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APPENDIX

A-1 Caculation of percent Crystallinity

For calculation of percent crystallinity, Select 5 order of intensity of XRD pattern

$$\%\text{crystallinity} = \frac{\sum \text{intensity of XRD of peak of product}}{\sum \text{intensity of XRD peak of product(Standrad)}} \times 100$$

Sum of intensity Standard zeolite X

Angle 2-theta	d value Angstrom	Intensity count
6.08968	14.50181	5570.0
9.99126	8.84591	1260.0
15.43613	5.73573	1110.0
23.32704	3.81028	1424.0
30.97054	2.88512	1365.0
Sum of intensity		10642

Sum of intensity zeolite X (Si/Al 4.05, fusion temperature 250 °C)

Angle 2-theta	d value Angstrom	Intensity count
6.1257	14.41457	1636
9.95851	8.87993	718
15.40206	5.79832	626
23.37362	3.80279	693
30.99923	2.88706	661
Sum of intensity		4334
% Crystallinity=(4334/10642)×100		40.7

A.1. 1 Calculation of % crystallinity(Si/Al 2.29)

Fusion temperature	Angle 2-theta	d value Angstrom	Intensity count
	6.10878	14.50276	1508
	9.99477	7.57857	584
	15.4452	5.75984	579
250 °C	23.3199	3.81582	660
	30.97095	2.89066	653
	Sum of intensity		3984
	Crystallinity(%)		37.4
	6.10878	14.40729	1823
	9.99477	8.82141	765
	15.4452	5.73371	650
350 °C	23.3199	3.80356	759
	30.97095	2.88163	729
	Sum of intensity		4726
	Crystallinity(%)		44.4
	6.10878	14.45652	5100
	9.99477	8.84281	1325
	15.4452	5.73266	1110
450 °C	23.3199	3.81141	1250
	30.97095	2.88508	1350
	Sum of intensity		10110
	Crystallinity(%)		95
	6.10878	14.53175	4937
	9.99477	8.86483	1255
	15.4452	5.79813	1056
550 C°	23.3199	3.81417	1209
	30.97095	2.88642	1222
	Sum of intensity		9679
	Crystallinity(%)		91

A.1.2 Calculation of % crystallinity(Si/Al 4.05)

Fusion temperature	Angle 2-theta	d value Angstrom	Intensity count
250 °C	6.10878	14.50276	1636
	9.99477	7.57857	718
	15.4452	5.75984	626
	23.3199	3.81582	693
	30.97095	2.89066	661
	Sum of intensity		4334
	Crystallinity(%)		40.7
350 °C	6.10878	14.40729	1822
	9.99477	8.82141	737
	15.4452	5.73371	628
	23.3199	3.80356	728
	30.97095	2.88163	713
	Sum of intensity		4628
	Crystallinity(%)		43.5
450 °C	6.10878	14.45652	2820
	9.99477	8.84281	950
	15.4452	5.73266	766
	23.3199	3.81141	896
	30.97095	2.88508	855
	Sum of intensity		6287
	Crystallinity(%)		59.1
550 C°	6.10878	14.53175	2671
	9.99477	8.86483	914
	15.4452	5.79813	724
	23.3199	3.81417	830
	30.97095	2.88642	845
	Sum of intensity		5989
	Crystallinity(%)		56.2

A.1.3 Calculation of % crystallinity(Si/Al 4.62)

Fusion temperature	Angle 2-theta	d value Angstrom	Intensity count
250 °C	6.10878	14.50276	1822
	9.99477	7.57857	737
	15.4452	5.75984	540
	23.3199	3.81582	728
	30.97095	2.89066	713
	Sum of intensity		4540
	Crystallinity(%)		42.7
350 °C	6.10878	14.40729	1889
	9.99477	8.82141	703
	15.4452	5.73371	599
	23.3199	3.80356	724
	30.97095	2.88163	694
	Sum of intensity		4609
	Crystallinity(%)		43.3
450 °C	6.10878	14.45652	2671
	9.99477	8.84281	914
	15.4452	5.73266	724
	23.3199	3.81141	830
	30.97095	2.88508	848
	Sum of intensity		5984
	Crystallinity(%)		56.2
550 C°	6.10878	14.53175	2671
	9.99477	8.86483	919
	15.4452	5.79813	724
	23.3199	3.81417	830
	30.97095	2.88642	845
	Sum of intensity		5984
	Crystallinity(%)		56.2

A.1.4 Calculation of % crystallinity(Si/Al 2.29)

Fusion time	Angle 2-theta	d value Angstrom	Intensity count
15 min	6.10878	14.50276	972
	9.99477	7.57857	502
	15.4452	5.75984	535
	23.3199	3.81582	593
	30.97095	2.89066	634
	Sum of intensity		3236
	Crystallinity(%)		30.4
30 min	6.10878	14.40729	1888
	9.99477	8.82141	740
	15.4452	5.73371	626
	23.3199	3.80356	729
	30.97095	2.88163	751
	Sum of intensity		4734
	Crystallinity(%)		44.5
45 min	6.10878	14.45652	2800
	9.99477	8.84281	769
	15.4452	5.73266	1500
	23.3199	3.81141	1200
	30.97095	2.88508	1500
	Sum of intensity		9800
	Crystallinity(%)		92.1
60 min	6.10878	14.53175	4937
	9.99477	8.86483	1255
	15.4452	5.79813	1056
	23.3199	3.81417	1209
	30.97095	2.88642	1222
	Sum of intensity		9679
	Crystallinity(%)		91.0

A.1.5 Calculation of % crystallinity(Si/Al 4.05)

Fusion time	Angle 2-theta	d value Angstrom	Intensity count
15 min	6.10878	14.50276	972
	9.99477	7.57857	502
	15.4452	5.75984	535
	23.3199	3.81582	593
	30.97095	2.89066	634
	Sum of intensity		3236
	Crystallinity(%)		30.4
30 min	6.10878	14.40729	2144
	9.99477	8.82141	797
	15.4452	5.73371	684
	23.3199	3.80356	748
	30.97095	2.88163	762
	Sum of intensity		5135
	Crystallinity(%)		48.2
45 min	6.10878	14.45652	2500
	9.99477	8.84281	1100
	15.4452	5.73266	1000
	23.3199	3.81141	688
	30.97095	2.88508	850
	Sum of intensity		6438
	Crystallinity(%)		60.5
60 min	6.10878	14.53175	2671
	9.99477	8.86483	914
	15.4452	5.79813	724
	23.3199	3.81417	830
	30.97095	2.88642	845
	Sum of intensity		5984
	Crystallinity(%)		56.2

A.1.6 Calculation of % crystallinity(Si/Al 4.62)

Fusion time	Angle 2-theta	d value Angstrom	Intensity count
15 min	6.10878	14.50276	972
	9.99477	7.57857	502
	15.4452	5.75984	535
	23.3199	3.81582	593
	30.97095	2.89066	634
	Sum of intensity		3236
	Crystallinity(%)		30.4
30 min	6.10878	14.40729	1888
	9.99477	8.82141	740
	15.4452	5.73371	626
	23.3199	3.80356	726
	30.97095	2.88163	751
	Sum of intensity		4736
	Crystallinity(%)		44.5
45 min	6.10878	14.45652	1897
	9.99477	8.84281	2500
	15.4452	5.73266	606
	23.3199	3.81141	688
	30.97095	2.88508	718
	Sum of intensity		6409
	Crystallinity(%)		60.2
60 min	6.10878	14.53175	2671
	9.99477	8.86483	914
	15.4452	5.79813	724
	23.3199	3.81417	830
	30.97095	2.88642	845
	Sum of intensity		5984
	Crystallinity(%)		56.2

A.1.7 Calculation of % crystallinity(Si/Al 2.29)

CrystallizationFusion temperature	Angle 2-theta	d value Angstrom	Intensity count
30 °C	6.10878	14.50276	0
	9.99477	7.57857	0
	15.4452	5.75984	0
	23.3199	3.81582	0
	30.97095	2.89066	0
	Sum of intensity		0
	Crystallinity(%)		0
60 °C	6.10878	14.40729	0
	9.99477	8.82141	0
	15.4452	5.73371	0
	23.3199	3.80356	0
	30.97095	2.88163	0
	Sum of intensity		0
	Crystallinity(%)		0
90 °C	6.10878	14.45652	4937
	9.99477	8.84281	1255
	15.4452	5.73266	1056
	23.3199	3.81141	1209
	30.97095	2.88508	1222
	Sum of intensity		9679
	Crystallinity(%)		91

A.1. 8 Calculation of % crystallinity(Si/Al 4.05)

CrystallizationFusion temperature	Angle 2-theta	d value Angstrom	Intensity count
	6.10878	14.50276	0
	9.99477	7.57857	0
	15.4452	5.75984	0
30°C	23.3199	3.81582	0
	30.97095	2.89066	0
	Sum of intensity		0
	Crystallinity(%)		0
	6.10878	14.40729	0
	9.99477	8.82141	00
	15.4452	5.73371	0
60°C	23.3199	3.80356	0
	30.97095	2.88163	0
	Sum of intensity		0
	Crystallinity(%)		0
	6.10878	14.45652	2671
	9.99477	8.84281	914
	15.4452	5.73266	724
90°C	23.3199	3.81141	830
	30.97095	2.88508	845
	Sum of intensity		5984
	Crystallinity(%)		56.2

A.1.9 Calculation of % crystallinity(Si/Al 4.62)

Crystallization temperature(°C)	Angle 2-theta	d value Angstrom	Intensity count
30°C	6.10878	14.50276	0
	9.99477	7.57857	0
	15.4452	5.75984	0
	23.3199	3.81582	0
	30.97095	2.89066	0
	Sum of intensity		0
	Crystallinity(%)		0
60°C	6.10878	14.40729	0
	9.99477	8.82141	0
	15.4452	5.73371	0
	23.3199	3.80356	0
	30.97095	2.88163	0
	Sum of intensity		0
	Crystallinity(%)		0
90°C	6.10878	14.45652	2671
	9.99477	8.84281	914
	15.4452	5.73266	724
	23.3199	3.81141	830
	30.97095	2.88508	845
	Sum of intensity		5984
	Crystallinity(%)		56.2

A.1.10 Calculation of % crystallinity(Si/Al 2.29)

Crystallization Time	Angle 2-theta	d value Angstrom	Intensity count
1 h	6.10878	14.50276	505
	9.99477	7.57857	793
	15.4452	5.75984	0
	23.3199.	3.81582	0
	30.97095	2.89066	0
	Sum of intensity		0
	Crystallinity(%)		12.2
2 h	6.10878	14.40729	4937
	9.99477	8.82141	1255
	15.4452	5.73371	1056
	23.3199	3.80356	1209
	30.97095	2.88163	1222
	Sum of intensity		9679
	Crystallinity(%)		91.0
3 h	6.10878	14.45652	4272
	9.99477	8.84281	1110
	15.4452	5.73266	940
	23.3199	3.81141	1068
	30.97095	2.88508	1083
	Sum of intensity		8473
	Crystallinity(%)		79.6

A.1.11 Calculation of % crystallinity(Si/Al 4.05)

Crystallization time	Angle 2-theta	d value Angstrom	Intensity count
	6.10878	14.50276	898
	9.99477	7.57857	0
	15.4452	5.75984	0
1 h	23.3199	3.81582	0
	30.97095	2.89066	0
	Sum of intensity		898
	Crystallinity(%)		8.4
	6.10878	14.40729	2671
	9.99477	8.82141	914
	15.4452	5.73371	724
2 h	23.3199	3.80356	830
	30.97095	2.88163	845
	Sum of intensity		5984
	Crystallinity(%)		56.2
	6.10878	14.45652	1946
	9.99477	8.84281	823
	15.4452	5.73266	659
3 h	23.3199	3.81141	758
	30.97095	2.88508	757
	Sum of intensity		4943
	Crystallinity(%)		46.4

A.1.12 Calculation of % crystallinity (Si/Al 4.62)

Crystallization time	Angle 2-theta	d value Angstrom	Intensity count
1 h	6.10878	14.50276	1223
	9.99477	7.57857	645
	15.4452	5.75984	520
	23.3199	3.81582	588
	30.97095	2.89066	593
	Sum of intensity		3539
	Crystallinity(%)		8.0
2 h	6.10878	14.40729	2671
	9.99477	8.82141	914
	15.4452	5.73371	724
	23.3199	3.80356	830
	30.97095	2.88163	845
	Sum of intensity		5984
	Crystallinity(%)		56.2
3 h	6.10878	14.45652	2267
	9.99477	8.84281	815
	15.4452	5.73266	634
	23.3199	3.81141	747
	30.97095	2.88508	771
	Sum of intensity		5234
	Crystallinity(%)		49.2

Zeolite Synthesis from Fly Ash from Coal-fired Power Plant by Fusion Method

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Abstract: *Coal fly ash (CFA) was used to synthesize zeolite by fusion method. The synthesis conditions were optimized to obtain the product with high cation exchange capacity (CEC). CFA was mixed with NaOH at various ratios and the results revealed that the optimal ratio between CFA and NaOH was 2.25. This gave the maximum CEC of 153.2 meq/100g at 550°C and 1 hour of fusion temperature and time, respectively. The fusion temperature that resulted in the best CEC result was 450°C. The optimal fusion time was found to be 2 hours and this gave the highest CEC of 219.7 meq/100g. The zeolite product was characterized using various techniques, i.e. X-ray diffraction scanning electron microscopy, and the results revealed that the zeolite of this work was closest to the property of the X- type zeolite.*

Keywords: Coal Fly Ash, Zeolite, Cation Exchange Capacity, Fusion Method

1. INTRODUCTION

The need of electrical power requires adequate supply of power stations [1]. Although there are various renewable energy sources that do not encounter environmental problems, a number of the power stations in Thailand are still fed by fossil fuels. Coal is one of the major fossil fuels used in local power stations [2]. In the production of electricity, coal are pulverized to powder form and blown into a furnace by high velocity hot air. The pulverized coal is burnt at a temperature higher than melting points of most minerals which is resided within the coal, causing the transformation of physical and chemical properties of such minerals. Some light minerals are not undergone reactions and are remained in the exhaust gas, and this is called fly ash. The amount of fly ash depends on the mineral matter content of coal. Fly ash is generally composed of Si and Al as major elements (in the form of aluminosilicate) and minor amounts of Fe, Na, K, Ca, P, Ti, and S. Other crystalline minerals are also present in small quantities such as mullite, quartz and lime anhydrite [3].

There are currently several technologies developed for the disposal of fly ash including the usage of fly ash as raw material substitution in cement production [4]. The major components (approx 80%) in fly ash are amorphous aluminosilicate glasses, the conversion of fly ash to zeolite has also been proposed as a viable method [1, 5]. This is not only to generate a useful adsorbent but also to increase the value of fly ash. A number of works has been published on the synthesis of zeolite, but all reported different results either in synthesizing conditions and the type of product.

For instance, Rayalu et al. [6] investigated the effect of the fusion reaction parameters such as ratio of NaOH to fly ash, fusion temperature, crystallization time, and reported that zeolite Y would be obtained best with the NaOH/fly ash of 6:5, fusion temperature of 600°C and crystallization time of 3.5 h. Similar conditions were employed by Somerset et al. [7] who reported the best condition at NaOH/fly ash ratio of 1.2, but the zeolite product of type-A. Different type of zeolite was obtained by Molina et al [8] who also reported similar synthesizing conditions, i.e. the ratio of NaOH/fly ash of 1.2, crystallization temperature of 90°C, and crystallization time of 2 h. This result was most familiar with that of Keka [9] who also obtained zeolite X with the NaOH/fly ash of 1/3.

Various synthesizing conditions led to zeolite with different properties. Wang [11] reported that with the fusion temperature of 100°C, the zeolite product had the highest adsorption affinity for methylene blue, whereas at 200-250°C, the product would have higher affinity for crystal violet.

The difference in the type of zeolite and the properties of zeolite obtained from the literature above could well be due to the differences in the properties of fly ash. Fly ash of different origin will be subject to a variation in their properties and this could have significant effect on the zeolite product. In this work, hence, the synthesis of zeolite was using fly ash from National Power Supply (NPS) in Prachinburi province, Thailand. The fusion method was based on the use of NaOH as the activation reagent. Several fabricating factors that could have strong effects on the properties of the zeolite product were examined, i.e. the ratio between NaOH and fly ash, time and temperature for the fusion process.

2. METHODOLOGY

2.1 Fly ash

Fly Ash is collected from the local coal fired thermal power station (NPS) which supplies electricity to the pulp and paper processes in commercial plantation area in Prachinburi province, Thailand. Chemical characterization analysis by X-ray fluorescence and the results are shown in Table 1.

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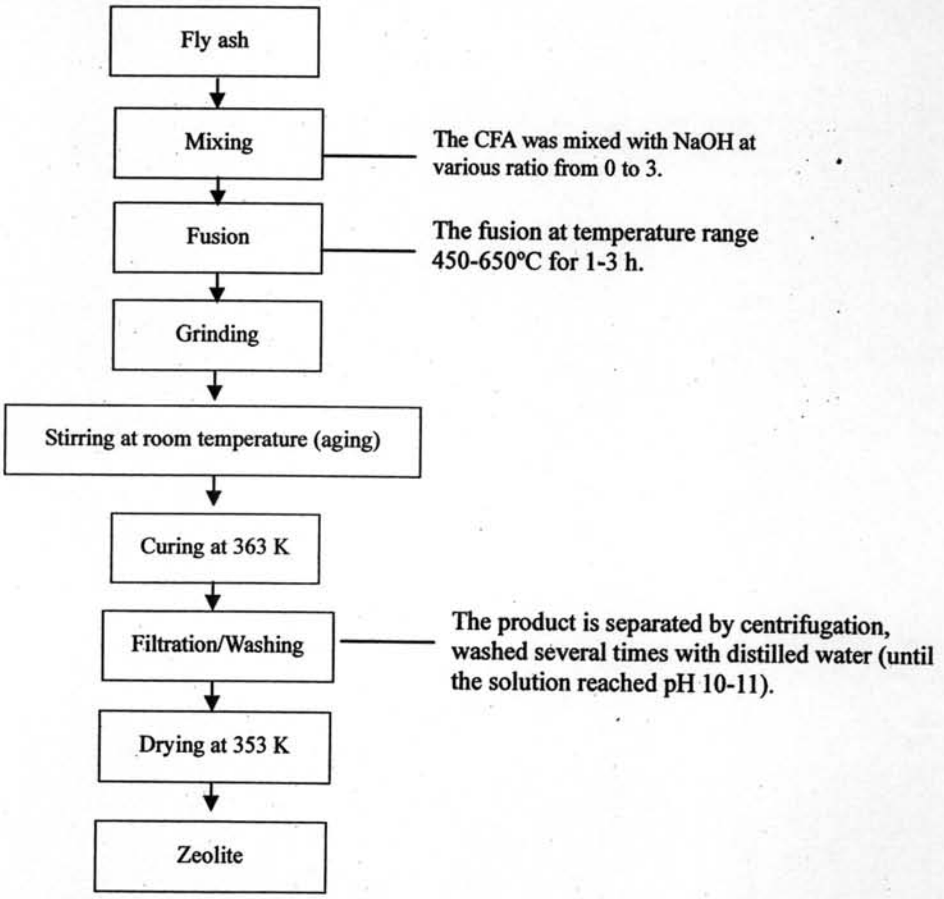
Table 1 Chemical composition of fly ash

Compound	Concentration (% by weight)
SiO_2	43.10
Al_2O_3	21.39
Na_2O	0.98
SO_3	0.01
K_2O	3.56
CaO	0.74
TiO_2	0.68
MnO	0.75
Fe_2O_3	5.27
P_2O_5	0.17
Free lime	0.72

2.2 Zeolite synthesis

Initially, the experiment was performed following the procedure suggested by Molina and Poole [7]. This method started with mixing 10 g of fly ash with 10 g of NaOH (to make the NaOH / PFA of 1). The mixture was burnt in air at 550°C for 1 h in arching furnace. The product was crushed and dissolved in 85 mL of distilled water and mixed in a shaking water bath at room temperature for 24 h. Crystallization was then performed under static condition of 90°C for 2 h where the crystal was collected through filtration technique. The crystal was washed several times with distilled water (until the solution reached pH 10) and dried overnight at 105°C [2]. For this work, the quality of zeolite was indicated by the cation exchange capacity (CEC) which represents the ability of the zeolite in absorbing the metal or other cation contaminants. The synthesizing condition as recommended by [7] were further examined to ensure the suitability for the fly ash and used in this work. This work was therefore divided into two parts. The first part aimed at the determination of the optimal NaOH/CFA ratio in the mixing process that would provide the zeolite with the highest CEC. The synthesis was repeated with NaOH/PFA ratios of 0.5, 1.25, 1.5, 1.75, 2, 2.25, 2.5, 3 g/g. The ratio with the highest CEC obtained from this part was used in subsequent experiments. The second part was to investigate of temperature and time in the fusion process. The range of furnace temperature examined here was 450, 550 and 650 °C, and for the time period of 0.5 1, 2 and 3 h. Detail of the experimental conditions are given in Fig.1. The final zeolite product was analyzed for its degree of crystallinity using X-ray (XRD) analysis (SIEMENS XED D5000, accurately in the 5-40° 2θ).



The CFA was mixed with NaOH at various ratio from 0 to 3.

The fusion at temperature range 450-650°C for 1-3 h.

The product is separated by centrifugation, washed several times with distilled water (until the solution reached pH 10-11).

Fig. 1 Synthesis of zeolite

2.3 Cation Exchange Capacity

The cation exchange capacity (CEC) was determined by mixing 3.2 g of zeolite (quantity "*m*" in Eq. 3.1) with 27 ml of 1.0 N CH_3COONa solution in a mechanical shaker for 5 min. The mixture was then centrifuged until clear supernatant was obtained and decanted from the mixture. This procedure was repeated another two times to ensure that all cations in the zeolite have been replaced with Na.

The Na laden zeolite was mixed with 27 ml of 99% isopropyl alcohol and shaken in a mechanical shaker for 5 min. The supernatant was removed and the procedure was repeated two times to ensure that the zeolite was clean and laden with Na only. The zeolite was then mixed with CH_3COONH_4 solution. The same procedure was applied with CH_3COONH_4 two times to ensure that all Na was replaced by NH_4 ion. After each step, the supernatant of CH_3COO^- solution was decanted to a 100 mL volumetric flask.

The supernatant was diluted to 100 mL (quantity "*V*" in Eq. 3.1) with ammonium acetate solution and finally the sodium concentration in this supernatant was determined by atomic absorption emission spectroscopy. The sodium binding capacity can be then calculated from:

$$q_e = \frac{(c_0 - c_e)}{MW \times m} \times V \qquad (1)$$

where q_e is ion adsorbed per unit mass of adsorbent (mmolg^{-1}), c_0 the initial metal ions concentration (mgL^{-1}) c_e the equilibrium metal ion concentration (mgL^{-1}), V the volume of the aqueous phase (mL), and m the amount of adsorbent(g), MW the molecular weight (g).

3. RESULTS AND DISCUSSION

The effects of three different factors on the fusion synthesis were investigated. These factors included the amounts of NaOH, fusion temperature, fusion time. The results are evaluated based on their CEC values.

Effect of NaOH

A series of experiments was undertaken to determine the effects of NaOH/fly ash ratio on zeolite formation under conditions described for each method. The CEC properties of the final products are presented in Fig. 1 where it was clearly observed that CEC could be enhanced just by increasing the quantity of NaOH during the fusion process. This increase was due to the higher degree of zeolization taking place as a result of an increase in the reaction extent associated with a higher NaOH content. This finding agreed well with the reports of [2, 12]. 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 benefit on the CEC, rather the CEC was drastically reduced in this high NaOH/CFA ratio range because of deterioration of properties of zeolite. The most suitable NaOH/CFA ratio for the adsorption of cationic species as observed from the high level of CEC was at 2.25. At this level, the maximum value of CEC was obtained at 154.7meq/100g.

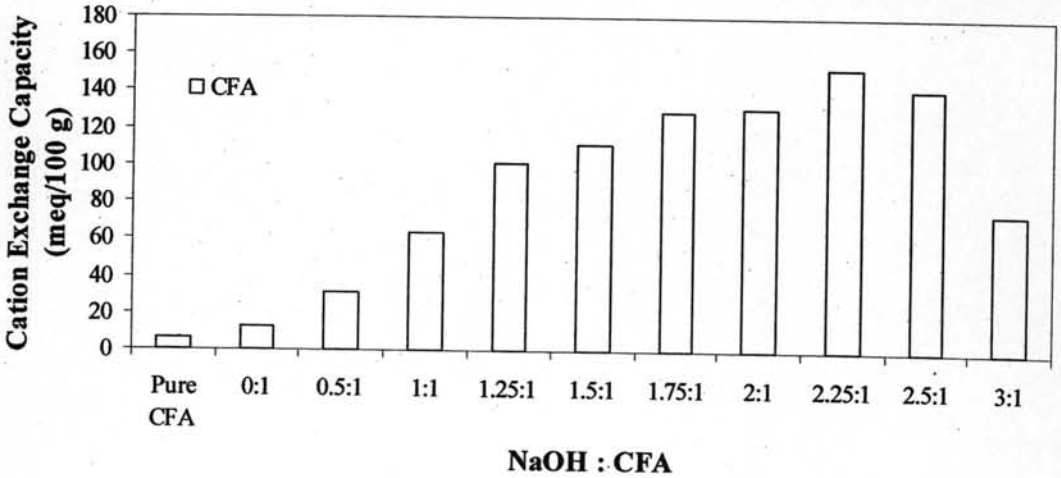


Fig. 2 Effect of NaOH/fly ash ratio on the CEC value of the zeolite
(fusion temperature 550 °C at 1 h, water content 85 ml, crystallization temperature 90°C at 2 h)

Effect of temperature and time of fusion

The fusion temperature and time are also important factors for the formation of zeolite. Fig. 4 demonstrates that the temperature could have strong effect on the properties of the obtained zeolite. There are various reports on the value of the fusion temperature and most indicated the temperature of higher than 500°C, e.g. Molina et al [7] reported the fusion temperature of 550°C. However, it was clear from the results in Fig. 4 that this might not always be the case, and for this work, the most suitable fusion temperature was 450°C. It was difficult to conclude on the optimal value of the fusion temperature as 450°C was already on the edge of the experimental condition employed in this work. This aspect must be further rectified with more experimental evidence.

The time for fusion process could also play a significant role in dictating the property of the final zeolite product. Literature stated that a shorter time would lead to an incomplete reaction whilst the longer time would result in a damage to the zeolite crystal structure and both led to a bad CEC property. In this work, the suitable fusion time was found to be around 2 hours. Fig. 4 illustrates that, at the best zeolite synthesizing conditions, i.e. 450 °C and 2 h, the attainable CEC could be as high as 220 meq/100g.

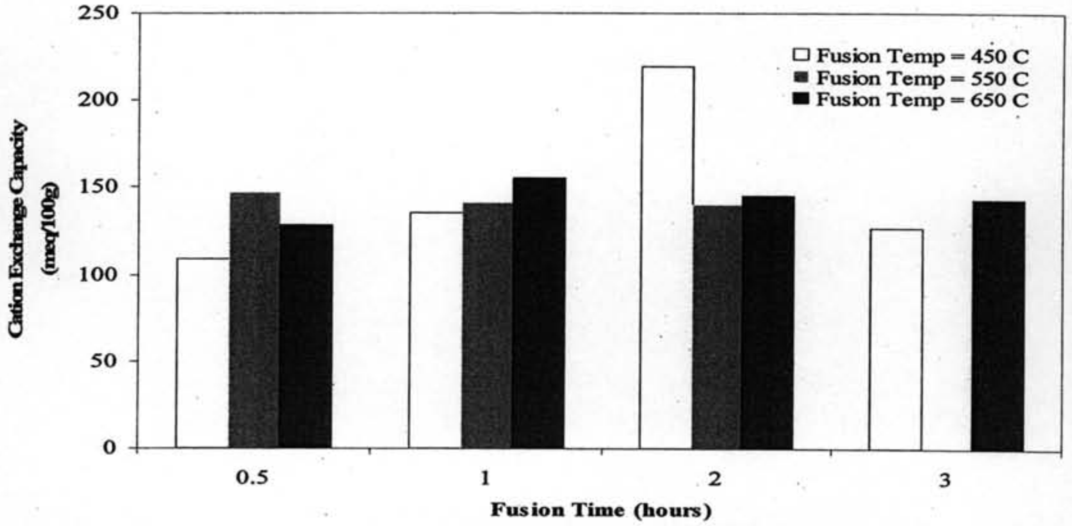


Fig. 3 Effect of temperature and time of fusion
(Ratio of NaOH 2.5 ,water 85 ml, crystallization temperature 90°C at 2 h)

Type of zeolite

The type of zeolite could be identified using the XRD pattern. For this work, the XRD of the zeolite products at various NaOH/fly ash ratio are presented in Fig. 5. This was considered to be closest to the XRD pattern of zeolite type X.

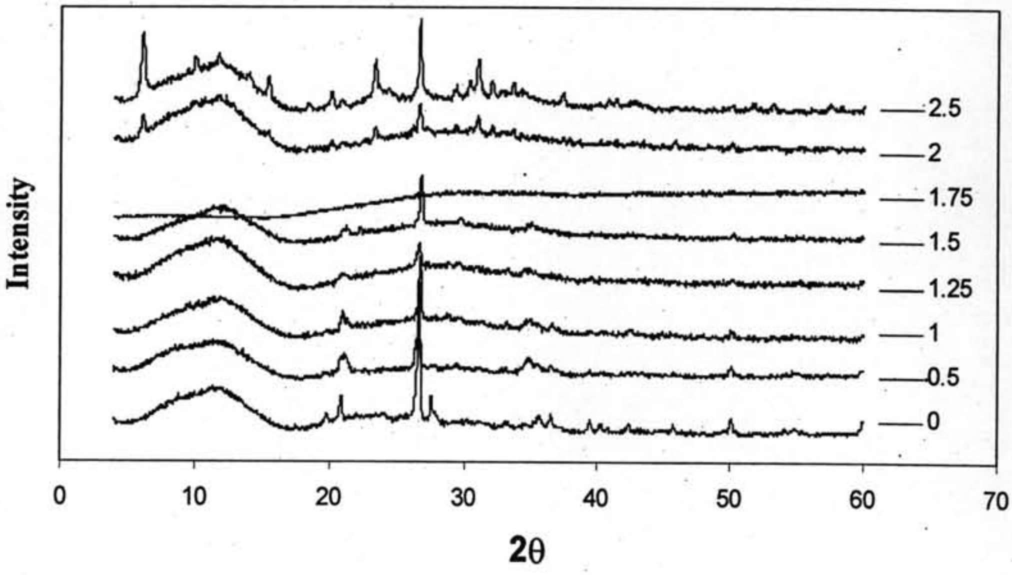


Fig. 4 XRD of patterns of fly ash, synthesized zeolite

4. CONCLUSION

The synthesis of zeolite was investigated in this work. The optimal condition for synthesis was as follows: NaOH/CFA ratio of 2.25, fusion temperature of 450°C, and 2 h of fusion time. This optimal condition was by far different from the reporting data in literature which supported the statement that fly ash from different origin would be subject to different synthesizing conditions. In addition, even the fly ash from the same source could be subject to a large variety in properties as the operating conditions of the power plant could be different or the property of the coal could be different. Further work is still needed to examine the effect of the variation in the raw materials on the properties of the zeolite.

5. ACKNOWLEDGMENTS

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BIOGRAPHY

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