrothermal conditions. The sources of silica-alumina are fly ash and kaolinite. In this experiment kaolinite from Hmawbe was used. Zeolites can be synthesized by the following methods:

- (1) Hydrogel Process
- (2) Kaolin Conversion Process
- (3) Fly Ash Conversion to Zeolites

Compositionally, zeolites are similar to clay minerals (kaolinite), both are aluminosilicates. They differ, however, in their crystalline structure. Kaolinite has a mineral layer charge and a low exchange capacity. Its surface area and absorption capacity is relatively low, but these properties of kaolin can be improved by proper treatment. Some groups have already studied the preparation of various zeolites from kaolin or other fly ashes and have made great progress in synthesis of zeolites A, P, faujasite type Y, faujasite type X, etc.

1.4. Applications of Zeolites

Natural zeolites are mainly used in building (cements) or in paper industry (fillers for paper). The largest amount of synthetic zeolites is used as ion-exchanger (water softener) in builders for detergents. Zeolites with the faujasite structure (FAU) are extensively used as catalysts and in gas separation. The potential applications of zeolites are listed in **Figure 1.1**.

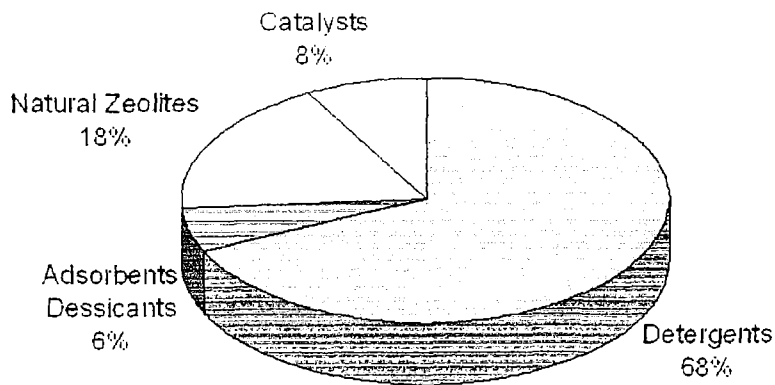


Figure 1.1. Potential applications of zeolites

The three important properties of zeolites, which make industrial potential applications, are their capacities-

(1) Adsorption: to sorb gases, vapours and liquids

Odor Control

The ability of zeolites to absorb certain gasses readily lends them to odor control applications. Public toilets, horse stable, poultry houses, cow milking stalls, calf pens and pig houses all release offensive ammonia fumes causing distress to the animal as well as human beings. These problems can be arrested by zeolite applications. Homeowners with pets often have pet litter trays which can lead to unpleasant and offensive odors. Zeolite placed in litter trays can eliminate the odors by absorbing ammonia and ammonium which are primary odor sources.

Water Adsorption / Desorption - Heat Storage and Solar Refrigeration

Natural zeolites possess a high affinity for water and have the capability of adsorbing and desorbing it without damage to the matrix or the crystal structure. This property makes them useful in desiccation as well as other unique commercial systems, such as heat storage. Without change in the matrix, coupled with a high heat of adsorption, allows natural zeolite to effectively store heat energy for late use.

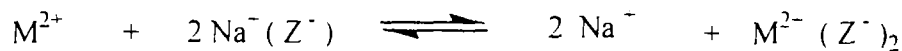
(2) Catalysis: to catalyse reactions

At the present time, zeolite based catalysts are employed in catalytic cracking, hydrocracking, isomerization of paraffins and substituted aromatics and in a process converting methanal to hydrocarbons. Catalytic cracking is the largest scale industrial process employing zeolite catalyst. Certain synthetic zeolites have revolutionized aspects of the petroleum industry.

(3) Ion exchange: to act as cation exchanger

Water Treatment

Synthetic zeolites are mainly used in water softening. The softening process can be carried out by allowing the hard water to percolate through a bed of zeolite exchanger in the sodium form. Replacement of calcium ions (or) magnesium ions in water by sodium ions takes place according to:



where (Z⁻) represents the zeolites. The exchanged material may be then regenerated for further water softening by washing a concentrated solution of sodium chloride.

Aquaculture

When fish feed, their normal biological processes quickly contaminate the water in which they live with ammonium. Natural zeolites can be used to ensure removal of this ammonium in fish farming, fish aquaria and in fish transport.

Agriculture and Horticulture

The correct type of zeolite application to soils may be expected to do the following:

- Act as a slow release mechanism of potassium and ammonium in a soil system
- Reduce the chance of ammonium toxicity by way of its cation exchange capacity avoiding root burning
- Improve the retention of ammonium, thus reducing the loss of nitrogen contained in fertilizer: such losses often occur by leaching and subsequent runoff resulting from heavy rainfall or excessive irrigation.

CHAPTER (2)

EXPERIMENTAL

2.1. Materials and Methods

Kaolin, aluminosilicate crystal, is a naturally occurring clay mineral deposit in various places of Myanmar such as Yemethin, Popa, Hmawbe, etc.. Mabisan clay from Hmawbi was used as raw material for the preparation of synthetic zeolites. The chemical composition of Mabisan clay is described in Table 2.1. The main components are aluminium silicate (85.99%) with small amount of other oxides. Commercial grade sodium hydroxide was used as an alkali source. Conversion of kaolin to synthetic zeolite P or faujasite type zeolite X by alkali fusion process prior to hydrothermal reaction was investigated in this experiment.

Table 2.1. Chemical composition of Mabisan clay

Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	LOI
Content (wt %)	51.82	34.17	1.72	0.79	0.72	0.41	11.65

2.2. Syntheses of Zeolites from Kaolin

In order to prepare zeolites from kaolin, kaolin was fused with NaOH (kaolin/NaOH = 1/1.55 in weight) at 820°C for 1h. After fusion, the fused kaolin was cooled and grounded. The fused powder was dissolved in various amounts of distilled water followed by stirring for 1h at room temperature. The mixture was aged for 1 day at the same temperature. This is the nucleation solution. Heat treatment of the fused solution resulted in the crystallization of zeolites. The solution was transferred to polyethylene container to crystallize hydrothermally at 90°C for a desired period. Subsequently, the zeolitic product was filtered, washed with distilled water and dried. A schematic diagram for synthesis of zeolitic materials from kaolin is shown in **Figure 2.1**.

A typical zeolite synthesis procedure from clay was performed as follows. 16.25 g of fused kaolin powder was dissolved in 230 ml of distilled water under stirring and stirred for another 1h. The slurry with a molar composition of 2.54 Na₂O: 1 Al₂O₃: 5.8 SiO₂ with various

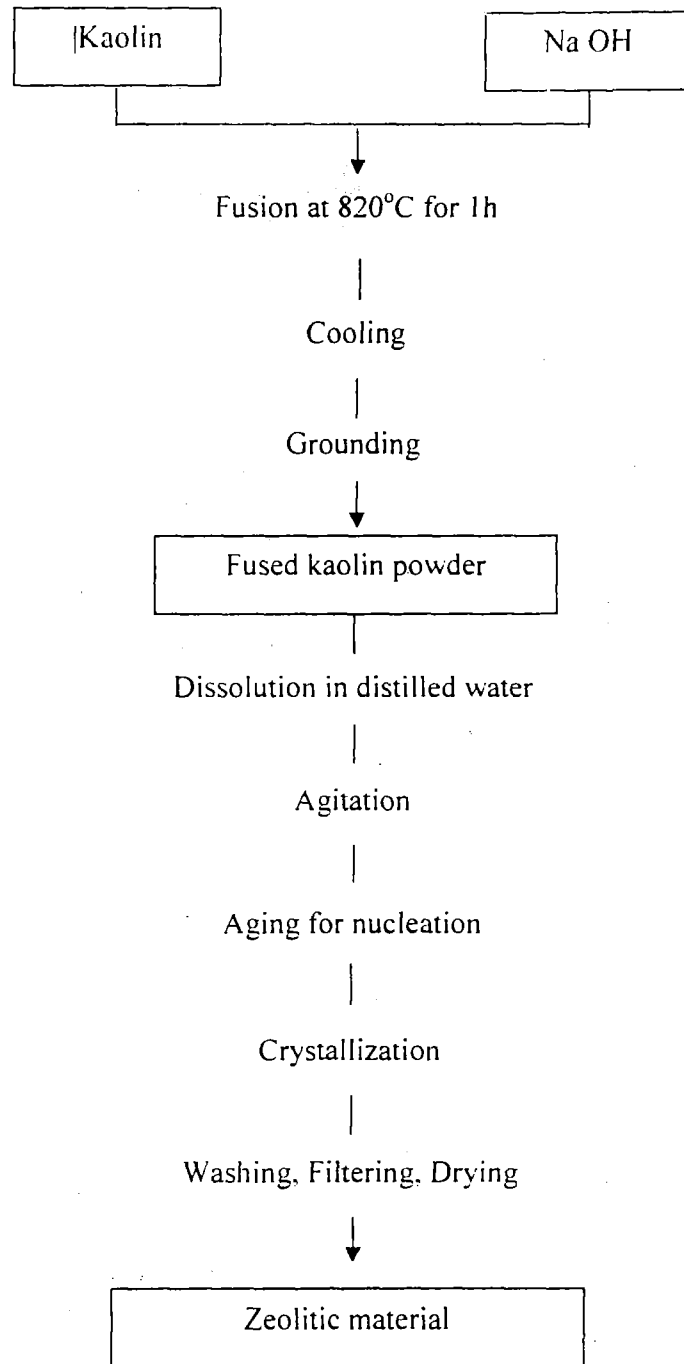


Figure 2.1. Schematic diagram for synthesis of zeolitic material from kaolin

amounts of water. The pH of slurry was found to be 13.6 and the slurry was kept at room temperature for 1d to construct the zeolite framework structure. Then the mixture was heated at 90°C for 4d or 5d. After heating the resultant precipitates were washed repeatedly with distilled water and dried at 100°C. The experimental conditions and molar ratio of the slurry in the synthesis of zeolites from kaolinite are described in Table 2.2.

In order to prepare the hydrogen zeolite (HZ), the faujasite type zeolite was treatment with 0.3 M hydrochloric acid solution for 30min followed by washing, filtering and drying.

Table 2.2. The experimental condition and molar ratio of the slurry in the syntheses of zeolites

Batch No.	Molar ratio				Stirring (h)	Aging (d)	Crystallization (d)
	SiO ₂	Al ₂ O ₃	Na ₂ O	H ₂ O			
Fused -4	2.54	1	5.8	484	-	-	4
F-I	2.54	1	5.8	421	1	1	4
F-II	2.54	1	5.8	213.7	1	1	4
F-IV	2.54	1	5.8	421	1	-	5
F-V	2.54	1	5.8	484	1	1	4

2.3. Characterization of Synthetic Zeolites

Chemical compositions of synthetic zeolites were analyzed. In order to know the crystal structure and the relative crystallinity, the X-ray diffraction (XRD) patterns were recorded on an X ray diffractometer. Rigaku D max 2200, Cu K α radiation, Ni filter at Department of Atomic Energy. A systematic investigation of the framework structures was carried out by FT-IR spectrophotometer. Genesis II, 1001 Fourier drive, USA and FT 45 at Pharmaceutical Research Department.

2.4. Water Treatment by Synthetic Zeolites Prepared

Water samples were collected from three different sources: (1). Kyaukse tubewell (2) Hlaingtharyar Industrial Zone Tubewell and (3) Pakokku tubewell. The investigation of ion exchange properties of prepared zeolites was carried out by using a batchwise operation. The water treatment column ($\phi = 2$ in and $l = 12$ in) was constructed by glass with an inlet and outlet of a water body to flow through it. The bottom of glass column was packed with cotton wool and the prepared zeolite was placed on it. The dosage of zeolite used for treatment was 5g/500 mL of water sample. The treated water was dropped from the outlet and collected to analyze. The water samples were analyzed before and after treatment.

CHAPTER (3)

RESULTS AND DISCUSSION

3.1. In situ Synthesis of Zeolites from Kaolinite

Zeolites are usually synthesized from kaolin in alkaline phase under hydrothermal conditions. In order to synthesize zeolites from kaolin, sodium hydroxide was used as an additive for alkali metal. Kaolin has a low exchange capacity. Its surface area and absorption capacity is relatively low, but these properties of kaolin can be improved by proper treatment.

Kaolin possesses the Si-O or Al-O octahedral and tetrahedral sheets which are inactive to modification or activation. This means that it is difficult to directly synthesize zeolites and the kaolin must be pre-activated to change this inert structure. The most effective way to activate such natural clay is to thermally transform the inert phase into the active phase at elevated temperatures in the presence of sodium hydroxide. Under the basic environment the inert skeletal structure of kaolin was converted into activated silicate and aluminate, which might be soluble in acidic or basic aqueous solutions.

Upon in-situ hydrothermal synthesis, the active Si and Al species dissolve in the alkali solution and form the supersaturated solution. They react with each other and reconstruct to form some ring-like reconstruction. It is this reconstruction and recrystallization of Si and Al species that results in the formation of faujasite type zeolite skeleton. The overall resulting data of the synthetic zeolites as regard to its synthesis conditions are presented in **Table 3.1**. It reveals the changes of chemical composition of zeolites prepared and corresponding $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio, percent yield and morphological structure of zeolites obtained with respect to variation of water volume, aging time and crystallization time. The yield percent of products ranged from 32-40%. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of zeolite P was found to be 2.5 and that of faujasite type zeolite was 2.67 in maximum value in this experiment. The corresponding Si/Al ratio varies from 1.5 to 3 for zeolite Y and the zeolite X has a Si/Al ratio of 1.2. The framework structures of types X and Y are topological related to the mineral faujasite as shown in **Figure 3.1**. Therefore, in this experiment the synthetic zeolite was faujasite type zeolite X as the Si/Al of synthetic zeolite ranged from 1.1 to 1.3.

Table 3.1. Synthesis conditions, chemical compositions and XRD results of synthetic zeolites

Batch No.	F/Water (g/mL)	Agitation (h)	Aging time (d)	Crystallization time (d)	Yield (%)	Chemical composition			XRD result
						SiO ₂ %	Al ₂ O ₃ %	SiO ₂ /Al ₂ O ₃	
Fused-4	1/14.2	-	-	4	36.3	41.8	17.9	2.51	Zeolite P+ Fau
F-I	1/12.5	1	1	4	34.5	38.0	28.1	2.3	Fau + zeolite P
F-II	1/6.2	1	1	4	32.0	36.1	26.2	2.3	Fau
F-IV	1/12.5	1	-	5	33.8	45.1	30.5	2.5	Fau +zeolite P
F-V	1/14.2	1	1	4	40.0	42.9	26.9	2.67	Fau
HZ	-	-	-	-	66.5	54.8	22.2	4.0	-

F = Fused kaolin powder

Fau = Faujasite

HZ = Hydrogen zeolite

In the synthesis of hydrogen zeolite (HZ) the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio was found to be decreased after treating the faujasite type zeolite with aqueous hydrochloric acid solution. It was obvious that some proportion of Al_2O_3 in zeolite was dissolved in acid solution. The yield percent of hydrogen zeolite was about 66% based on faujasite.

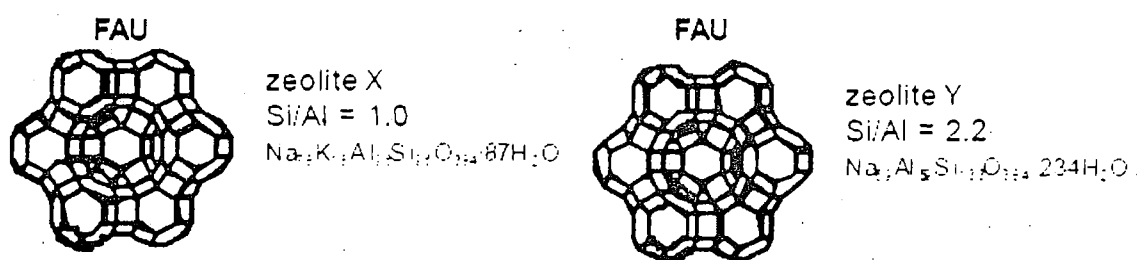


Figure 3.1 Crystal structures of faujasite type zeolite X and Y

3.2. The XRD Diffractogram of Synthetic Zeolites

The XRD patterns of prepared zeolites are found to be different in crystal structure and relative crystallinity influenced by the function of amount of water, aging time and crystallization time as shown in **Figure 3.2** to **3.6**. When the fused kaolin solution was cured at 90°C for 4 day -crystallization without aging, zeolite NaP was formed. A clear XRD pattern of zeolite NaP can be seen with faujasite type zeolite X which has a small relative crystallinity as a competitive phase present in the resulting product as shown in **Figure 3.2**. As shown in **Figure 3.6**, the XRD pattern of the synthetic zeolite is a typical structure of a faujasite type zeolite X which exhibits that it is feasible to in-situ synthesize faujasite by alkali fusion process prior to hydrothermal reaction.

3.3. Effect of Water Volume

The fused kaolin was mixed with water and aged for 1 d at room temperature. When the fused kaolin solutions were cured at 90°C for 4d, faujasite type zeolite X was formed. **Table 3.2** also shows the XRD pattern of the resulting products influenced by the amount of water added upon Na X zeolite synthesis.

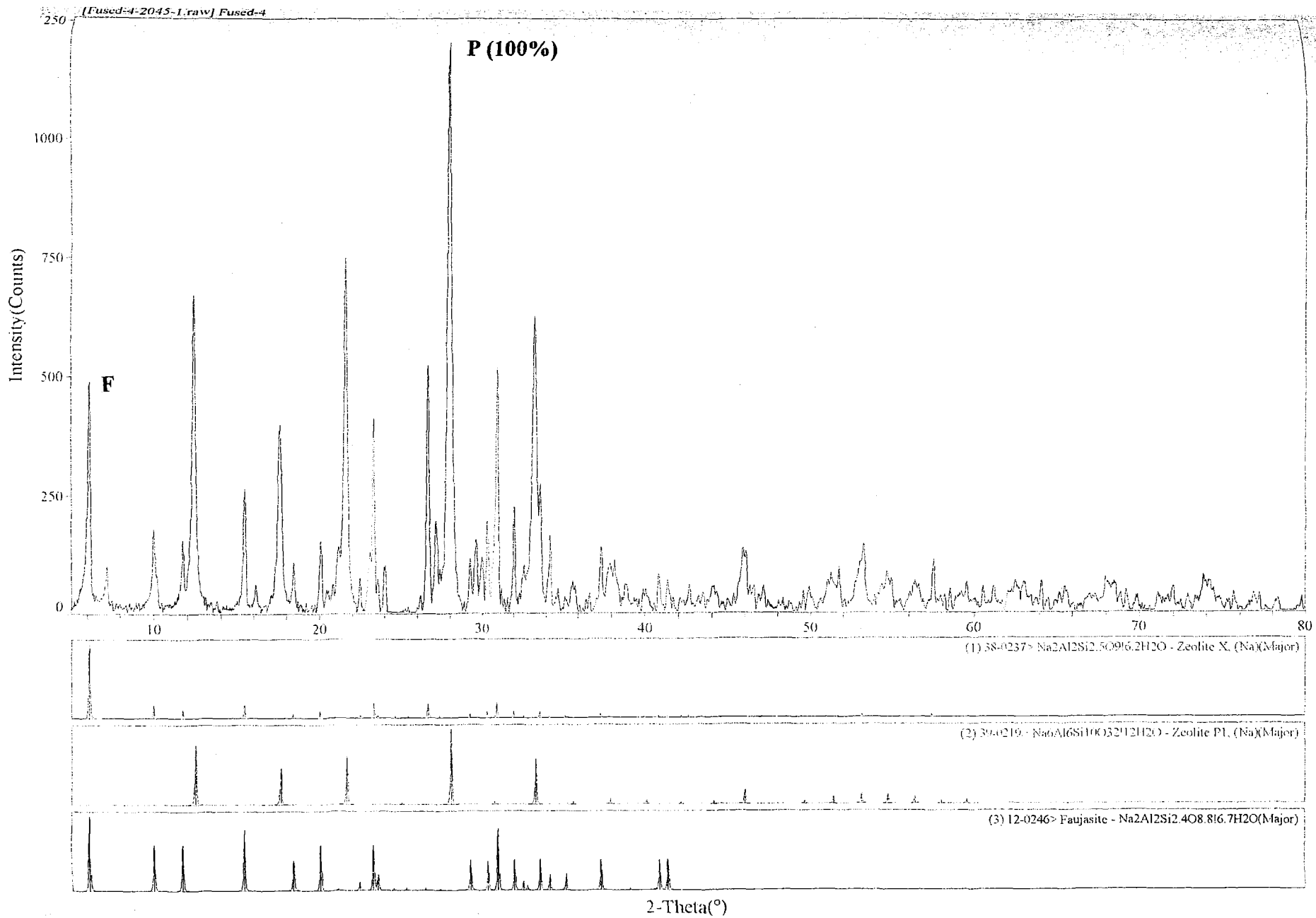


Figure 3.2. The XRD pattern of synthetic zeolite (zeolite NaP) prepared without aging Fused -4)

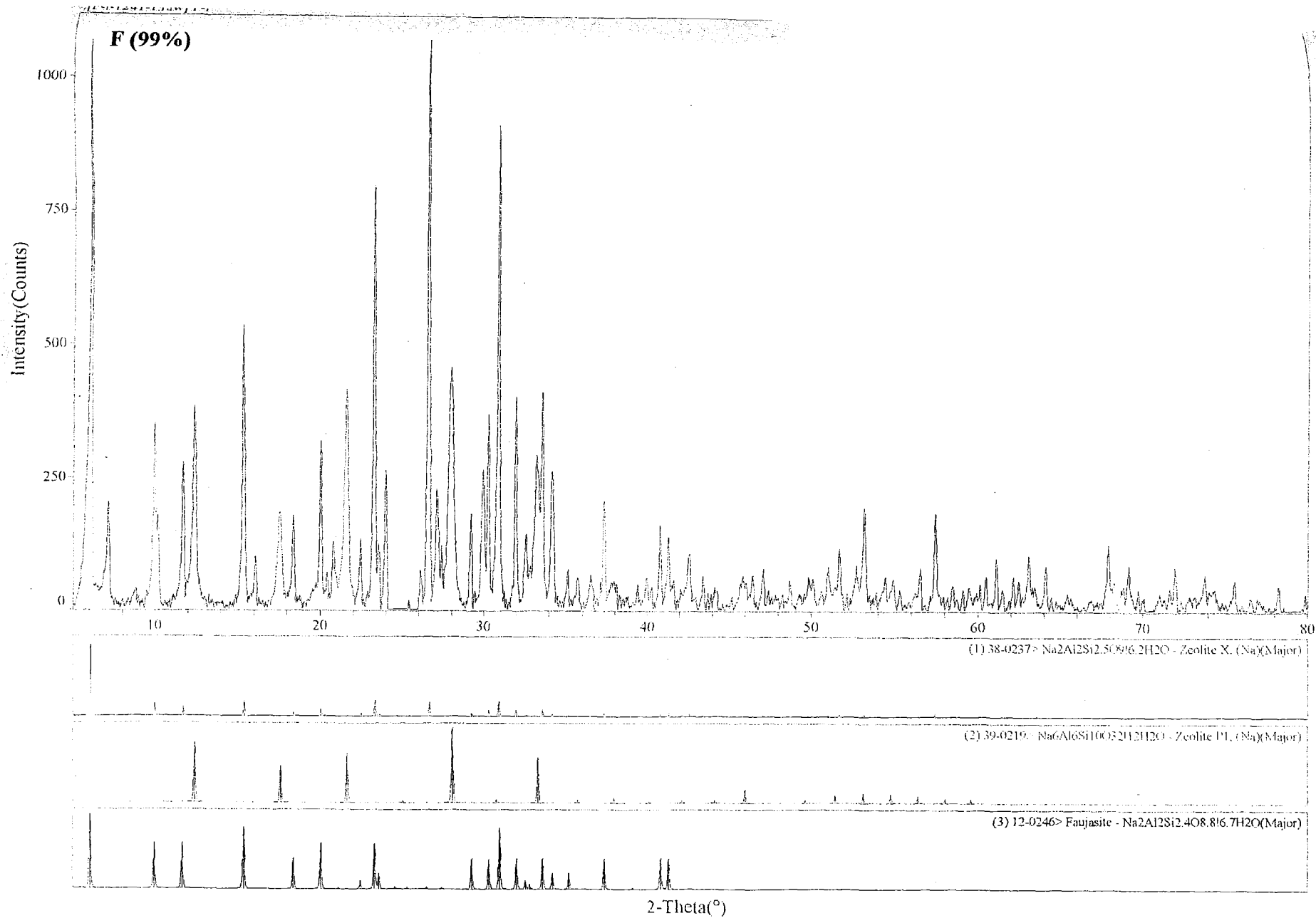


Figure 3.3. The XRD pattern of synthetic zeolite (faujasite) prepared (F-I)

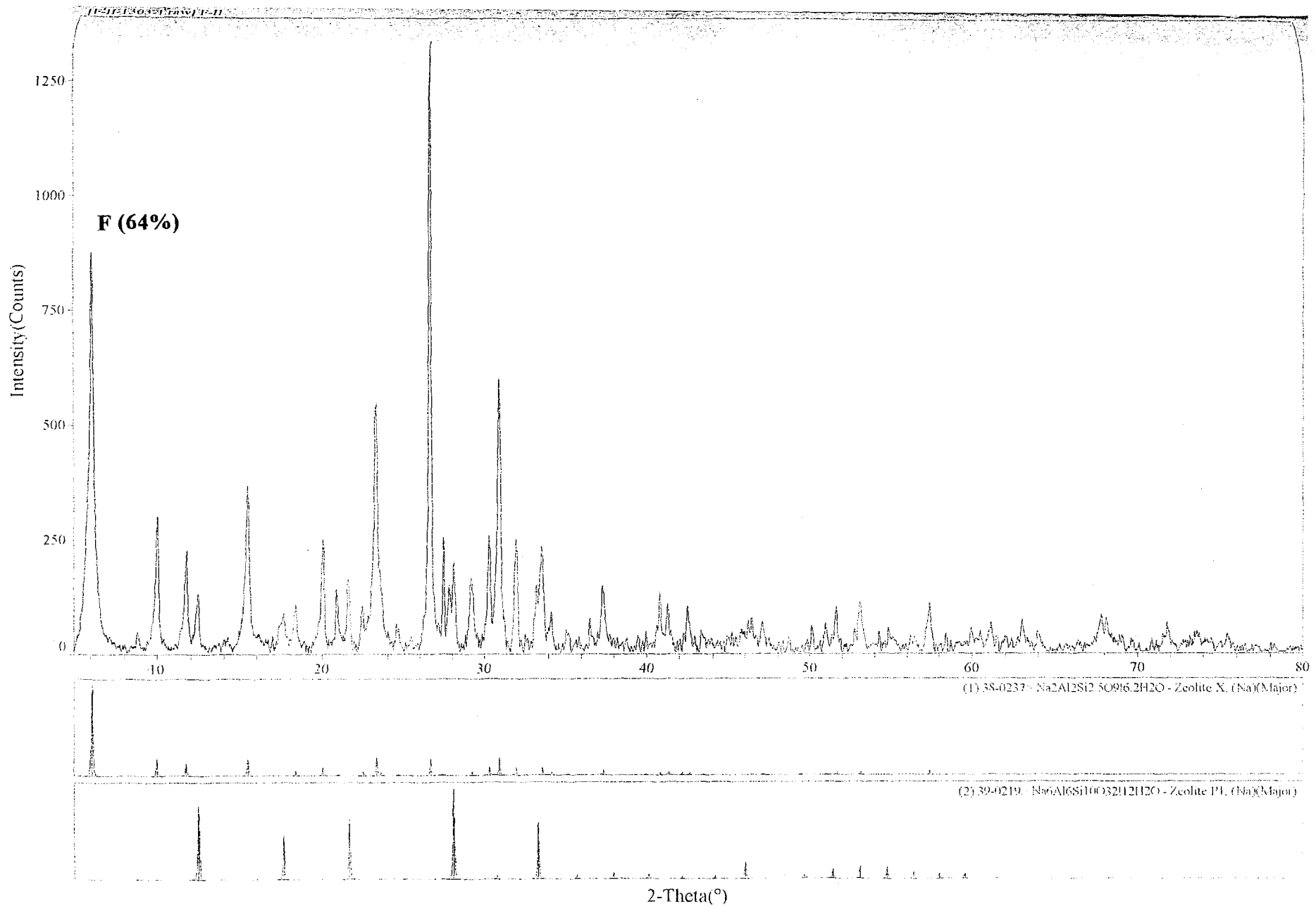


Figure 3.4. The XRD pattern of synthetic zeolite (faujasite) prepared (F-II)

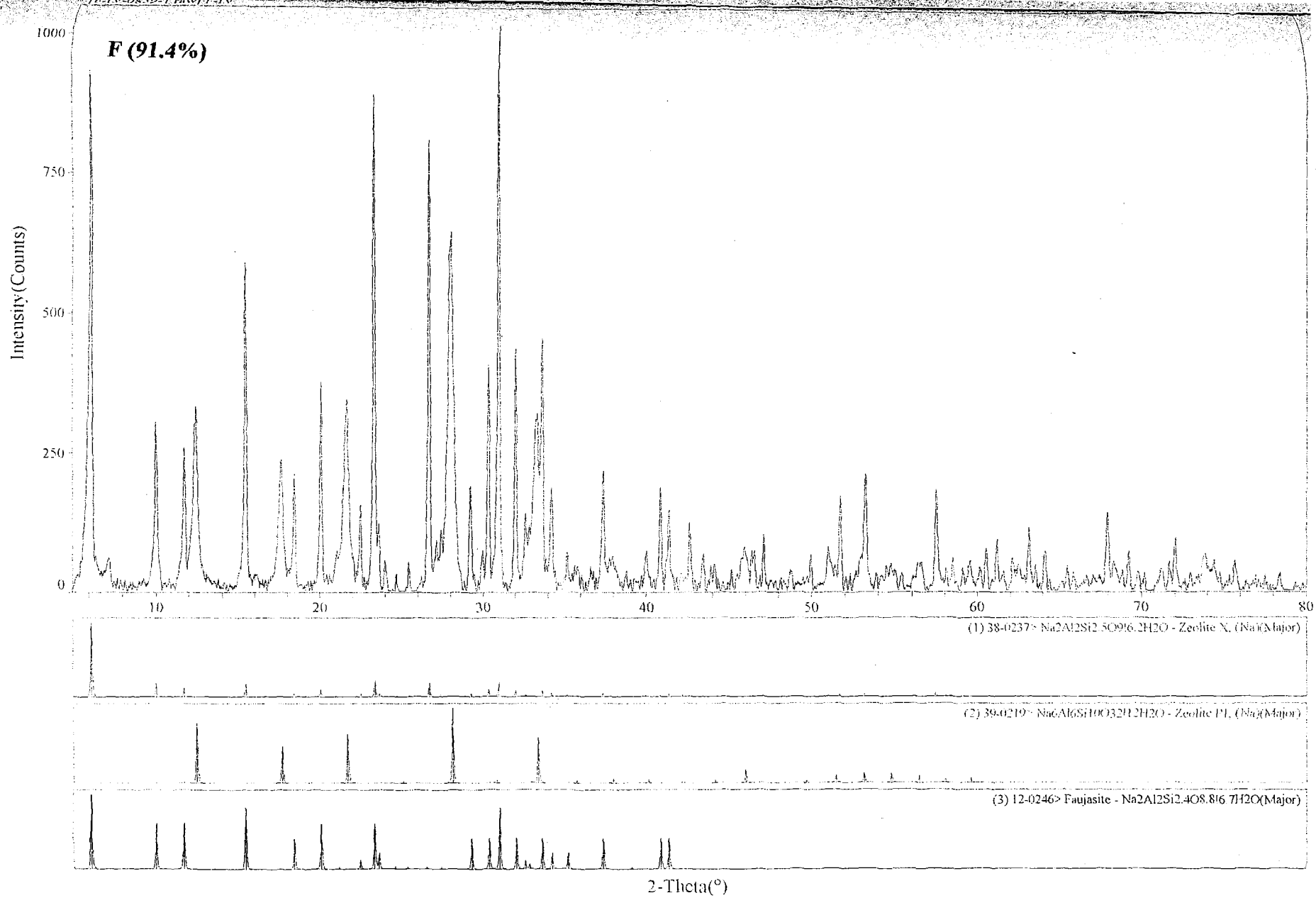


Figure 3.5. The XRD pattern of synthetic zeolite (faujasite) prepared (F-IV)

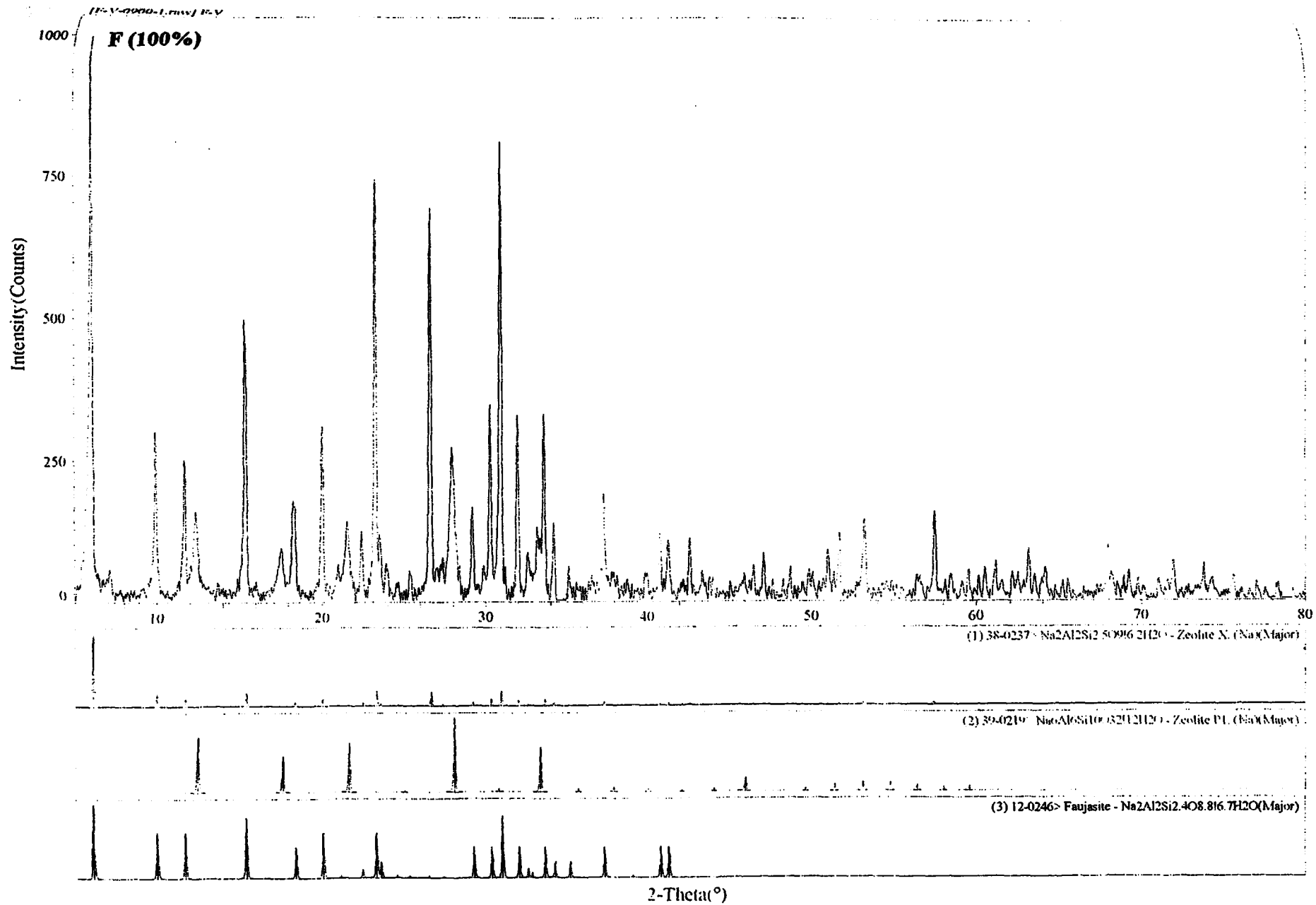


Figure 3.6. The XRD pattern of synthetic zeolite (faujasite) prepared (F-V)

Table 3.2 The effect of water added in the synthesis of zeolite

Sample	Water added* (mL)	XRD pattern	Agitation (h)	Aging time (d)	Crystallization time (d)
F-II	6.25	64% faujasite	1	1	4
F-I	12.3	99% faujasite			
F-V	14.2	100% faujasite			

* Volume of water added for 1g of fused kaolin

XRD analyses show that the amount of water exerts a strong influence on the relative crystallinity of zeolites prepared from kaolinite. An increase in the amount of water increases the relative crystallinity of the faujasite zeolite NaX. When 14.2 mL of water for 1g of fused kaolin was used, the maximum crystallinity of faujasite (100%) which ascribes the sharp diffraction peak of the faujasite can be exist. It is obvious that the water volume plays an important role in the formation of the zeolite prepared by kaolin fusion process.

The functions of water can be described as follows. (i) The water volume contributes to the pH value of the colloid system and accordingly influences the external dielectric constant and size of gel particles. (ii) The pH value influences the sedimentation velocity of gel particles, which results in the change in the gel concentration and the transfer rate of gel particles. This reveals that too much or too little water may inhibit the formation of faujasite from kaolin.

3.4. Effect of Aging Time

Aging is also an important parameter for the zeolite synthesis process. The effect of aging conditions on the resulting products is shown in **Table 3.3**. It indicates that the faujasite zeolite with high relative crystallinity could be obtained at the aging time for 1 day. Without aging in the synthesis of faujasite, zeolite P was found to be a competitive phase and the relative crystallinity of faujasite decreased as shown in the XRD diffraction patterns. Therefore, the pure faujasite with higher relative crystallinity can be developed at aging time for 1d and crystallization time for 4d (**Table 3.3**). However, prolonging the aging time can accelerate the growth and agglomeration of gel particles so that the faujasite zeolite possesses a lower micropore volume and surface area.

Table 3.3. Effect of aging time on the synthesis of zeolite

Batch No.	Agitation (h)	Aging time (d)	Crystallization time (d)	Water added* (mL)	XRD result
Fused -4	-	-	4	14.2	Zeolite P (100%) Faujasite (40%)
F-V	1	1	4	14.2	Faujasite (100%)
F-IV	1	-	5	12.5	Zeolite P (32%) Faujasite (91.4%)
F-I	1	1	4	12.5	Faujasite (99%)

* Volume of water added for 1g of fused kaolin

3.5. FT-IR Spectrum of Zeolite Prepared from Kaolin

Vibrations of the frameworks of zeolite give rise to typical bands in the mid and far infrared. The FT-IR spectrum of zeolite faujasite prepared is shown in **Figure 3.7**. The presence of Si-OH-Al band into a SiOH group in the zeolite prepared is indicated by the broad OH stretching band at 3479 cm^{-1} . A band at 462 cm^{-1} shows T-O bending of internal vibration of TO_4 . The vibration of double six membered rings is found at 566 cm^{-1} . Typical region for the spectra of the alumino silicate lattice is the mid infrared region of $400\text{-}1300\text{ cm}^{-1}$. The characteristics of the FT-IR spectrum of faujasite prepared are assigned in **Table 3.4**.

Title: Sample for F-5
Filter: Three Point Center of Gravity

cm-1	%T	cm-1	%T	cm-1	%T	cm-1	%T
461.86	4.60	565.87	11.47	676.20	13.06	964.58	1.07
974.34	0.97	983.62	0.96	992.70	0.97	3478.55	6.12

Name of owner: Dr.P
Sample: F-5

Comments : 1%KBr

Operator: Daw Khin Aye Than & Myint Myint Khine

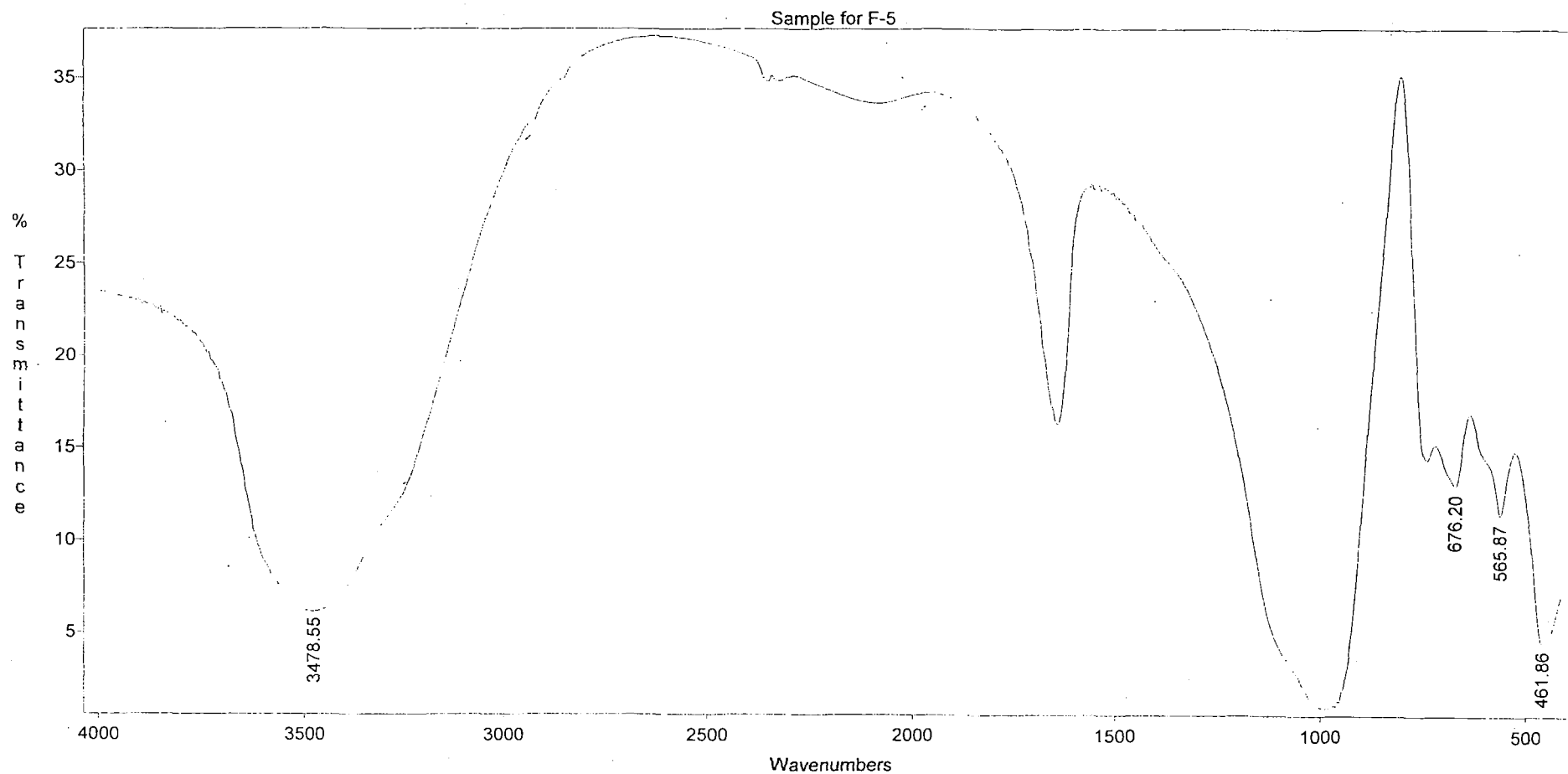


Figure 3.7. FT-IR spectrum of faujasite type zeolite X

Table 3.4. The characteristics of the FT-IR spectrum of faujasite

Frequency (cm ⁻¹)	Assignment
3479	OH stretching
1300-400	Aluminosilicate lattice
965-993	TO ₄ Asymmetrical stretching (Internal tetrahedral)
676	TO ₄ Symmetrical stretching (Internal tetrahedral)
566	Double ring (External linkage)
462	TO bending (Internal tetrahedral)

T = Si or Al

3.6. Ion Exchange Behavior of Synthetic Zeolites

Zeolites with different Si/Al ratios are expected to have different ion-exchange behavior since their structures are slightly different. The ion exchange capacity of zeolite P is very small. The better performance of faujasite type zeolites than zeolite P probably originates from faujasites having larger pore sizes associated with the D6R and cage units in their structures. The hardness of raw water and dissolved iron were found to be decreased by passing the column containing the zeolites prepared. It can be seen that the significant amount of calcium and magnesium ions can be removed by using faujasite type zeolite and zeolite P. Faujasite type zeolite has more cation-exchange activity than zeolite P according to the experimental results (Table 3.5). The hydrogen zeolite (HZ) prepared by treating with acid can reduce the pH, alkalinity, hardness, total dissolved solid and dissolved iron of raw water. This raw water is very hard and dissolved a large amount of salts. Thus the high dosage of zeolite may be required. If the dosage of hydrogen zeolite increases, the alkalinity and total dissolved solid of raw water may be significantly reduced to drinking water level.

Table 3.5. Changes of Some Chemical Constituents in Hard Water Treated by Synthetic Zeolites

	pH	Hardness (ppm)	Alkalinity (ppm)	Total Dissolved Solid (ppm)	Fe (ppm)
Raw water (I)					
(Kyaukse tubewell)	9.0	130	500	2400	8.0
Treated water by F-I					
(Faujasite)	9.0	10	460	2200	ND
Raw water (II)					
(Hlaing Tharyar Industrial Zone Tube well)	8.1	72	120	450	3.2
Treated water by F-I					
(Faujasite)	8.5	4	124	390	ND
Treated water by HZ					
	7.3	14	66.6	300	ND
Raw water (II)					
(Pakokku tubewell)	8.3	1546	1480	9990	0.6
Treated water by HZ					
	7.4	1370	1040	9077	ND

Dosage = 5g zeolite for 500 mL raw water

CHAPTER (4)

CONCLUSIONS

Synthesis of faujasite type zeolite from mineral kaolinite was successfully investigated by hydrothermal reaction under alkaline medium. The XRD pattern shows the crystal structure of faujasite zeolite. Water treatment by using zeolites prepared was carried out by batchwise column operation. The hardness of raw water and dissolved iron in water sample were found to be decreased. The H form zeolite can reduce the pH, hardness, alkalinity, total dissolved solid and iron of the very hard raw water. Therefore, synthetic zeolite, faujasite, could be used to soften the hard water.

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