CHAPTER IV

RESULTS AND DISSCUSSIONS

4.1 Characterization of coal fly ash

The results from XRD analysis of the coal fly ash (CFA) samples are given in Table 4.1. The four samples were collected at different dates where Sample 1 was taken in July, Sample 2 in August, Sample 3 in September, and Sample 4 in October, all in the year 2006. This was to check for the consistency of the results which could be subject to the variation in the quality of the raw material. It can be seen from these results that major compositions of all the samples were Si, Al, and Fe whereas minor compositions were K and Ca, and others were trace elements. From this result, the molar ratios between Silicon and Aluminium could be calculated as 2.29, 4.05 and 4.62 for the Samples 1, 2, and 3 respectively. This information revealed the possibility in converting such material into zeolite with the Si/Al molar ratio of about 2<Si/Al<5 (zeolites A, X, Y and faujasite). The BET surface area of the CFA samples # 1, 2, and 3 were about 45, 42 and 38 m^2/g, respectively. Note that Sample # 4 was not evaluated for it's BET. The SEM image in Fig. 4.1 demonstrates that the morphology of CFA was amorphous.

4.2 Effect of NaOH/CFA on adsorption characteristics

A series of experiments were undertaken to determine the effects of NaOH/CFA weight ratio on zeolite formation. As a preliminary examination, the adsorption characteristics was evaluated using their cationic exchange capacity or CEC as an indicator and the CEC of the final products are presented in Fig. 4.2. The results showed clearly that CEC could be enhanced just by increasing the quantity of NaOH during the fusion process. This increase was due to a higher degree of zeolization taken place as a result of an increase in the reaction extent associated with a higher NaOH content (Shigemoto et al., 1993). However, there seemed to be a limit on the level of NaOH and the increase in NaOH/CFA ratio beyond 2.5:1 did not seem to have benefits on the CEC value, rather the CEC was drastically reduced at this high NaOH/CFA ratio. This could be due to the deterioration of zeolite properties. To

investigate this effect, it was better to look at the XRD pattern of the zeolite products obtained from different synthetic conditions.

The XRD pattern of the zeolite product X was employed to evaluate for the crystallinity percentage. In the calculation of this crystallinity percentage, the intensity of the five most distinguishable XRD peaks were chosen where:

$$\%\text{crystallinity} = \frac{\sum \text{intensity of XRD of peak of product}}{\sum \text{intensity of XRD peak of product}} \times 100 \qquad [4.1]$$

The results of calculation are given in the right axis of Fig. 4.2 and Table 4.2. From this result, it was clear that the zeolite obtained from the NaOH/CFA ratio of 2.25 exhibited the highest crystallinity. This could be explained using the concept of crystalization growth constant (Kg). Lindner and Lechert (1994) and Iwasaki et al (1997) proposed that Kg increased with alkalinity according to the following relationship:

$$Kg \propto A^P \qquad [4.2]$$

where A is the concentration of sodium hydroxide in the liquid phase, and p is a constant which was related to the molar ratio Si/Al in the zeolite (which was specifically referred to faujasite in both references). If this relationship was also true for the zeolite obtained from this work, it shows that an increase in alkalinity would enhance the crystallinity of the zeolite product. This explained its high CEC property where the crystallinity had not reached its maximum at low NaOH/CFA ratio and therefore did not exhibit high CEC property. On the other hand, as NaOH/CFA ratio exceeded 2.25, the extent of the reaction might be too high that the structure of the zeolite was destroyed and %crystallinity decreased. This agreed with the statement from Breck (1998) who showed that the crystallization of zeolite became unstable at an extremely high alkallinity. Therefore a lower CEC was observed.

Although the highest CEC value (153.2 Meq/100 g) was achieved with the NaOH/CFA ratio of 2.25, the yield was not as good as that obtained at the ratio of 1.75. The calculation illustrated that the yield obtained from the preparation at the NaOH/CFA ratio of 2.25 was only 41% when compared with 62% at the ratio of 1.75. This must be due to the weight loss during the reaction with NaOH. In addition, the handling of the sample at the NaOH/CFA ratio of 2.2.5 was difficult as the mixed sample combined into a solid slug. Hence, the selected NaOH/CFA ratio for subsequent experiments was 1.75.

4.3 Effect of Si/Al on adsorption characteristics

The Si/Al molar ratio is also considered to be quite important because SiO_2 and Al_2O_3 are the major compositions of zeolite. The variety in Si/Al would directly affect the properties of the zeolite products. Some zeolites such as zeolite alpha and zeolite X, crystallization could be increased by decreasing Si/Al ratio, and on the other hand, some zeolites such as ZSM5 [Breck, 1988] crystallization increases with increasing Si/Al ratio. Although the CFA samples employed in this work were obtained from the same source, they were collected from different time periods and therefore contained different Si/Al ratios (see Table 4.1). Despite so, all of the synthesized zeolite was found to be closest to that of type X. Fig. 4.3 illustrates the XRD pattern of zeolite X standard whereas Fig. 4.4 shows the XRD patterns of the product from this work at various Si/Al ratios and different fusion temperature. In such case, the increase in crystallinity could be achieved by decreasing Si/Al ratio. The results as shown in Fig. 4.5 confirmed just that for the whole range of fusion temperature used in the syntheses. As the CEC depended significantly on the crystallinity of the zeolite, it also varied inversely with the Si/Al ratio, and this is demonstrated in Table 4.2. The CEC values obtained with the Si/Al ratios of 2.29, 4.05, and 4.62 were 250, 240, and 221 meq/100g, respectively.

High Aluminium content in the zeolite product which was attributed to the high Aluminium content in the original CFA could be the main reason for the finding above. The number of negative charge or equivalent to cation (e.g. Na) in the zeolite structure depends on the ratio of Aluminium in its structure and therefore the zeolite with more Aluminium would contain higher amount of cation which could exchange with other cations during the adsorption process (Rabo, 2001). Therefore one would observe a higher CEC in zeolite with high Aluminium content. For instance, Sample # 2 which was obtained from CFA with Si/Al of 4.05, the CEC was 240 meq/100g whereas Sample # 1 from CFA with Si/Al of 2.29 had a higher CEC of 250 meq/100g (see Fig. 4.5 for more detail).

In terms of the completeness in the formation of zeolite, the crystallinity percentage of each zeolite products was calculated using the same technique as explained in the previous section, and the results are shown in Fig. 4.6 and Table 4.2. The highest peak intensity was obtained from the Si/Al of 2.29, and the intensity decreased when increasing Si/Al ratio. The highest crystallinity percentage was as high as 91.0% using Si/Al ratio of 2.29. An increase in Si/Al ratio decreased the

crystallinity percentage. The %crystallinity decreased from 91.0 to 52.6 when Si/Al ratio increased from 2.29 to 4.05 and remained at 52.6 when the Si/Al ratio increased further to 4.62.

It was still unknown, with the results from this work, of why the zeolite products generated from the raw materials with different Si/Al ratio gave the same type of zeolite at the end of the synthesis. This finding was also reported by Somerset et al., 2005. It should be noted, however, that the properties of the raw material (CFA) outside the range investigated in this work might lead to different types of zeolite products. This work provided a reasonable range of consistency (three months of sample collection) but it will be better to extend the scope of the work, e.g. to include the CFA from the range of one operating year of the power plant to ensure that the effect of annual changes in environment was more completely studied.

Fig. 4.6 illustrates that the Si/Al ratio also affected BET surface area and BET surface area of the zeolite crystal decreased with increasing Si/Al ratio. The BET surface areas were 324, 289, and 236 m^2/g using Si/Al ratio of 2.29, 4.05, and 4.62, respectively. It is also noted here that the following investigation was based on the synthesis at 550°C for 1 h, 85 ml of water in the CFA/NaOH mixture, mixing temperature of 30°C at 12 h, and crystallization temperature of 90°C for 2 h. These conditions might not be at the optimal. Due to our inadvertent experimental limitation, the optimal conditions were only revealed from the CEC analyses which were completed after the preparation steps.

4.4 Effect of fusion temperature on zeolite properties

The fusion is a critical step in the synthesis of zeolite and the fusion temperature is a very important factor in controlling the properties of the zeolite product. Fig. 4.6 demonstrates the effect of temperature on the properties of the obtained zeolite. There are various reports on the value of the fusion temperature and most recommended the temperature of higher than 500°C for a proper synthesis, e.g. Rayalu et al. [2000] and Sommerset et al. [2005] reported the optimum fusion temperature of 600 °C, Molina and poole [2003] reported the fusion temperature of 550°C, Ojha [2004] and Shigemoto et al. [2006] reported the optimum fusion temperature of 550 °C. For this work, the optimum fusion temperature was 450°C which was the temperature that gave the highest level of CEC for all Si/Al ratios.

The effect of fusion temperature on the % crystallinity of the zeolite product was also illustrated in Fig. 4.6. At the Si/Al ratio of 2.29, the crystallinity percentages of 37.4, 44.4, 95.0, and 91.0 were obtained from the fusion temperature of 250, 350, 450, and 550°C, respectively. The highest % crystallinity was found at 450°C, and at other Si/Al ratios, the highest % crystallinity was also found to occur at the same temperature. It could, hence, be concluded that the optimum fusion temperature for this experiment was 450°C. Below and beyond this, a complete crystal could not be achieved at the level obtained at 450°C and impurities might be resulted. These impurities might be due to the remaining impurities in raw materials or could be from the formation of some other types of zeolites or the mixture between zeolite and quartz. The results of BET surface area and pore volume are also showed in Figure 4.6. At Si/Al ratio of 2.29, the surface areas were 60, 112, 340, and 324 m^2/g, the Pore volumes were 7.32, 10.11, 86.35, and 72.52 m^3/g for fusion temperatures of 250, 350, 450, and 550°C, respectively. Figure 4.5 shows that CEC of the zeolite product was also highest at the fusion temperature of 450°C, regardless of the Si/Al ratio.

4.5 Effect of fusion time on zeolite properties

A proper amount of time is needed for the fusion process, and this was often reported to differ from case to case. For instance, Rayalu et al. [2000] reported the fusion time range from 1-2 h, Molina and poole[2003] reported the fusion time at 2 h, Ojha et al[2004] reported fusion time of 2 h, Somerset et al. [2005] 1-2 h, and Shigemoto et al. [2006] 1 h. This could be due to the different in the type of raw materials used in the syntheses. However, there were statements that a shorter time would lead to an incomplete reaction whilst a longer time would result in damage of the zeolite crystal structure and both led to a bad CEC property. In this work, a suitable fusion time was found to be around 45 min for all CFA samples, i.e. at the Si/Al ratio of 2.29, 4.05, and 4.62, the maximum CEC values were 297, 288, and 278 meq/100 g at the fusion time of 45 min, respectively (see Fig. 4.7).

The XRD patterns of zeolite X synthesized from CFA at various fusion times are shown in Fig. 4.8. In the case of short fusion time, such as 15 and 30 min, the peak characteristics of zeolite could not be found and the only peak presented was quartz. Zeolite X peaks were found when the fusion time was 45 and 60 min. The crystallinity percentage calculated from the peak intensities are shown in Fig. 4.9. As

seen from the figure, the crystallinity percentage obtained from 45 min fusion time was higher that from 60 min. Hence, the fusion time of 45 min was chosen to be the optimal. Similarly the BET surface area and pore volume as shown in Fig. 4.9 were found to be the greatest at the fusion time of 45 min.

4.6 Effect of crystallization temperature on zeolite properties

The effect of crystallization temperature on the XRD pattern of the zeolite products is presented in Fig. 4.10. At 30 and 60°C, there were no appreciable crystalline phases in the spectrum. It was generally accepted that a higher temperature could result in the formation of large particle size. Hence, depending on temperature, the crystals could be so small as to be undetectable with the detection limits (Breck, 1974). The crystallization temperature of 90°C provided well crystallized zeolite, and consequently high cation exchange capacity (Table 4.2 and Figure 4.11). The reason could be that higher temperature supports the rate of crystallization of zeolite better than the lower temperature (Tsitsishuili, 1992). This could be explained using the following equation where Kg varies with temperature according to Arrhenius relationship:

$$Kg = Ae^{-Ea/RT}$$ [4.3]

where Kg is the crystal growth, A the appropriate constant, E_a the activated energy of the crystal growth process, R the ideal gas constant (8.3143 $JK^{-1}mol^{-1}$), and T the absolute temperature. It could be that conditions at 30 and 60°C did not possess adequate energy that overcame the activated energy of zeolite x ($\approx$ 62.5 $kJmol^{-1}$) and therefore the crystal could not be properly formed. On the other hand, the crystallization temperature of 90°C allowed the reaction to occur and the formation of zeolite x was observed.

The effects of crystallization temperature on BET surface area, pore volume, and % crystallinity are elucidated in Figure 4.12. All confirmed that 90°C was the best condition for the synthesis of the zeolite from CFA.

4.7 Effect of crystallization time on zeolite properties

Fig. 4.13 illustrates the effect of crystallization time on the CEC of the zeolite products. The results suggested that the optimal crystallization time was 2 h. A shorter or a longer time would result in a worse CEC. In the case of short crystallization time

(1 h), the peak of zeolite could not be found and the only peaks presented quartz (see Fig. 4.14). Peaks of zeolite X could only be apparent after 2 and 3 h of crystallization time. The peak intensities reached the highest at 2 h and the crystallinity percentages are shown in Fig. 4.15. It was true that a longer crystallization time allowed a better formation of larger crystal. However, a too long crystallization time resulted in the change of zeolite structure which could be caused by the presence of impurities such as some other types of zeolites or quartz. These impurities lowered the level of crystalinity of the zeolite products. The CEC value was also decreased because the CEC value was directly depended on the zeolite structure (Tsitsishvili, 1992).

Table 4.2 shows the crystallinity percentages calculated from the peaks intensities at different crystallization times. The highest crystallinity percentage was observed at 2 h crystallization time for all Si/Al ratios. By using Si/Al ratios of 4.05 and 4.62, one hour crystallinity was not enough for the zeolite to form and the final synthesized products were quartz. The reduction in peak intensities after 2 h occurred. Hence, 2 h crystallization time was the optimum and was chosen for further studies. The results of BET surface area and pore volume are illustrated in Figure 4.15 which demonstrated also that the crystallinity time of 2 hours was the most suitable for the generation of zeolite X from the CFA.

4.8 Effect of amount water during the crystallization period on zeolite properties

The ratio of Si/Al and water during the crystallization period indicated the type of zeolite produced in the reaction and also the crystallinity of the synthesized zeolite. As shown in empirical formula of zeolite ($M_{2/n}Al_2O_3*xSiO_2*yH_2O$), water is one major component in zeolite structure. The change in amount of water during the crystallization period led to changes in the structure of zeolite. Amorphous could be obtained when an unsuitable amount of water was used. In this work, Amount of water at 65 ml CFA was best for the synthesis of zeolite. The increase of water to 85 ml reduced the peak intensity of zeolite X (Fig. 4.16). At 115 ml of water, no crystal growth was detected and the product was still in amorphous form. The results could be discussed using Eq.4.4:

$$Kg \propto f(C) \tag{4.4}$$

where Kg is the crystal growth, $f(C)$ the concentration function, C the concentration of sodium hydroxide. It is not unexpected that dilution of crystallizing system (e.g., an

increase of water content) caused a decrease of the concentration of reactive species in the liquid phase (C), thereby a decrease of the crystal growth rate. The crystallinity percentages acquired from various amount of water are shown in Fig. 4.17. SEM images of the product from the syntheses are displayed in Fig. 4.18. From this figure, it was clear that the crystal was only formed when the water was lower than 85 and amount of water at 65 ml provided well crystallized zeolite. The effect of amount of water during the crystallization period as in Fig. 4.19 emphasized again that 65 ml was best for this synthesis.

4.9 Effect of mixing temperature on zeolite properties

The effect mixing temperature was studied by varying the temperature in the range of 30 and 90°C using the constant Si/Al ratio of 2.32, fusion temperature 550°C, fusion time 1 h, water 85 ml, crystallization temperature 90°C, crystallization time 2 h. Fig. 4.20 shows the XRD pattern of the products where the peak intensity of zeolite X increased with increasing mixing temperature. This might be because high temperature led to an increase in crystallization rate (Breck, 1987). Although an increase in peak intensities of zeolite X was observed at high temperature, some unexpected peaks were also presented at this condition. These unexpected peaks might come from the occurrence of impurities. As presented in Fig. 4.21, SEM images illustrate, in addition to the formation of zeolite X, the occurrence of zeolite P. This was because there were overlapping formation conditions where the two zeolites could form simultaneously, and this is shown diagrammatically as in Figure 4.22. This was possible as the dissolution of Silicon become more favor at high temperature. As a result, the synthesized zeolite contained more Silicon in its structure which makes the properties closer to a higher Silicon content zeolite P. Similar finding was reported by Molina and poole (2003). Zeolite P acted as the impurities in the process of zeolite X synthesis, and this drastically reduced the CEC value as demonstrated in Fig. 4.23. This was because zeolite P contained lesser amount of Aluminium and therefore the number of cation for ion exchange process became more limited than that of zeolite X.

4.10 Effect of mixing time on zeolite properties

The effect of mixing time was studied by varying the time of mixing from 6 to 48 h using the constant Si/Al ratios of 2.32, fusion temperature 550°C, fusion time 1 h, water 85 ml, crystallization temperature 90 °C, crystallization time 2 h. The effect of mixing time was studied at mixing times of 6, 12, 24, and 48 h. From the XRD pattern in Fig. 4.24, the zeolite was not formed unless mixing time was equal to 12 h or more. However, a much too long mixing time of 48 h was not beneficial for the formation of zeolite and there could be a formation of zeolite P as impurity. Crystallinity percentage was calculated compared with standard zeolie X and shown in Fig. 4.25. As seen from the figure, the mixing time of 12 h was the optimum as it gave the highest crystallinity percentage, followed by 24 and 48 h (although the % crystallinity was quite close at these three mixing times).

Table 4.1 Results of XRF analysis of starting material

Compound	% CONCENTRATION			
	SAMPLE			
	1	2	3	4
Na	0.89	0.99	0.69	0.72
Mg	0.65	0.56	0.55	0.55
Al	8.28	5.76	5.40	7.90
Si	19.1	24.3	25.97	19.10
K	2.57	2.78	3.54	2.65
Ca	3.29	4.12	3.45	3.26
Ti	0.67	0.27	0.35	0.56
Mn	0.12	0.14	0.21	0.14
Fe	8.90	6.86	6.39	8.50
Rb	0.04	0.03	0.03	0.30
Sr	0.16	0.20	0.15	0.16
P	0.20	0.25	0.31	0.24
S	0.75	0.41	0.44	0.69
Zn	0.03			
Si/Al	2.29	4.05	4.62	2.32

Table 4.2 Reaction conditions, cation exchange capacity(CEC), and % crystallinity

Si/Al Ratio	NaOH concentration	Fusion		Mixing			Crystallization		CEC (meq/100g)	Percent Crystallization
		Temp (°C)	Time (min)	Temp (°C)	Time (h)	Water (ml)	Temp (°C)	Time (h)		
Effect of NaOH concentration										
	0	550	60	30	24	85	90	2	12.5	0.00
	5	550	60	30	24	85	90	2	31.5	30.2
	10	550	60	30	24	85	90	2	63.5	50.4
	12.5	550	60	30	24	85	90	2	100	74.2
	15	550	60	30	24	85	90	2	111	87.9
1.71	17.5	550	60	30	24	85	90	2	128	89.9
	20	550	60	30	24	85	90	2	130	89.8
	22.5	550	60	30	24	85	90	2	153	100
	25	550	60	30	24	85	90	2	141	89.2
	30	550	60	30	24	85	90	2	75.0	30.0
Effect of a mount of water										
2.32	17.5	550	60	30	24	65	90	2	289	99.2
	17.5	550	60	30	24	85	90	2	279	89.3
	17.5	550	60	30	24	115	90	2	257	28.9
Effect of mixing temperature										
2.32	17.5	550	60	30	24	85	90	2	279	65.8
	17.5	550	60	60	24	85	90	2	274	54.0
	17.5	550	60	90	24	85	90	2	220	54.0
Effect of mixing time										
2.32	17.5	550	60	30	6	85	90	2	251	0.00
	17.5	550	60	30	12	85	90	2	303	32.6
	17.5	550	60	30	24	85	90	2	279	32.5
	17.5	550	60	30	48	85	90	2	193	26.3

Table 4.2 (Cont) Reaction conditions, cation exchange capacity(CEC) , and % crystallinity

Si/Al Ratio	NaOH concentration	Fusion		Mixing			Crystallization		CEC (meq/100g)	Percent Crystallization
		Temp (°C)	Time (min)	Temp (°C)	Time (h)	Water (ml)	Temperature (°C)	Time (h)		
Effect of fusion temperature										
2.29	17.5	250	60	30	12	85	90	2	222	37.4
	17.5	350	60	30	12	85	90	2	229	44.4
	17.5	450	60	30	12	85	90	2	265	95.0
	17.5	550	60	30	12	85	90	2	250	91.0
4.05	17.5	250	60	30	12	85	90	2	205	40.7
	17.5	350	60	30	12	85	90	2	223	43.5
	17.5	450	60	30	12	85	90	2	253	59.1
	17.5	550	60	30	12	85	90	2	240	56.2
4.62	17.5	250	60	30	12	85	90	2	193	42.7
	17.5	350	60	30	12	85	90	2	221	43.3
	17.5	450	60	30	12	85	90	2	238	56.2
	17.5	550	60	30	12	85	90	2	221	56.2
Effect of fusion time										
2.29	17.5	550	15	30	12	85	90	2	248	30.4
	17.5	550	30	30	12	85	90	2	253	44.5
	17.5	550	45	30	12	85	90	2	297	92.1
	17.5	550	60	30	12	85	90	2	250	91.0
4.05	17.5	550	15	30	12	85	90	2	241	30.4
	17.5	550	30	30	12	85	90	2	262	48.2
	17.5	550	45	30	12	85	90	2	288	60.5
	17.5	550	60	30	12	85	90	2	240	56.2
4.62	17.5	550	15	30	12	85	90	2	238	30.4
	17.5	550	30	30	12	85	90	2	255	44.5
	17.5	550	45	30	12	85	90	2	277	60.2
	17.5	550	60	30	12	85	90	2	221	56.2

Table 4.2 (Cont) Reaction conditions, cation exchange capacity(CEC), and % crystallinity

Si/Al Ratio	NaOH concentration	Fusion		Mixing		Water (ml)	Crystallization		CEC (meq/100)	Percent Crystallization
		Temp (°C)	Time (min)	Temp (°C)	Time (h)		Temperature (°C)	Time (h)		
Effect of crystallization temperature										
2.29	17.5	550	60	30	12	85	30	2	180	0.00
	17.5	550	60	30	12	85	60	2	201	0.00
	17.5	550	60	30	12	85	90	2	250	91.0
4.05	17.5	550	60	30	12	85	30	2	177	0.00
	17.5	550	60	30	12	85	60	2	180	0.00
	17.5	550	60	30	12	85	90	2	240	56.2
4.62	17.5	550	60	30	12	85	30	2	188	0.00
	17.5	550	60	30	12	85	60	2	197	0.00
	17.5	550	60	30	12	85	90	2	221	56.2
Effect of crystallization time										
2.29	17.5	550	60	30	12	85	90	1	210	12.2
	17.5	550	60	30	12	85	90	2	250	91.0
	17.5	550	60	30	12	85	90	3	220	79.6
4.05	17.5	550	60	30	12	85	90	1	183	8.40
	17.5	550	60	30	12	85	90	2	240	56.2
	17.5	550	60	30	12	85	90	3	199	46.4
4.62	17.5	550	60	30	12	85	90	1	177	0.80
	17.5	550	60	30	12	85	90	2	221	56.2
	17.5	550	60	30	12	85	90	2	191	49.2

Acc.V Spot Magn Det WD Exp   10 µm
12.0 kV 4.0 2000x SE 14.5 1   UC 0.3
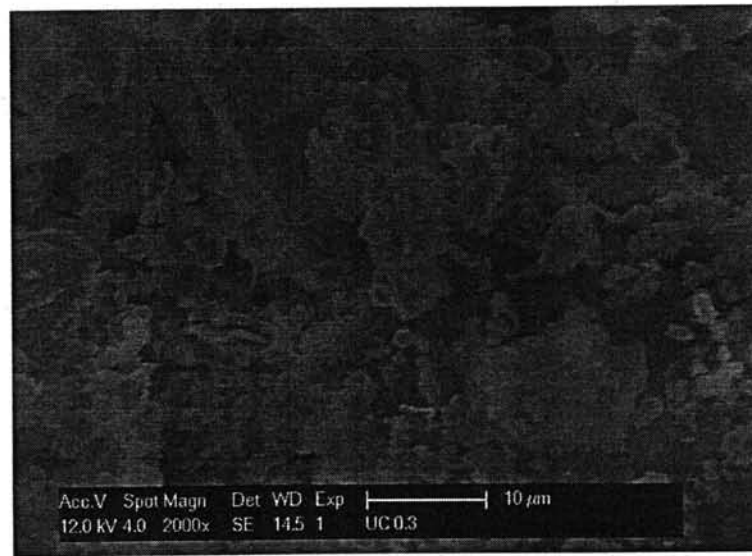
Figure 4.1 SEM of CFA
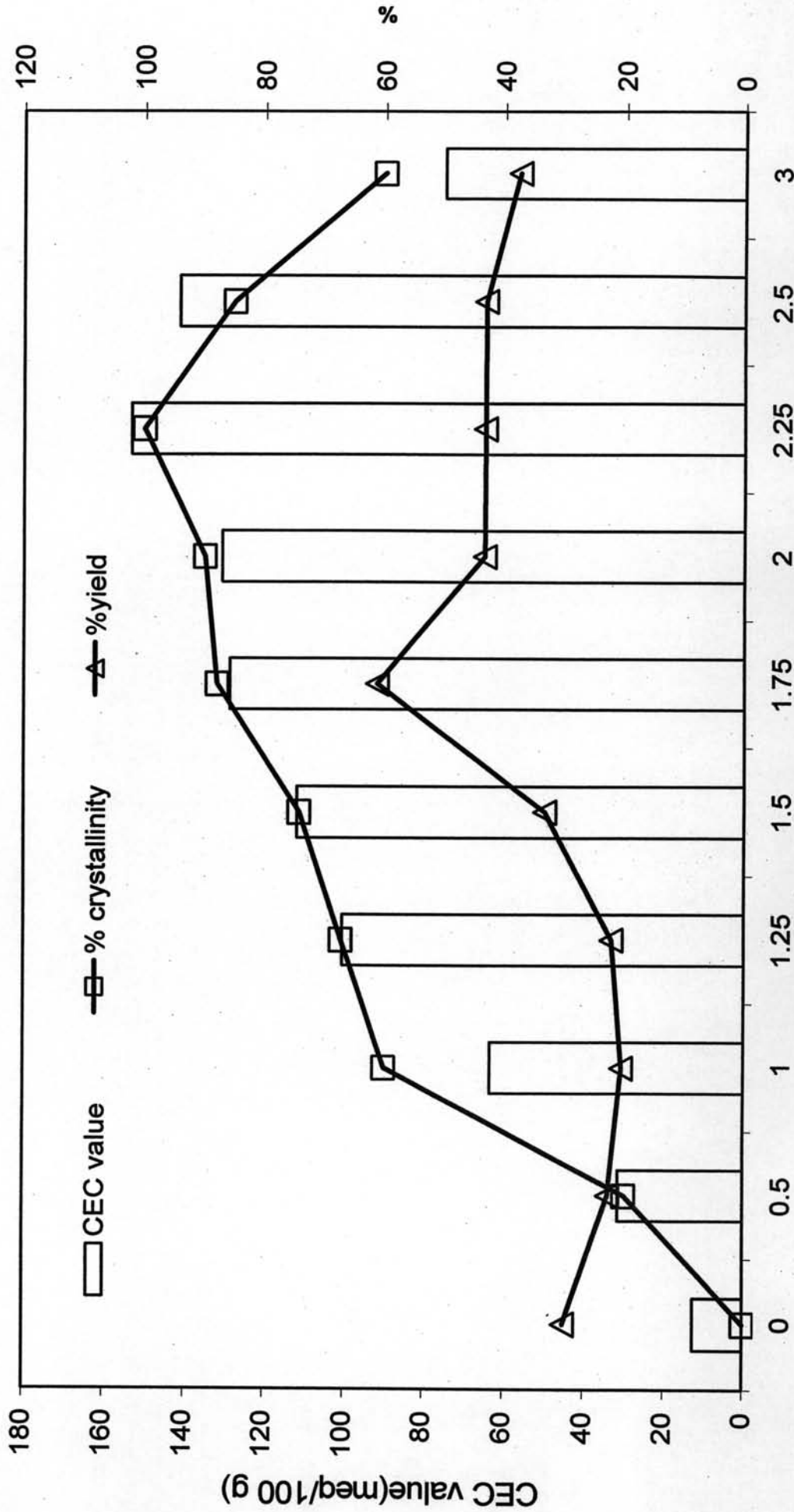
Figure 4.2 Effect of NaOH/ CFA weight ratio on CEC and % Crystallinity of the zeolite
(Fusion temperature 550 °C at 1 h, Water 85 ml, Mixing temperature 30 °C at 12 h, Crystallization temperature 90 °C at 2 h)
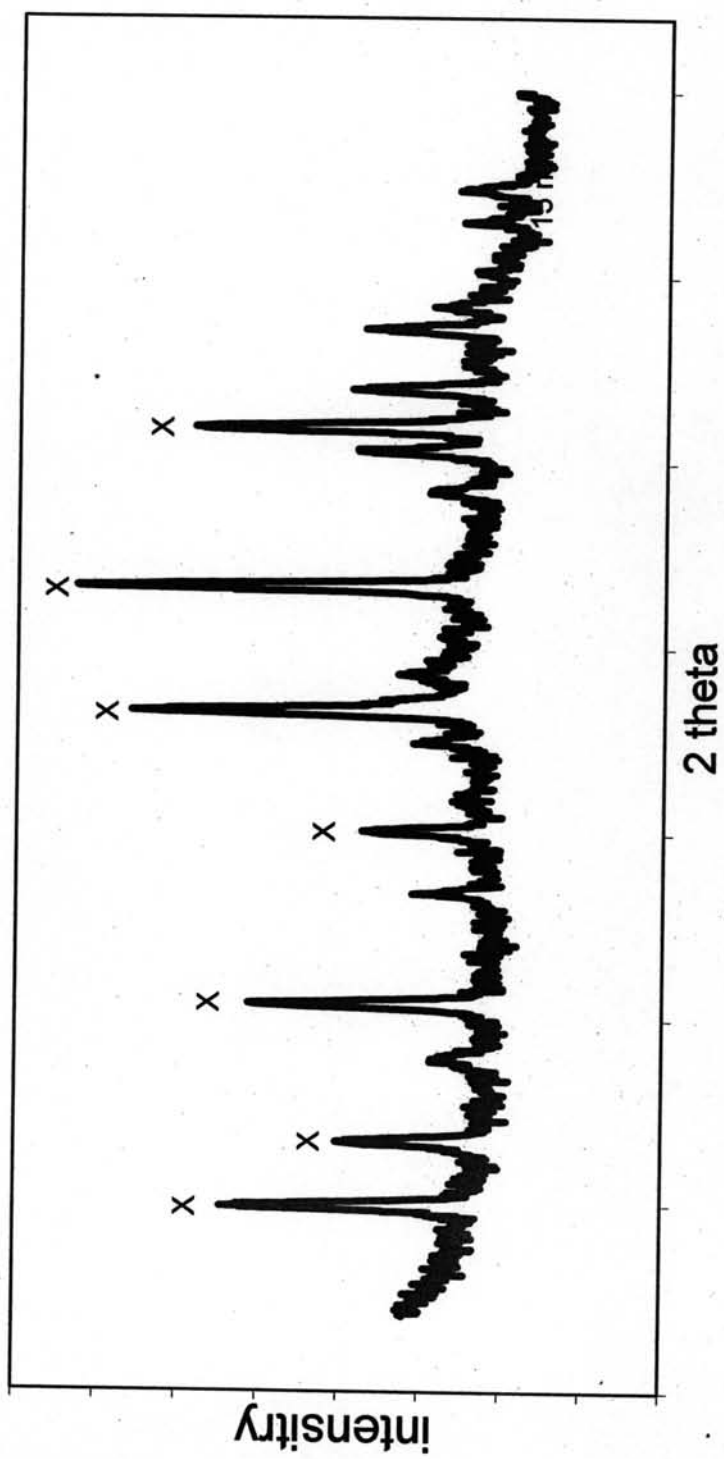
Figure 4.3 XRD of zeolite X Standard
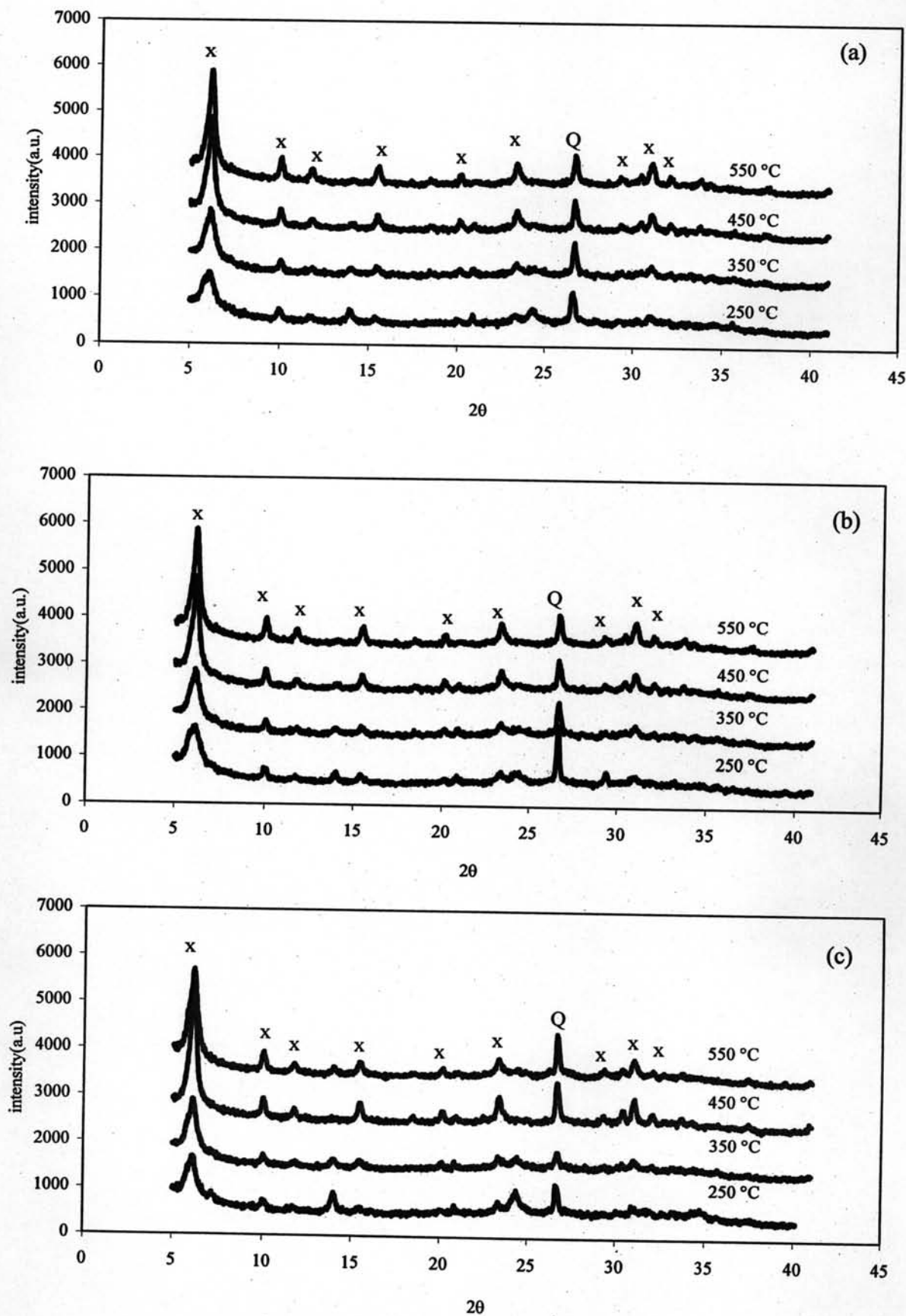
X = Zeolite X and Q = quartz

Figure 4.4 XRD patterns for zeolite products obtained with different fusion temperature
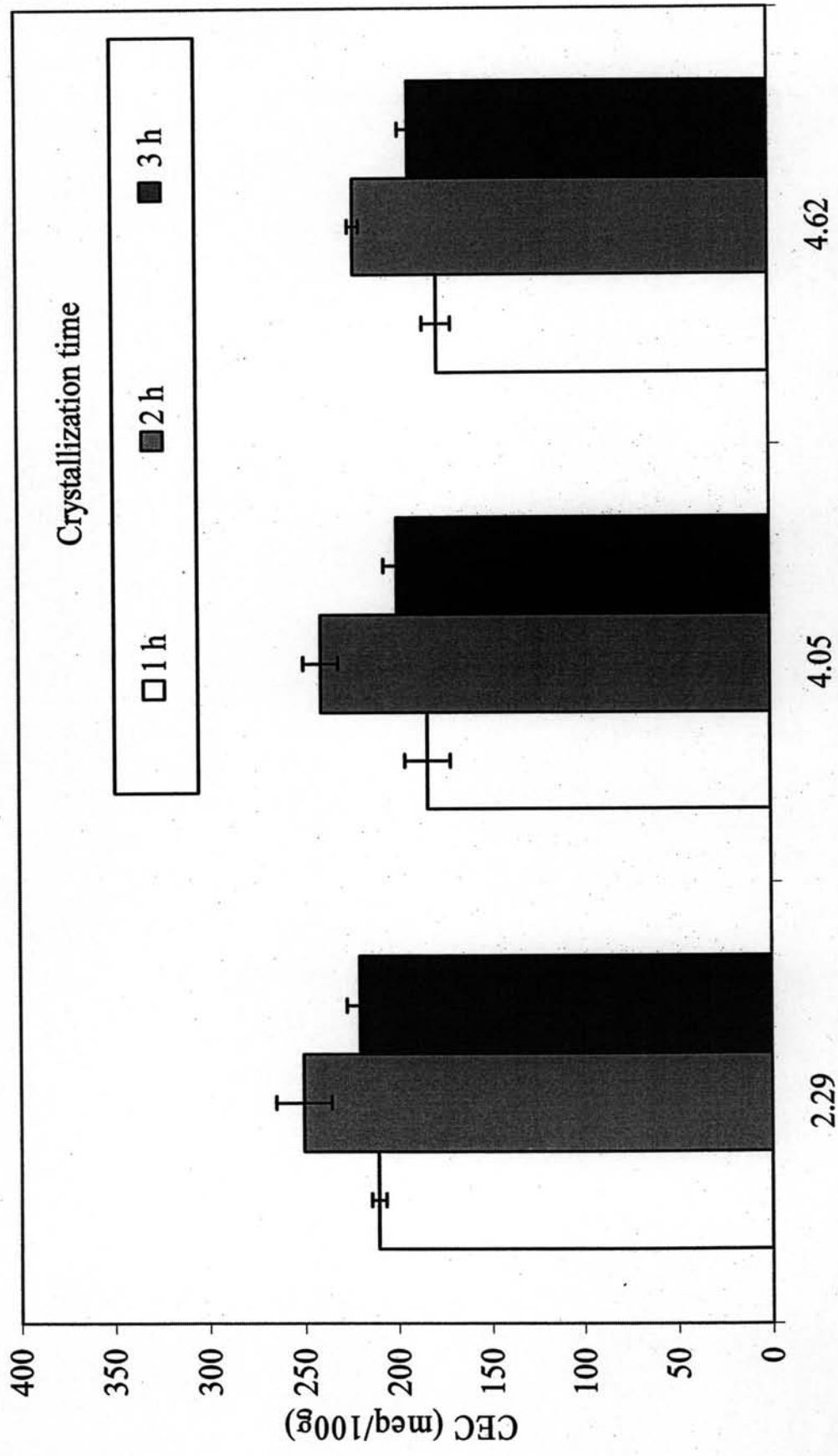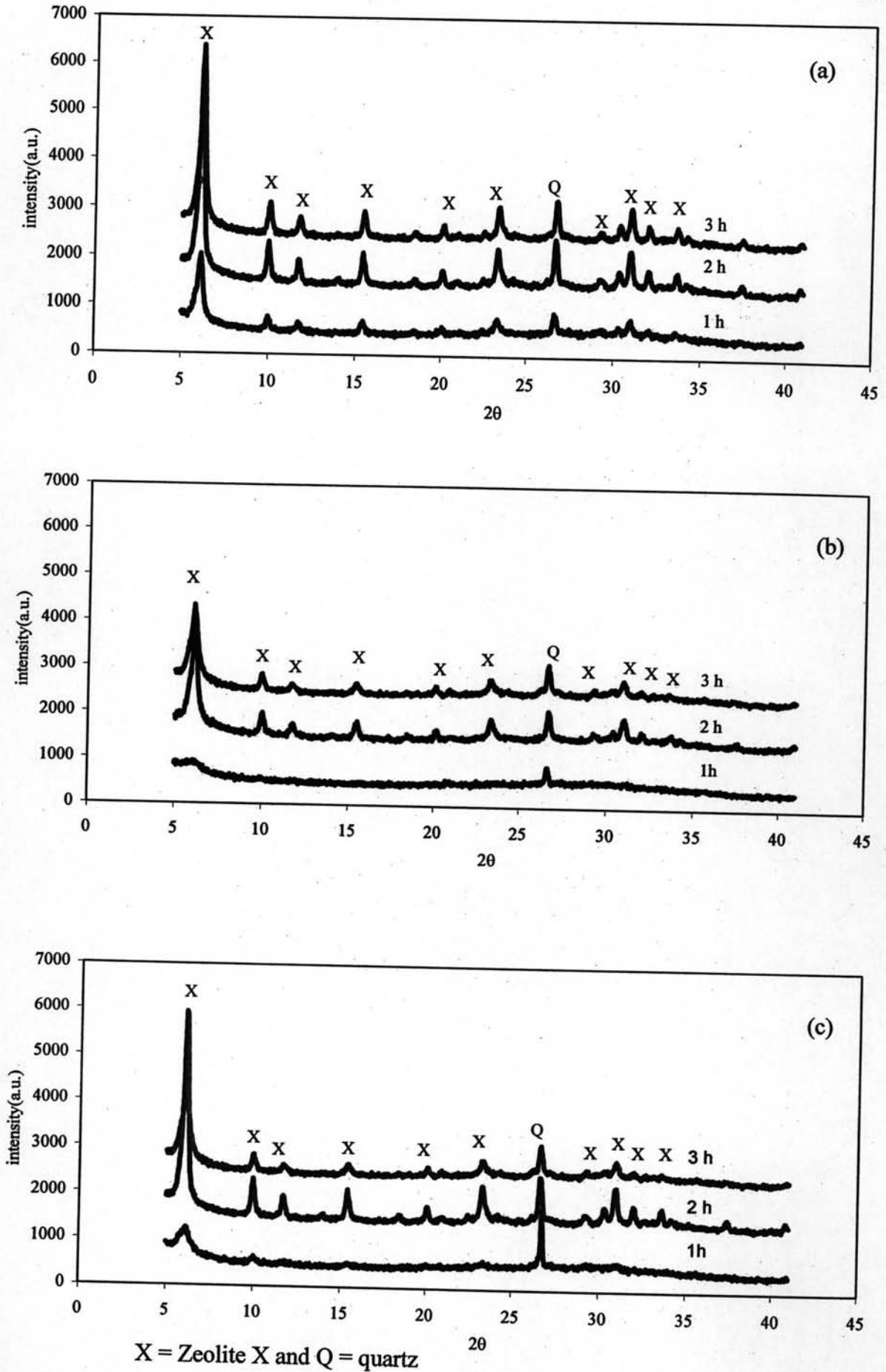(a) Si/Al = 2.29 (b) Si/Al = 4.05 Si/Al = 4.62
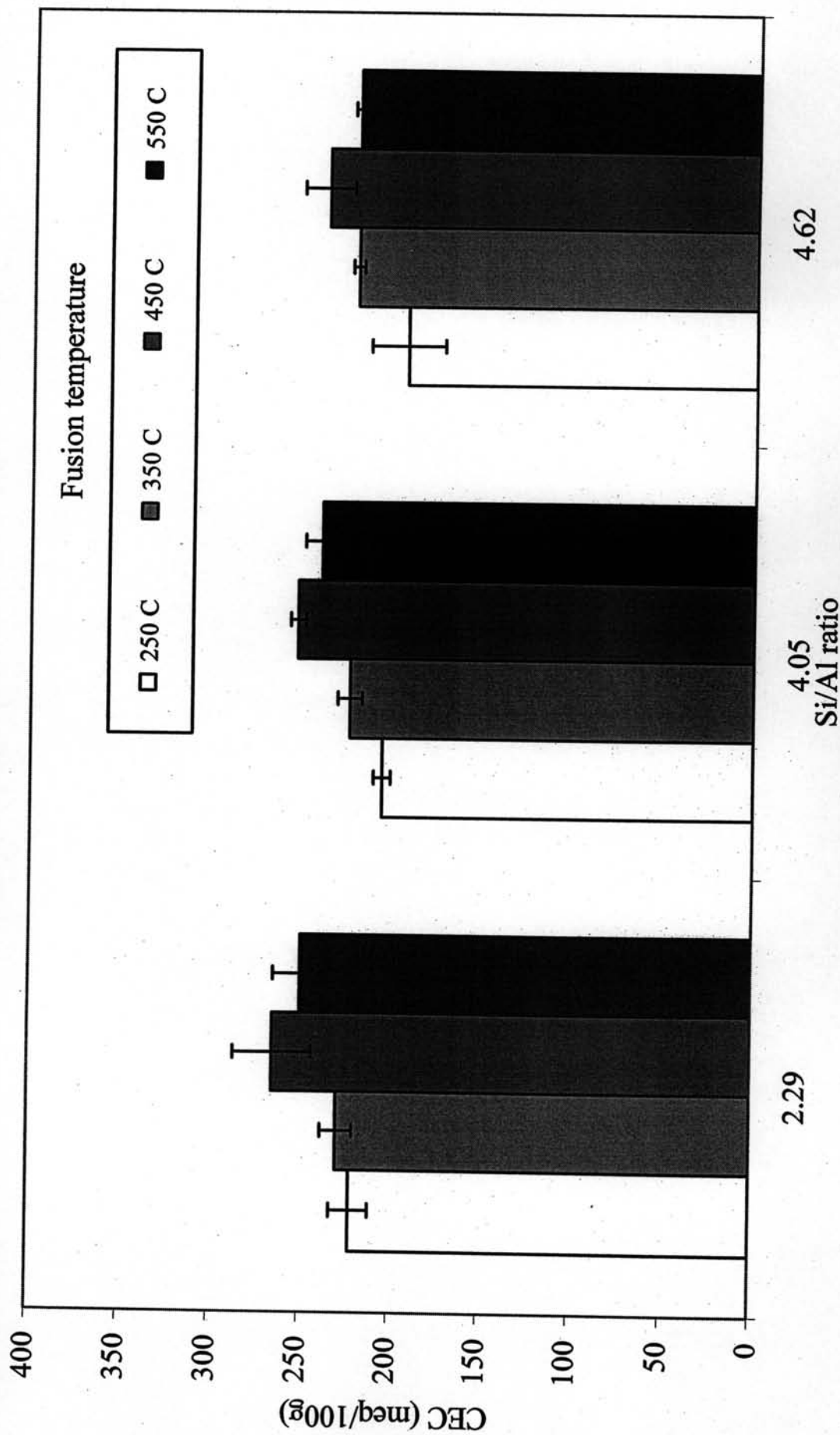
Figure 4.5 Effect of fusion temperature on CEC value

(Fusion time 2 h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C at 2 h)
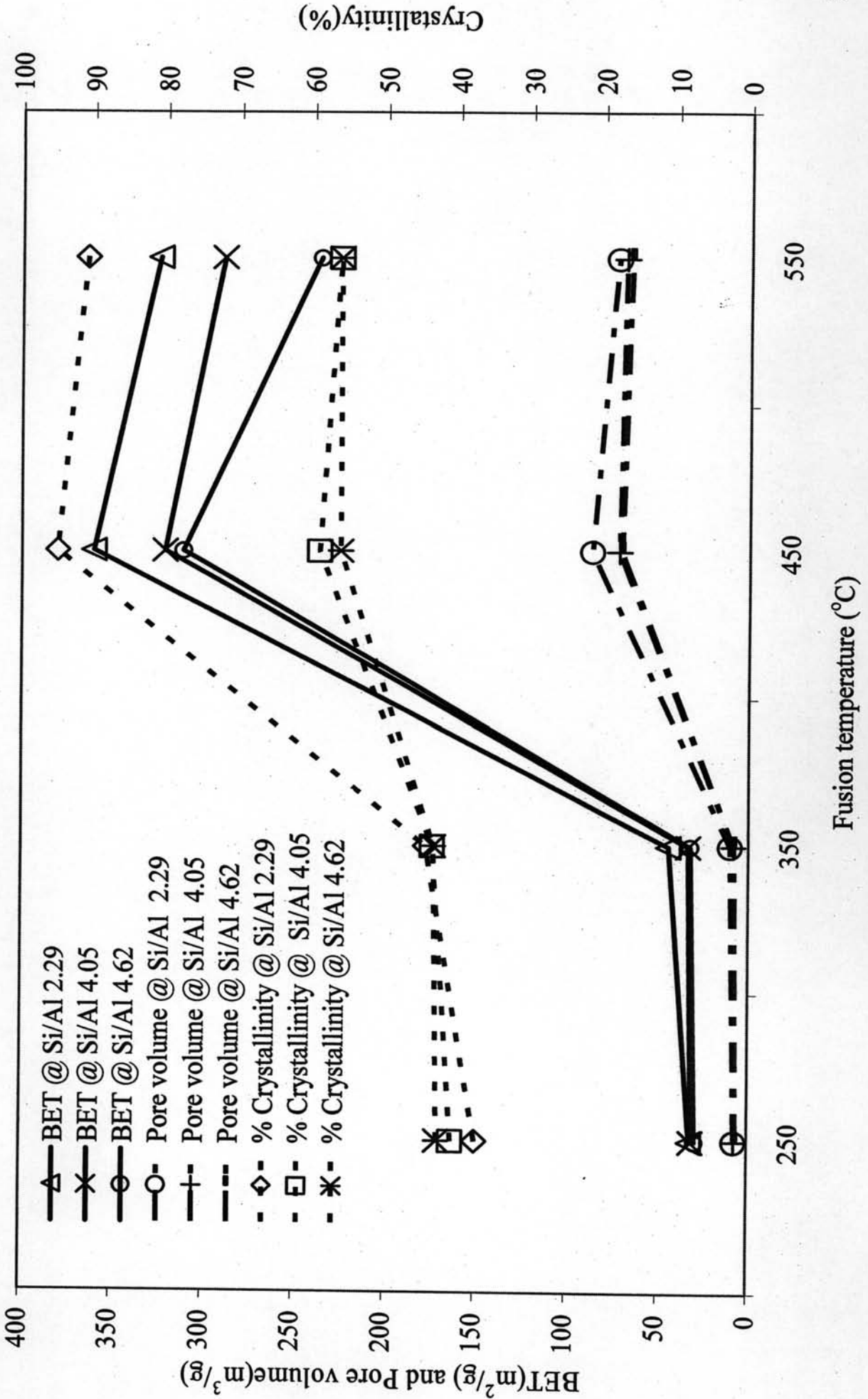
Figure 4.6 Effect of fusion temperature on properties of zeolite

(Fusion time 1 h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C at 2 h)
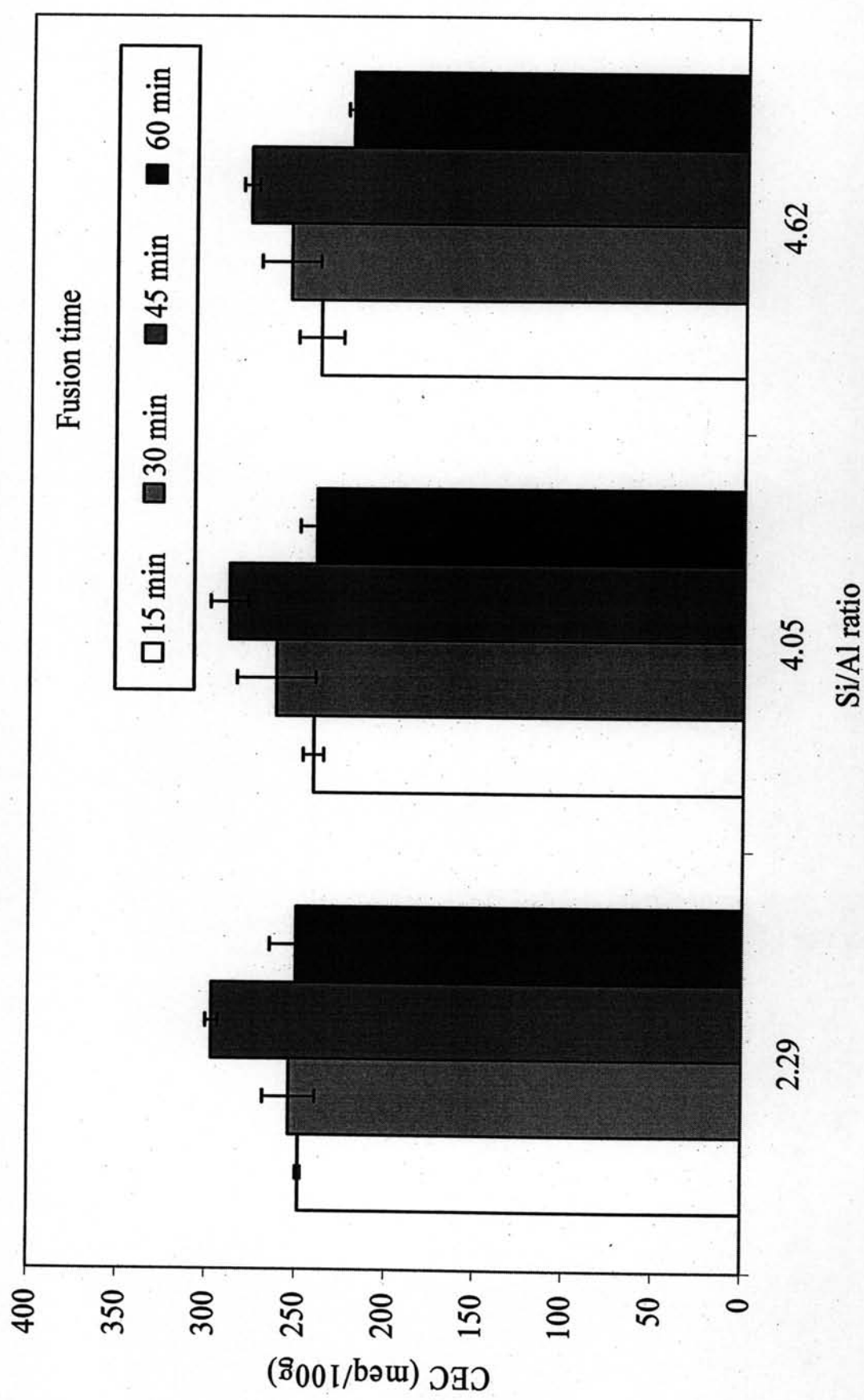
Figure 4.7 Effect of fusion time on CEC value
(Fusion temperature 550°C, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C at 2 h)
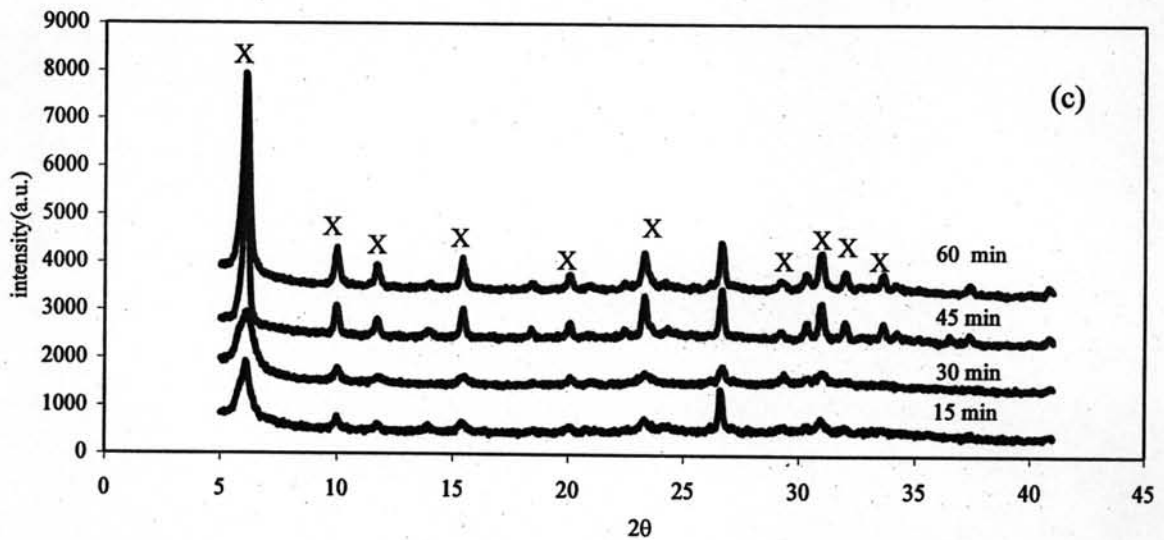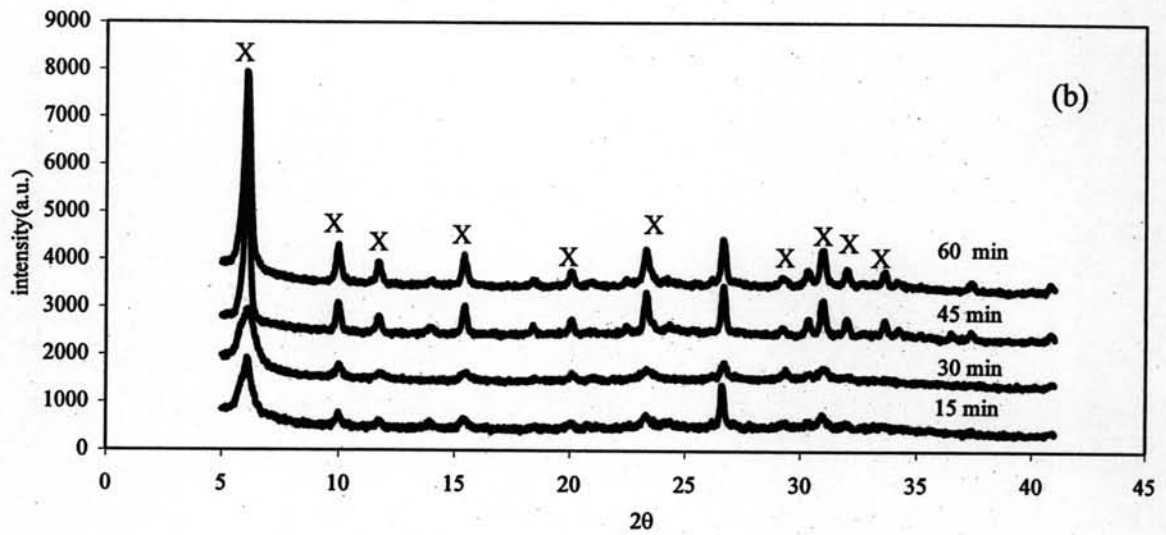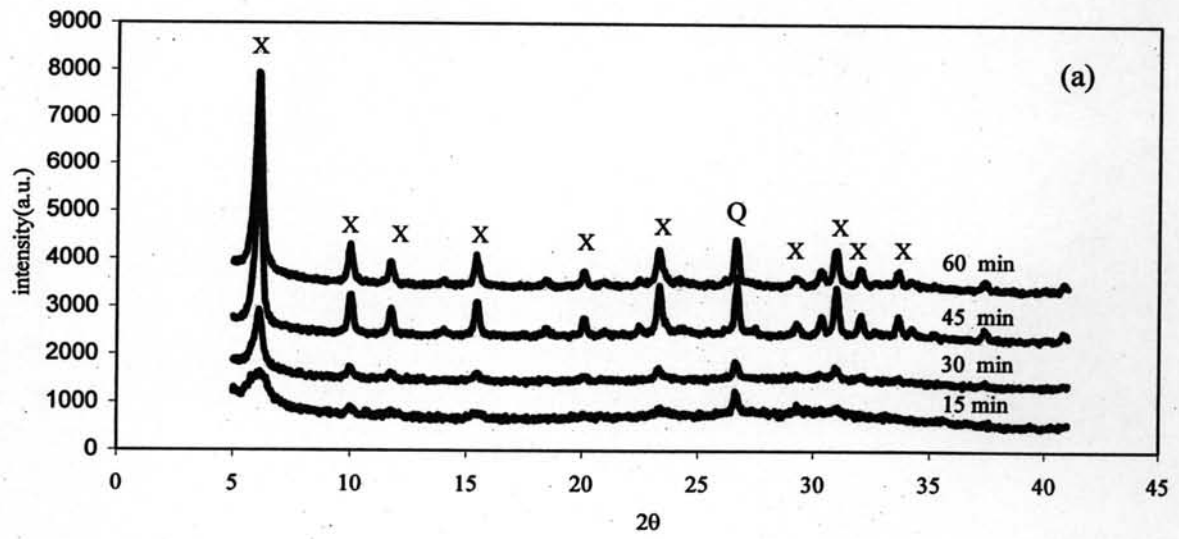
X = Zeolite X and Q = quartz

Figure 4.8 XRD patterns for zeolite products obtained with different fusion time
(a) Si/Al = 2.29 (b) Si/Al = 4.05 (c)Si/Al = 4.62
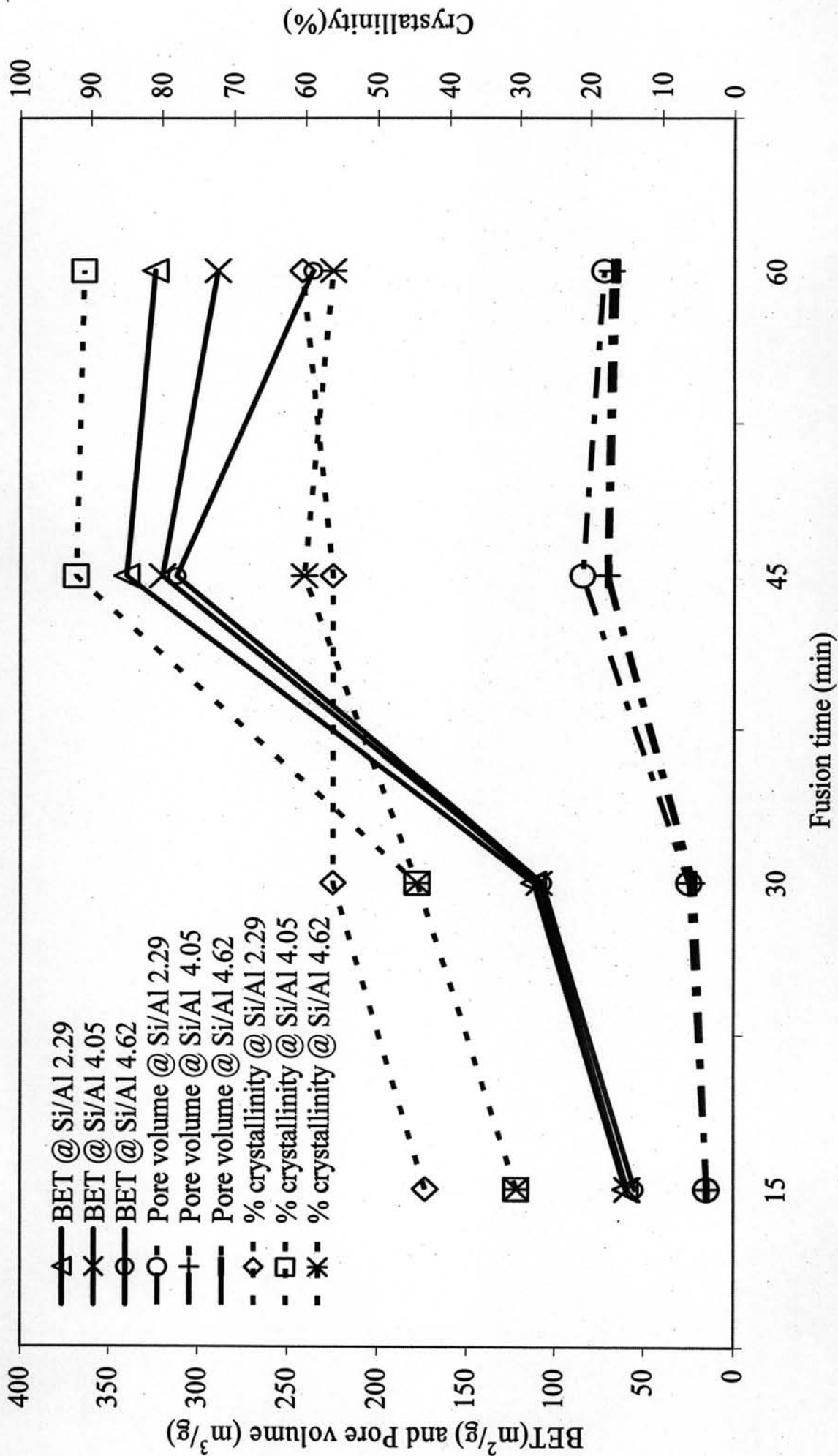
Figure 4.9 Effect of fusion time on zeolite properties

(Fusion temperature 550°C, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C at 2 h)
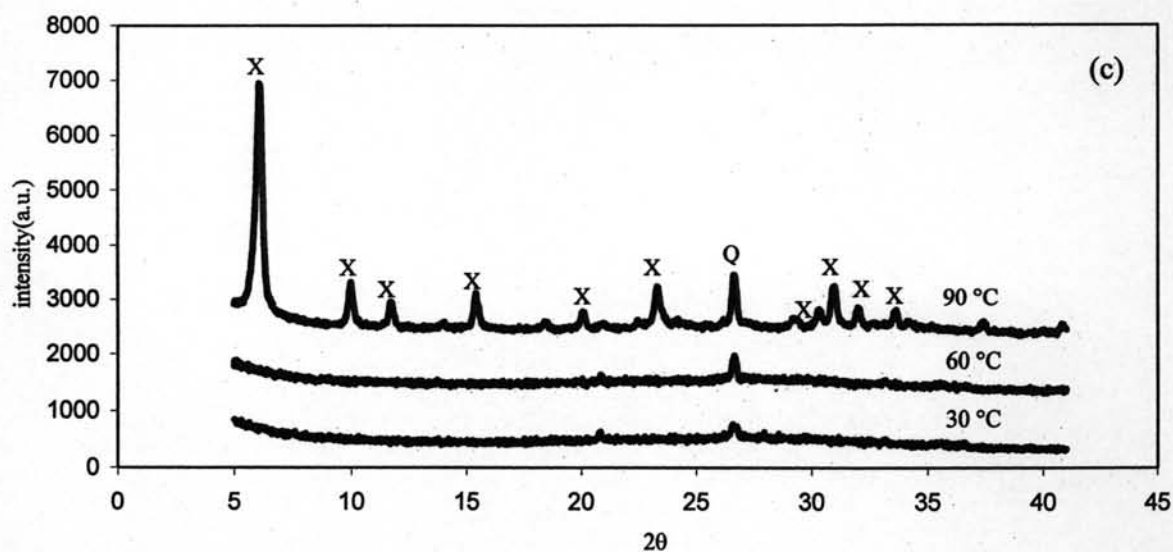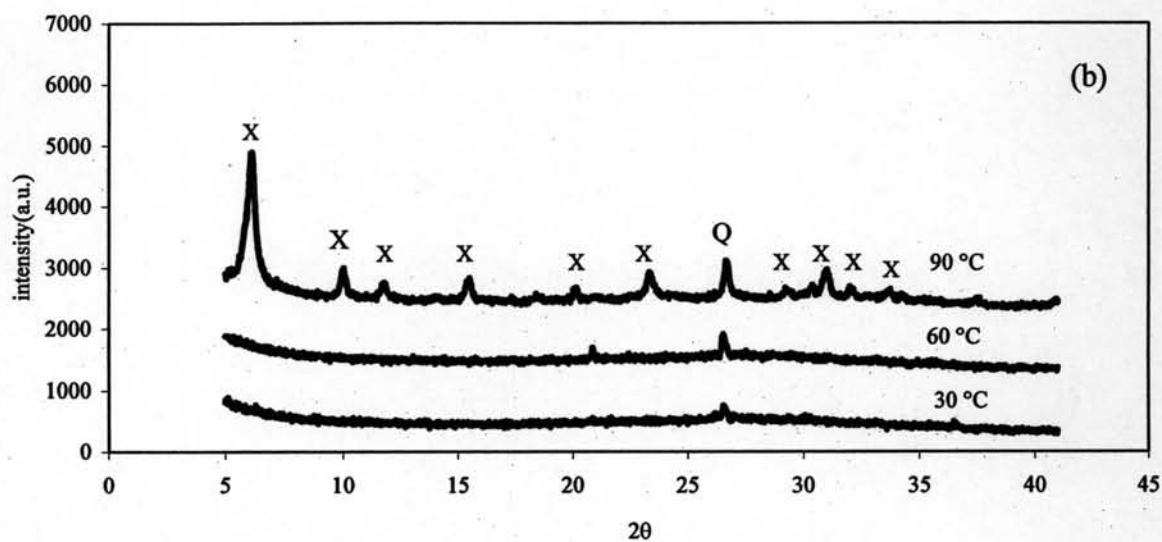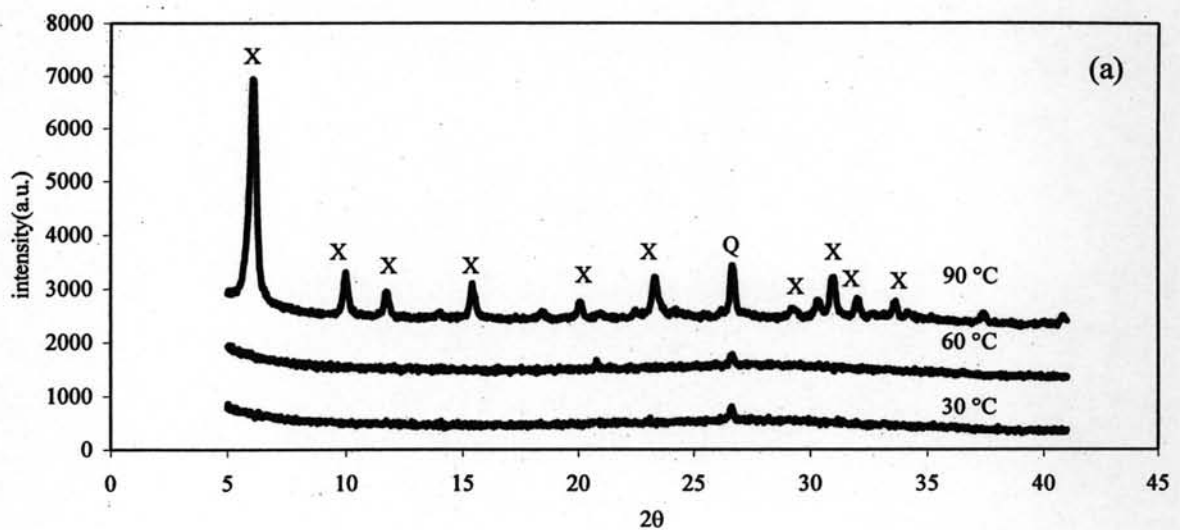
X = Zeolite X and Q = quartz

Figure 4.10 XRD patterns for zeolite products obtained with different crystallization temperature
(a) Si/Al = 2.29 (b) Si/Al = 4.05 (c)Si/Al = 4.62
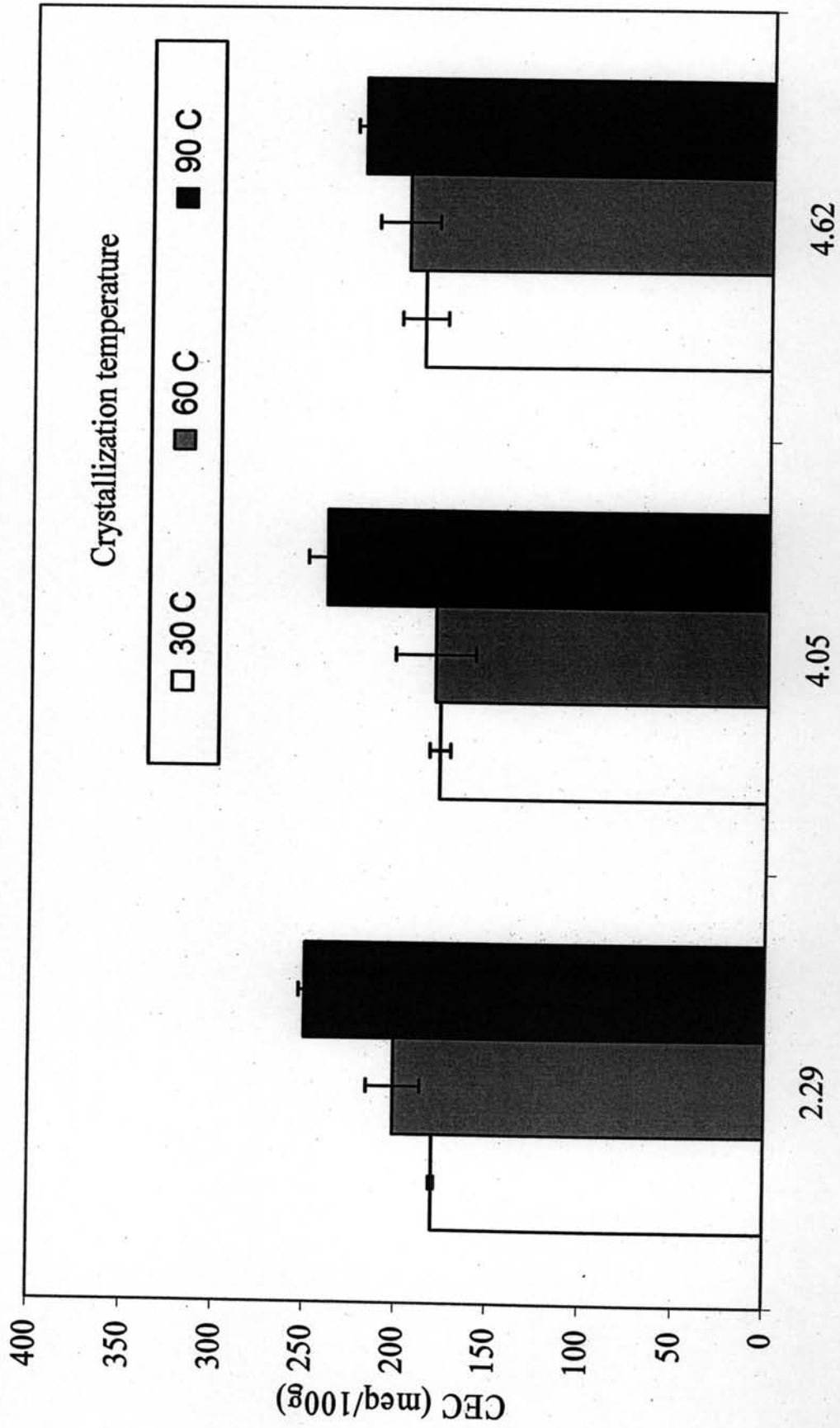
Figure 4.11 Effect of crystallization temperature on CEC value
(Fusion temperature 550°C at 2 h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization time 2 h)
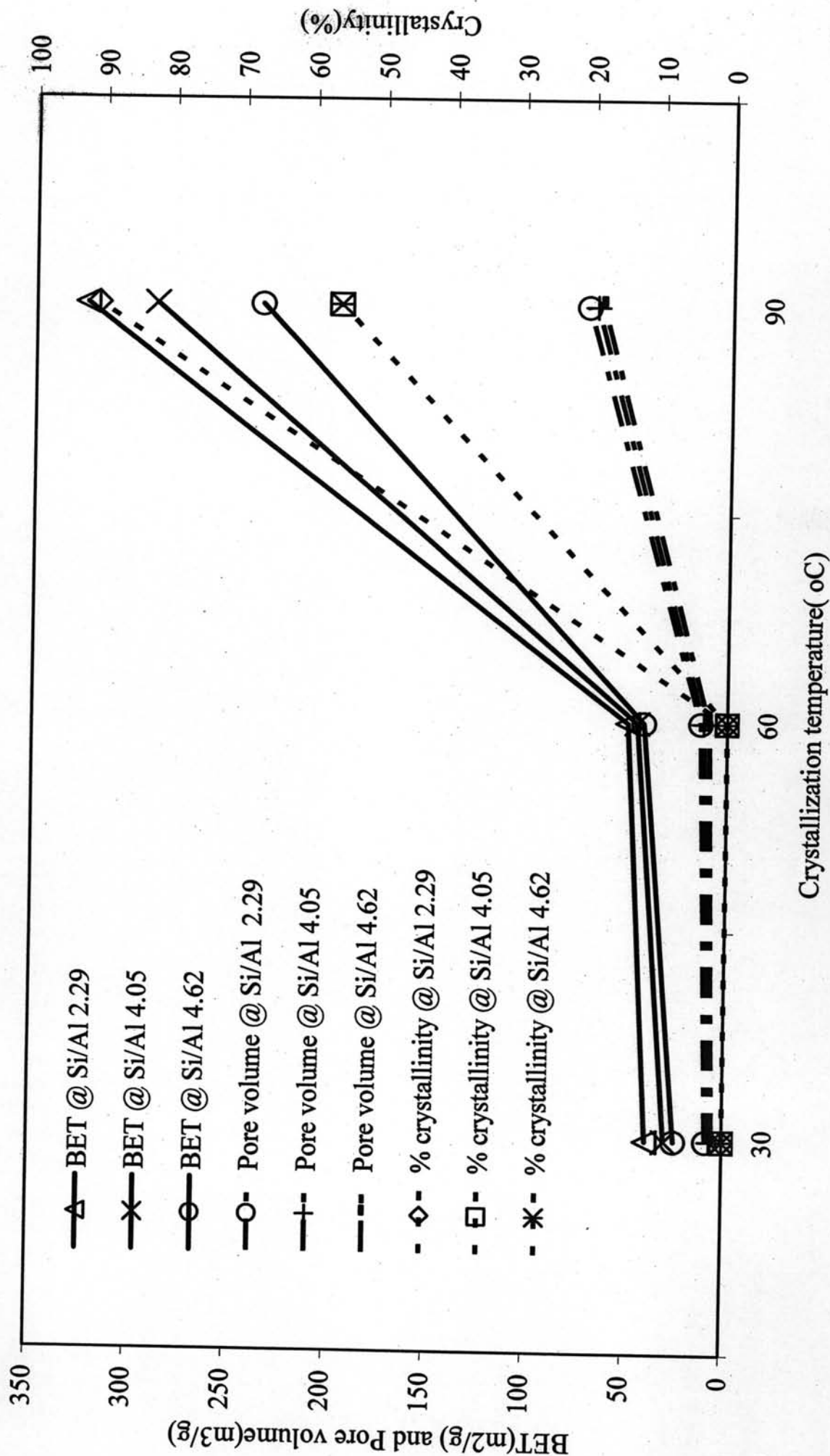
Figure 4.12 Effect of crystallization temperature on zeolite properties
(Fusion temperature 550°C at 1h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 2 h)

Figure 4.13 Effect of crystallization time on CEC value
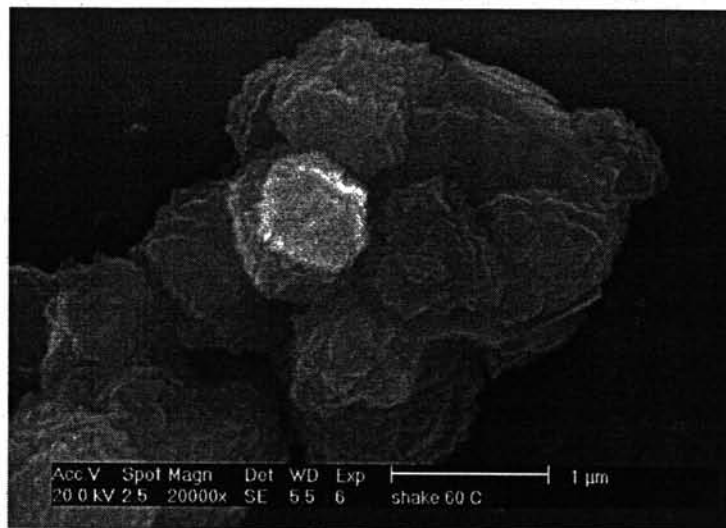(Fusion temperature 550°C at 2 h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C)

Figure 4.14 XRD patterns for zeolite products obtained with different crystallization time
(a) Si/Al = 2.29 (b) Si/Al = 4.05 (c)Si/Al = 4.62
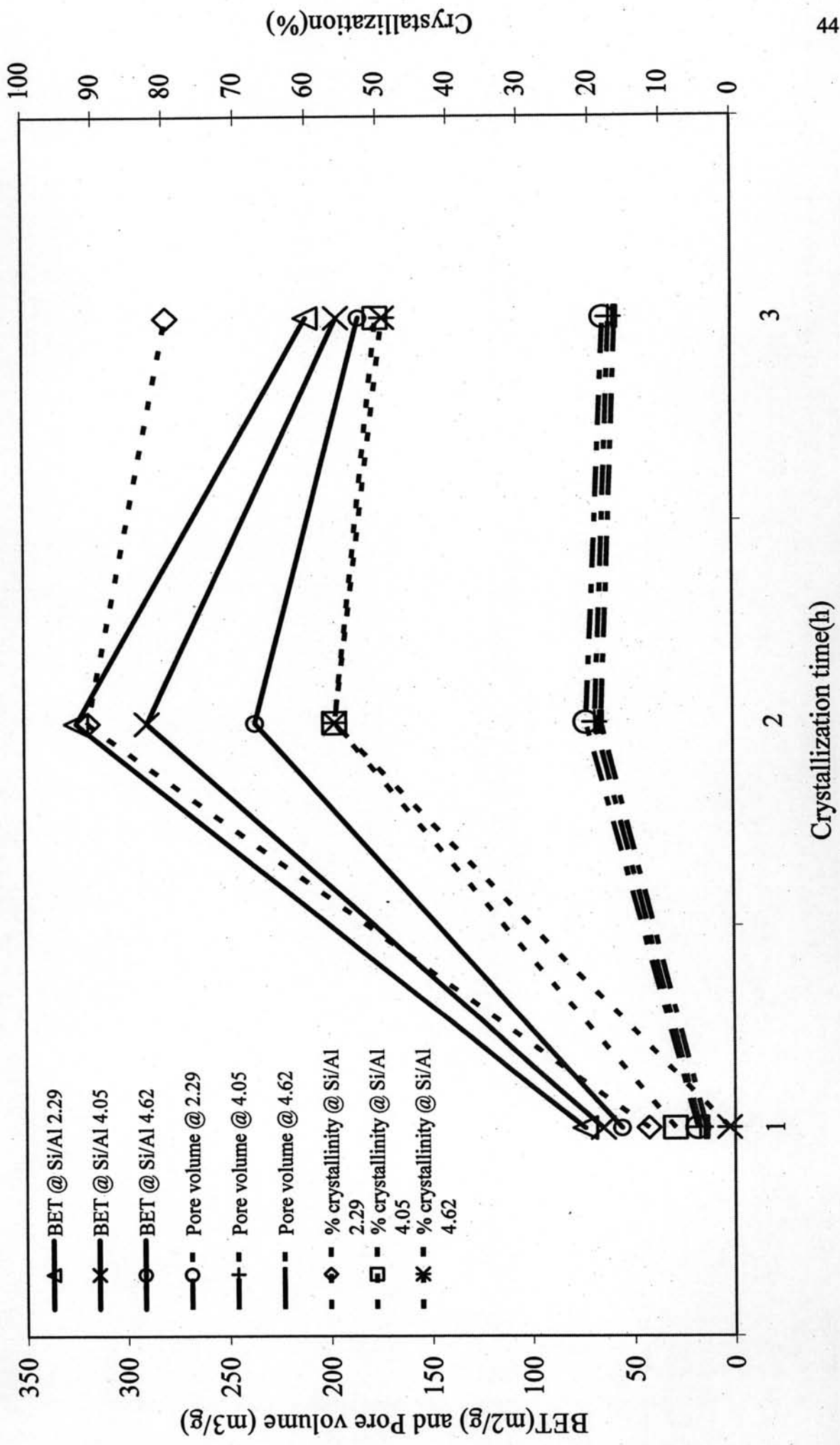
Figure 4.15 Effect of crystallization time on zeolite properties

(Fusion temperature 550°C at 1 h, Water 85 ml, Mixing temperature 30°C at 12 h, Crystallization temperature 90°C)
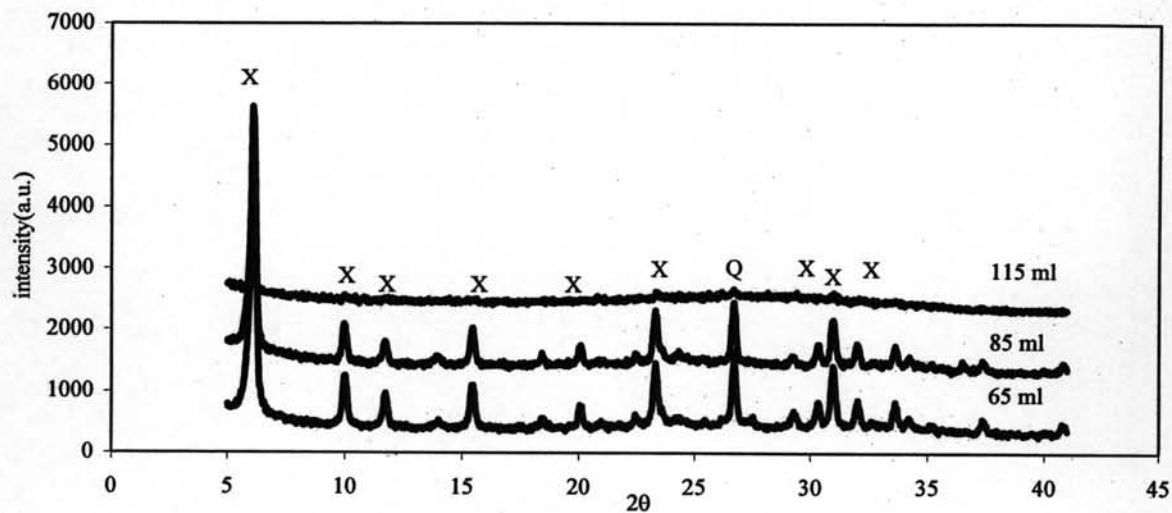
X =Zeolite X and Q = quatz

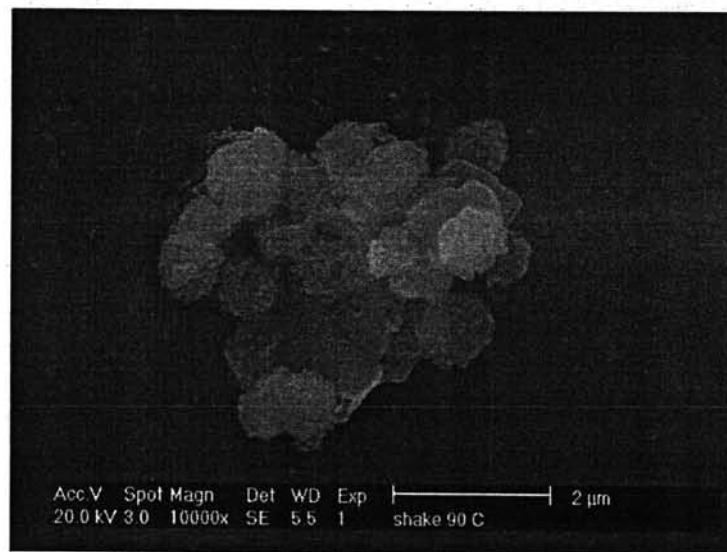Figure 4.16 XRD pattern for zeolite product obtained from different amount waters
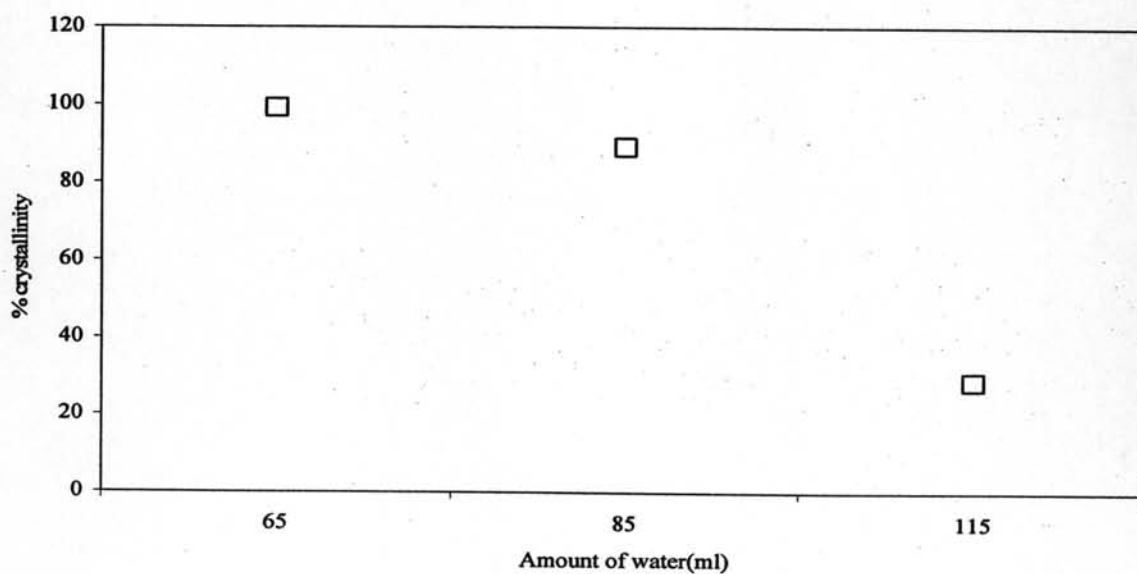
Figure 4.17 Effect of amount water on crystallinity percentage of zeolite product
(Fusion temperature 550 °C at 2 h, Mixing temperature 30 °C at 2 h,
Crystallization temperature 90 °C at 2)
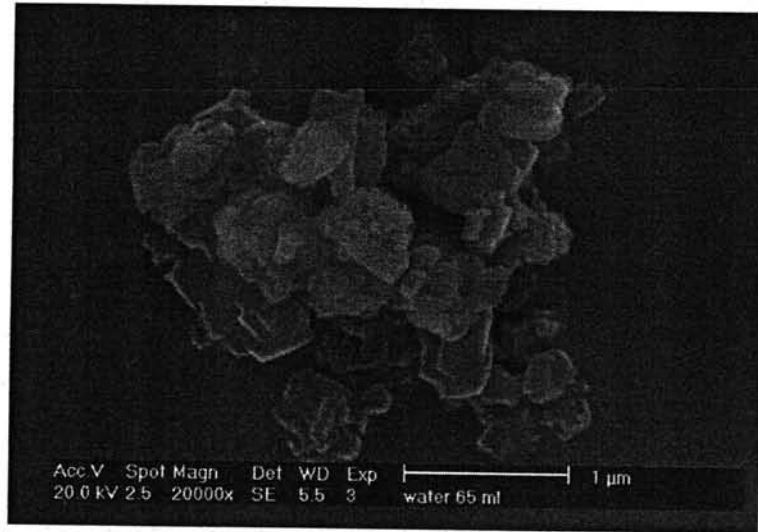
(a)
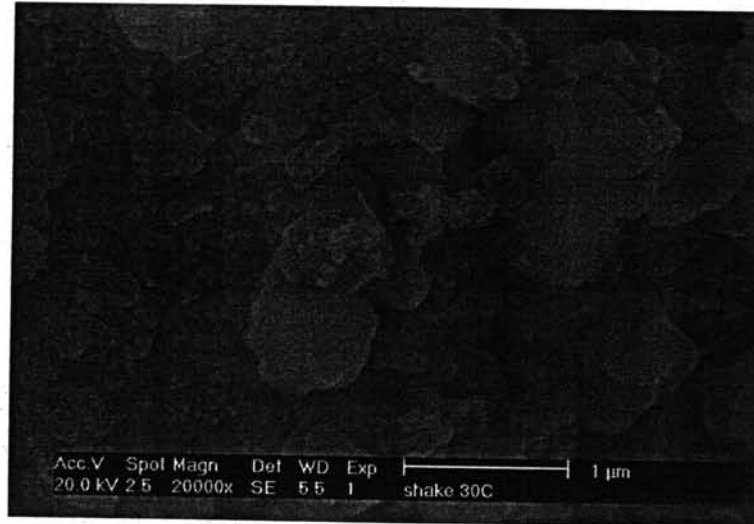
(b)

(c)

Figure 4.18 SEM of product prepared From different water contents
(a) water 65 ml (b) water 85 ml (c) water 115 ml
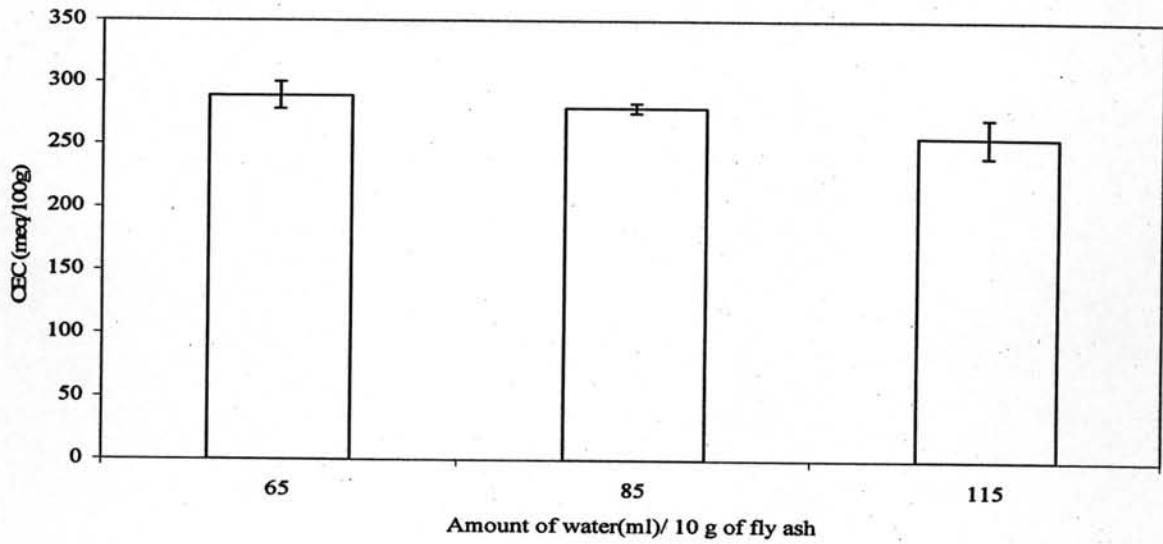
Figure 4.19 Effect of amount of water during crystallization period on CEC value
(Fusion temperature 550 °C at 2 h, Mixing temperature 30 °C at 12 h,
Crystallization temperature 90 °C at 2 h)
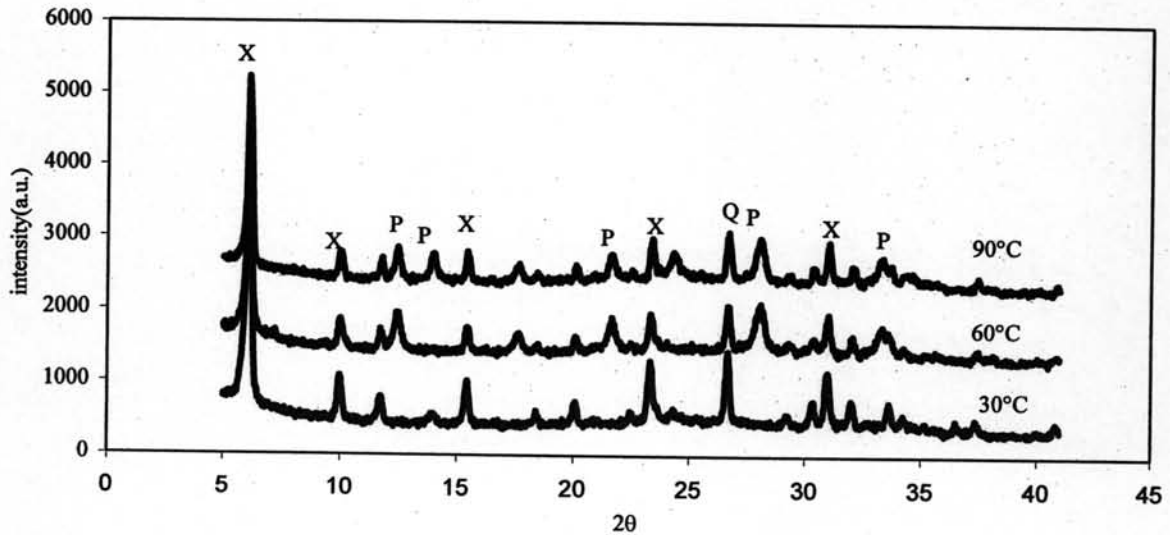
X = Zeolite X, P = Zeolite P, and Q = quatz

Figure 4.20 XRD patterns for zeolite products obtained from diffrent mixing
temperature

(a)

(b)

(c)

Figure 4.21 SEM of product prepared from different mixing temperature
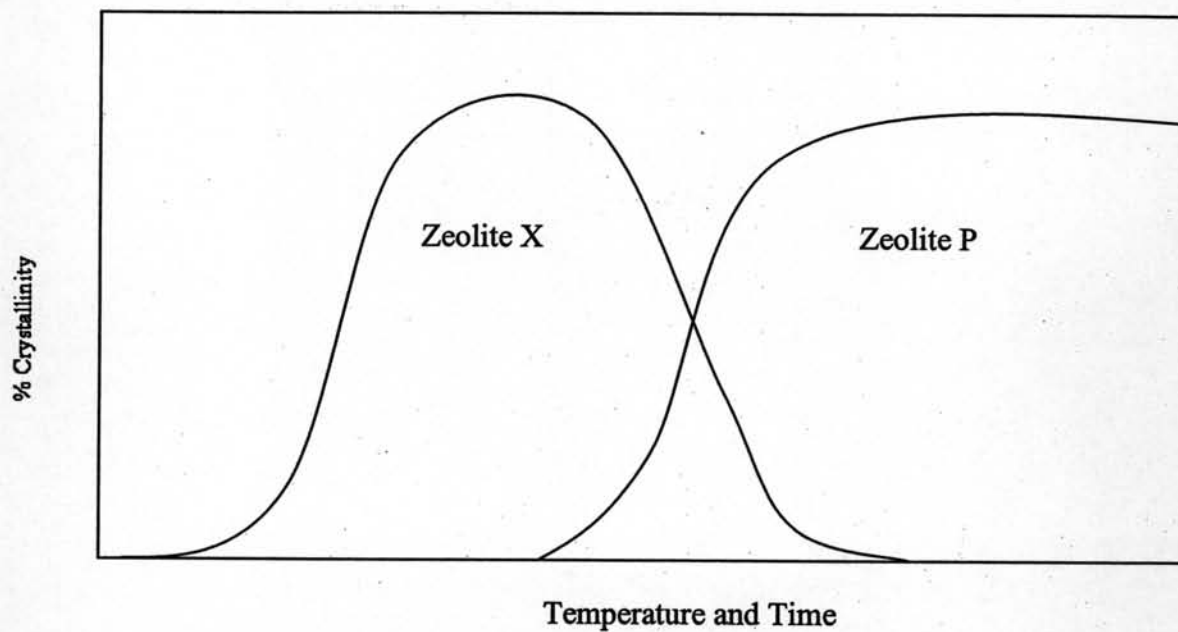(a) 30°C   (b) 60°C   (c) 90°C
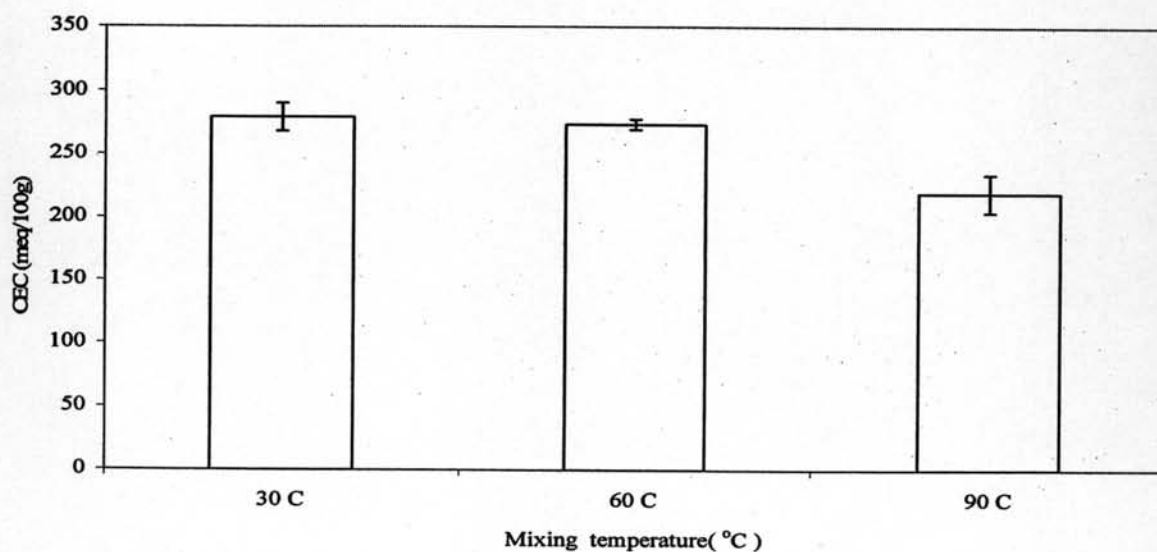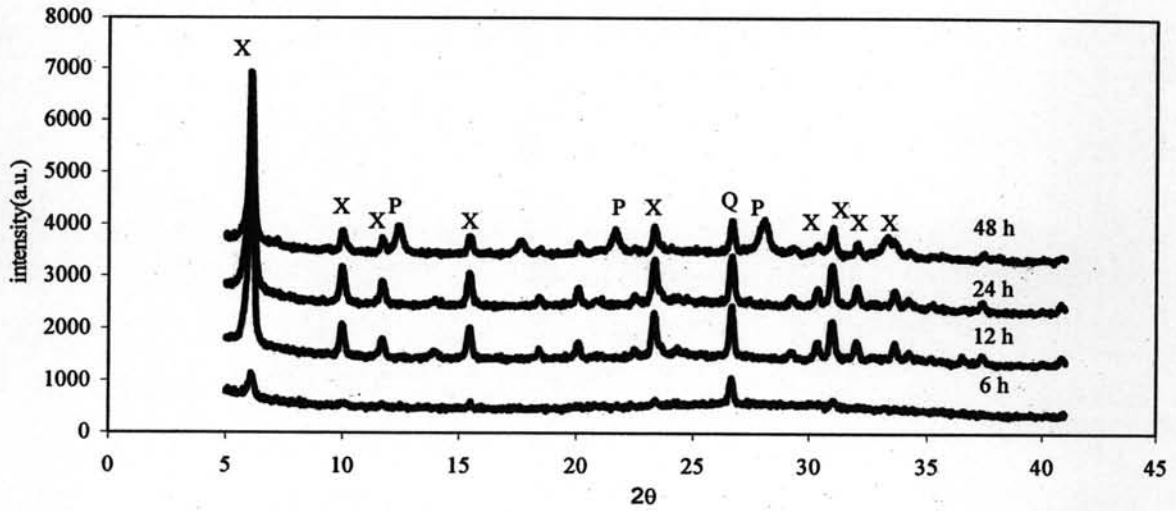
Figure 4.22 Zeolite X overlap Zeolite P

Figure 4.23 Effect of Mixing temperature period on CEC value
(Fusion temperature 550 °C at 2 h, Water 85 ml, Mixing time at 12 h,
Crystallization temperature 90 °C at 2 h)

X = Zeolite X, P = Zeolite P,and Q = quatz

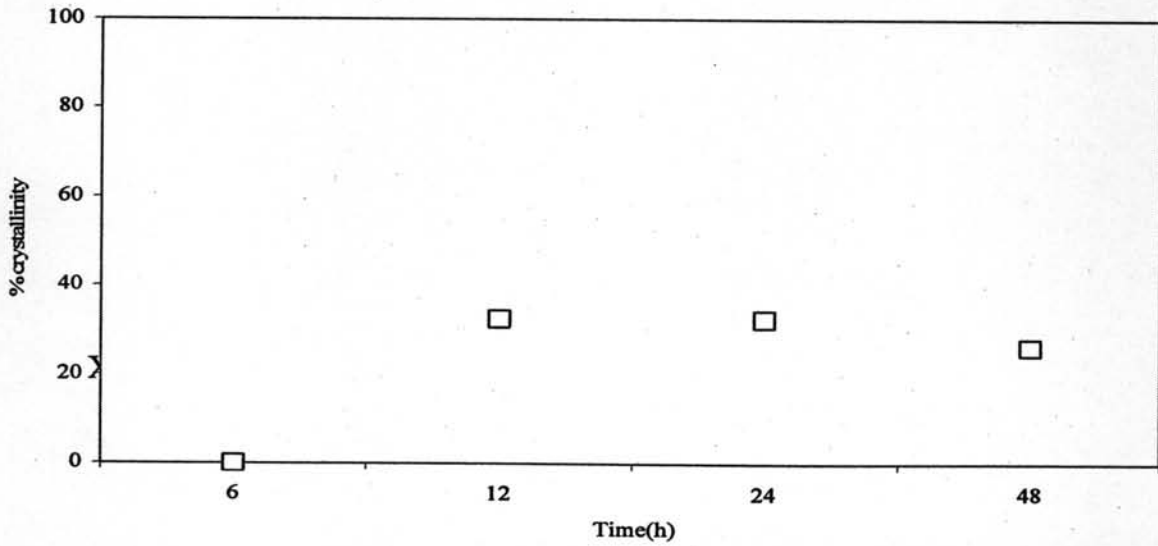Figure 4.24 XRD pattern for zeolite products obtained with different mixing time

Figure 4.25 Effect of mixing time on crystallinity percentage of zeolite product
(Fusion temperature 550 °C at 2 h, Mixing temperature 30 °C,
Crystallization temperature 90 °C at 2)