

Synthesis of Zeolite A from By-Product of Aluminum Etching Process: Effects of Reaction Temperature and Reaction Time on Pore Volume

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Abstract: Problem statement: Increasing of the carbon dioxide (CO₂) concentration in the atmosphere creates a global warming. Zeolite is used as CO₂ absorbent. **Approach:** By-products from aluminum etching process, which carry a large amount of Al₂O₃ content, are useable as raw materials for synthesis zeolites type A using hydrogel process. A pore volume of zeolites is the key to achieve high CO₂ absorption capacity. In this study, two-factor factorial experiments with 3 replicates and $\alpha = 0.05$ have been conducted to examine the influence of reaction temperature and reaction time on the pore volume and to determine the process conditions for containing the highest pore volume. Three levels of the reaction temperature of 75, 85 and 95°C and three levels of the reaction time of 1, 2 and 3 h (s), while the stirring speed is controlled at 300 rpm, are selected for the experiment. The mole ratios of the starting reactants are fixed at 2 of SiO₂/Al₂O₃, 2 of Na₂O/ Al₂O₃ and 85 of H₂O/Na₂O. Analysis of surface area is performed by the BJH method and the DR method for analysis of gas adsorption-desorption using nitrogen. **Results:** The results found that there was significant interaction between the reaction temperature and the reaction time and at 95°C of 3 hours gave the highest pore volume, resulting in 0.0205cc g⁻¹ of the BJH method and 0.0065 cc g⁻¹ in the DR method. Besides, the CO₂ absorption test is also performed with the zeolite synthesized at 95°C. **Conclusion:** Zeolite syntheses of by product from alkaline etching of the aluminum industry have a major analysis agree well with those of reference zeolite A. Adsorption test with CO₂, yield the best result with ZA31, with the capacity of 0.1023 ccg⁻¹.

Key words: Waste product, hydrogel process, zeolite A, BET technique, BJH method, DR method, Brunauer-Emmett-Teller (BET), Pore Volume (PV), gas absorbed, Zeolite A (ZA)

INTRODUCTION

The properties and uses of zeolites are being explored in many scientific disciplines: modern inorganic chemistry, physical chemistry, colloid chemistry, biochemistry, mineralogy, geology, surface chemistry, oceanography, crystallography, catalysis and in all types of chemical engineering process technology. The wide variety of applications includes separation and recovery of normal paraffin hydrocarbons, catalysis of hydrocarbon reactions, drying of refrigerants, separation

of air components, carrying catalysts in the curing of plastics and rubber, recovering radioactive ions from radioactive waste solutions, removing carbon dioxide and sulfur compounds from natural gas, sampling air at high altitudes and removal of atmospheric pollutants such as sulfur dioxide (Breck, 1984).

Commercial adsorbents which exhibit ultraporosity and which are generally used for the separation of gas and vapor mixtures include the activated carbons, activated clays, inorganic gels such as silica gel and activated alumina and the crystalline aluminosilicate

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zeolites. Zeolite molecular sieves have pores of uniform size (3-10 Å) which are uniquely determined by the unit structure of the crystal. These pores will completely exclude molecules which are larger than their diameter.

Zeolites are crystalline, hydrated aluminosilicates of group I and group II elements (as formed in nature or synthesized), in particular, sodium, potassium, magnesium, calcium, strontium and barium. Structurally the zeolites are “framework” aluminosilicates which are based on an infinitely extending three-dimensional network of AlO_4 and SiO_4 tetrahedra linked to each other by sharing all of the oxygen's. The chemical formula of zeolite is best expressed for the crystallographic unit cell as: $\text{M}_{2/n}\text{O} \cdot \text{Al}_2\text{O}_3 \cdot x\text{SiO}_2 \cdot y\text{H}_2\text{O}$ (Breck, 1984). In this oxide formula, x is generally equal to or greater than 2 since AlO_4 tetrahedra are joined only to SiO_4 tetrahedra, n is the cation valence. The framework contains channels and interconnected voids which are occupied by the cation and water molecules as shows in Fig. 1 (Auerbach *et al.*, 2003).

Hydrogel process of zeolites is generally synthesized from sodium aluminosilicate gel prepared from various silica and alumina ratio as raw material (Breck, 1984). The gels are crystallized in a closed hydrothermal system at temperatures varying generally from room temperature to about 175°C. The alkali metals form soluble hydroxides, aluminates and silicates. These materials are well suited for the preparation of homogenous mixture. The gel preparation and crystallization is represented schematically using the $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$ (zeolite A) system as shows in Fig. 2 (Breck, 1984).

The gel is probably produced by the copolymerization of the individual silicate and aluminates species by a condensation-polymerization mechanism. The gel composition and structure appear to be controlled by the size and structure of the polymerizing species.

The etching process in the aluminum profile is etched by sodium hydroxide solution which produces an aluminum hydroxide ($\text{Al}(\text{OH})_3$) as by-product, which carry a large amount of Al_2O_3 content. It produces a low wholesale price aluminum hydroxide ($\text{Al}(\text{OH})_3$) normally further used for producing alum ($\text{Al}_2(\text{SO}_4)_3$).

Thus, higher valued added product such as zeolite should be considered as in this research.

This research focuses on reaction temperature and reaction time as controlling factor for synthesis of zeolite A and also on its pore volume. This study aims to achieving the following: (i) synthesis of zeolite A by hydrogel process using raw material from the byproduct of aluminium etching process, (ii) analysis of surface area is performed by the BJH method and the DR method for analysis of gas adsorption-desorption using nitrogen., (iii) CO_2 adsorption capacity.

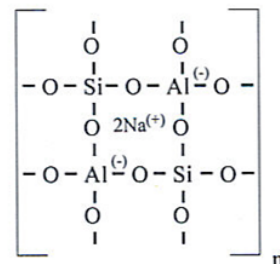


Fig. 1: The aluminosilicate framework of zeolite A

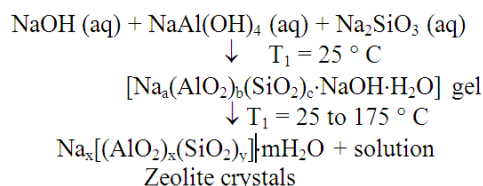


Fig. 2: The crystallization of synthesis zeolite

MATERIALS AND METHODS

Materials: Synthetic zeolite was synthesized by hydrogel process, using as sodium metasilicate (from Ajax Finechem, lab grade), sodium hydroxide (from Carlo Erba, lab grade) and by-product from aluminum etching process.

Synthesis of zeolites: Preparing of the reactant sodium silicate solution; weigh sodium metasilicate about 42 g, dissolve in water at temperature 50°C to make the mole ratio of reactants of 2 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 2$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 2$). Preparing of sodium aluminates solution; weigh by-product from aluminum etching process about 10.7525 g to make the mole ratio of reactants of 2 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 2$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 2$), dissolve into water 240 cm^3 and add 30 cm^3 of 50% NaOH at temperature 60°C for 30 min before filtrating. The hydrogel process of the synthetic was carried out between sodium silicate solution and sodium aluminates solution at the range of temperature from 75-95°C in stirrer tank reactor, temperature controlled by water bath with fixed stirring speed at 300 rpm for 1-3 h. The experimental design was reported in Table 1, with the crystallization products obtained by filtrating, washed with deionized water and dried in an air oven at 100°C for 16 h.

Characterization: The chemical composition of by-product, referenced Zeolite A (ZA) and synthetic

Table 1: List of experimental design: temperature and reaction time. (The mole ratios of the starting reactants are fixed at 2 of SiO₂/Al₂O₃, 2 of Na₂O/ Al₂O₃ and 85 of H₂O/Na₂O.)

Samples	Time (h)	Temp. (°C)
ZA11	1	95
ZA12	1	85
ZA13	1	75
ZA21	2	95
ZA22	2	85
ZA23	2	75
ZA31	3	95
ZA32	3	85
ZA33	3	75

Note: ZAab: a is reaction time (1= 1 h, 2 = 2 h, 3 = 3 h) and b is reaction temperature (1= 95°C, 2 = 85°C, 3 = 75°C)

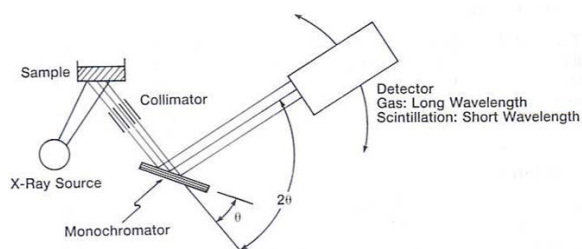


Fig. 3: Schematic of a wavelength dispersive X-Ray fluorescence spectrometer (Sibilia, 1996)

zeolites were determined by X-ray fluorescent spectroscopy (Philips PW-2404) is, a non-destructive method for qualitative and quantitative analysis of elemental composition for a wide range of materials. The method is extremely useful because of the ease of sample preparation. For a qualitative elemental analysis, the sample can be run “as received” with virtually no sample preparation. The sample can be in any shape (powder, pellet, film, fiber, or chunk of material) and state (liquid, solid, or suspension).

The sample can be several centimeters in diameter or weigh as little as a few milligrams. XRF is based on the photoelectric effect. When an atom is irradiated with highly energetic photons, an electron from one of the inner shells may be ejected. As the vacant place is filled by an electron from an outer shell, a photon, whose energy is characteristic of the atom, is released. This radiation is called the fluorescent radiation and each element has its own set of characteristics emission or XRF lines. The intensity and the energy of these lines are measured using a spectrometer.

The spectrometer depends on the method used to separate the energies of the emitted radiation. In the wavelength dispersive X-Ray Fluorescence (WDXF) spectrometer, a crystal grating is used to separate the energies and the intensities are measured by a conventional X-ray detector (Fig. 3) (Sibilia, 1996).

The surface areas of various samples were determined by gas adsorption and desorption (Micromeritics ASAP 2010). The specific surface areas of the samples were calculated using the Brunauer-Emmett-Teller (BET) single-point method. Approximately 0.01-0.03 g of the sample was placed in the sample cell, heated to 623 K and held at that temperature for 6 h under a N₂/He flow. The sample was then cooled to room temperature and dipped into liquid nitrogen. After the adsorption of nitrogen reached equilibrium, the sample cell was then dipped into a water bath at room temperature. The amount of nitrogen desorbed was measured by a gas chromatograph.

The surface area of powdered or porous solid can be calculated from the volume of gas absorbed onto the surface of the solid. In general, solids adsorb gases weakly bound due to Van der Waal's forces only, to cause sufficient gas to be absorbed for surface area measurement. The volume of gas absorbed increases with increasing pressure. The physical absorption of gases by solids increases with decreasing temperature and with increasing pressure. The process is exothermic, i.e., energy is released. The investigative procedure has first to establish what is known as an absorption (or desorption) isotherm. This, quite simply, is a measure of the molar quantity of gas n (or standard volume V_a , or general quantity q) taken up, or released, at a constant temperature usually T by an initially clean solid surface as a function of gas pressure P . Most frequently the test is conducted at a low temperature, usually that of liquid nitrogen (LN₂) at its boiling point (77.35 K at one atmospheric pressure) (Sibilia, 1996).

Convention has established that the quantity of gas absorbed is expressed as its volume at standard conditions of temperature and pressure (0°C and 760 torr and signified by STP), while the pressure is expressed as a relative pressure. This is the actual gas pressure P , divided by the vapor pressure P_0 of the adsorbing gas (called the adsorptive prior to adsorption and adsorbate after ward) at the temperature at which the test is conducted. All analyses first must establish information in the form of quantity adsorbed (or desorbed) V_s pressure. Therefore the measurements requirement of the highest quality cannot be overemphasized. These data are gathered at one temperature. Plots of V_a as the ordinate against P/P_0 as the abscissa reveal much about the structure of the adsorbing material (called the adsorbent) simply from their shape.

The Dubinin-Radushkevich method (DR), while the concept of an adsorbed layer of gas on the walls of large pores is valid, is not necessarily applicable when the pores are a little more than one gas molecule wide as in micro pores. This led Dubinin and Radushkevich to develop an empirical equation applicable to volume filling, in microporous carbons. Their theory incorporates earlier work by Polanyi, in regard to the adsorption potential A (Webb, 1997).

Where:

$$A = RT \ln \left(\frac{P_0}{P} \right) = -\Delta G \quad (1)$$

and expresses the differential molar work of adsorption. In this expression, R is the universal gas constant, T the absolute temperature, P_0/P the reciprocal relative pressure and ΔG the change in the Gibbs free energy. The Dubinin-Radushkevich (D-R) (Paul) equation has the form:

$$W = W_0 \exp \left[- \left(\frac{A}{\beta E_0} \right)^2 \right] \quad (2)$$

Where:

W = The quantity, adsorbed at relative pressure P/P_0 and temperature

T, W_0 = The limiting micropore volume

E_0 = The characteristic energy of adsorption for the reference vapor, benzene and β the affinity coefficient

This has a value of unity when the adsorptive is the reference vapor. Substituting Eq. 3 for Eq. 3, then extracting the logarithm gives (Webb, 1997):

$$\ln W = \ln W_0 - \left(\frac{RT}{\beta E_0} \right)^2 \left(\ln^2 \frac{P_0}{P} \right) \quad (3)$$

Some data sets from experimental isotherms, when plotted as $\ln W$ against $\ln^2(P_0/P)$, produce straight lines over a range of gas pressures. When a straight line is achieved, its slope is the value of $-(RT/\beta E_0)^2$ and the y-axis intercept is $\ln W_0$. W_0 , being a gas volume, must be converted to its liquid volume to characterize appropriately the total micro porosity. Using nitrogen gas also produces straight lines on micro porous solids.

BJH method, the procedure for calculating pore size distribution using the Kelvin equation, involves an imaginary emptying of condensed adsorptive in the pores in a stepwise manner, as the relative pressure is

likewise decreased. The mathematics of the technique is equally applicable, whether following the adsorption branch of the isotherm downward from high to low pressure, or following the desorption branch. In either case, the condition must be set arbitrarily where all pores are to be considered filled. Typically this is taken to be at about 99.5% relative pressure ($P/P_0 = 0.995$). The calculation method follows generally that described hence called the BJH method. As an example, it is convenient to imagine the isotherm as a series of steps downward of equal relative pressure, say 0.95, 0.90, 0.85. The amount of adsorptive lost in each step (after conversion from gas volume at standard conditions to a liquid volume) represents the core volumes of pores emptied in that step (Webb, 1997):

$$\ln \left(\frac{P^*}{P_0} \right) = - \left(\frac{2\gamma v \cos \theta}{RT r_m} \right) \quad (4)$$

Where:

P^* = The critical condensation pressure

γ = The liquid surface tension

v = The molar volume of condensed adsorptive

θ = The contact angle between the solid and condensed phase (taken to be zero when the adsorptive is nitrogen, hence $\cos \theta = 1$)

r_m = The mean radius of curvature of the liquid meniscus and P^*/P_0 , R and T as used previously

It follows when the condensate meniscus is concave ($\theta < 90^\circ$), which capillary condensation will proceed in the pores of radius r_m . This will happen as long as the adsorptive pressure is greater than the critical condensation and can or cannot occur at a given relative pressure.

Now from Eq. 4 (Webb, 1997), with the substitution of (r-t) for r_m , a value of r is calculated. At this point another choice needs to be made, with regard to the shape of the pore. For example, if a straight cylindrical pore model is chosen, substituting the value of r_m and the volume of pores of size, r_m . This will happen into the general equation for the volume of a right circular cylinder (volume = $\pi r^2 L$) allows resolution of the total length of pores. From this we can calculate the wall area of the pores using the menstruation equation, area = $2\pi r L$. All of the above was only for the first step down the isotherm. A pore distribution of radius (or diameter), area and volume will not have been achieved, until the process is completed.

The CO_2 adsorption using small column test equipment was conducted on the adsorbents of the

synthesized zeolite. Adsorbent (1g) of the synthesized zeolite was placed in a u-tube stainless steel column 1 cm diameter, using CO₂ (99.5%) as test gas with N₂ as carrier with the total flow rate 15 cm³ min⁻¹. A Gas Chromatograph (GC) was used to analyze the concentration of CO₂ in the inlet and outlet gases.

Design of experiments: In this study, we conducted two-factor factorial experiments with 3 replicates and $\alpha = 0.05$ in order to examine the influence of reaction temperature and reaction time on the pore volume and to determine the process conditions for containing the highest pore volume. We used three levels of the reaction temperature of 75°C, 85 and 95°C and three levels of the reaction time of 1, 2 and 3 h (sec), while the stirring speed is controlled at 300 rpm,. The mole ratios of the starting reactants are fixed at 2 of SiO₂/Al₂O₃, 2 of Na₂O/Al₂O₃ and 85 of H₂O/Na₂O. Analysis of surface area is performed by the BJH method and the DR method for analysis of gas adsorption-desorption using nitrogen.

RESULTS

Chemical characterization: The chemical composition of the aluminum etching by-product consisted of the following oxides expressed as percentage by weight; Al₂O₃, 92.17 %; Na₂O, 6.03 %; SiO₂, 0.47 % (Hussaro *et al.*, 2008). It carries a large amount of Al₂O₃ content which is useable as raw materials for synthesis of zeolites. Si and Na compositions could be adjusted by the addition of sodium metasilicate.

The results of the mole ratio and the chemical composition in the referenced zeolite A (ZA) and synthetic zeolites were reported in Table 2. These results show that the mole ratio of Na₂O/Al₂O₃ and SiO₂/Al₂O₃ of ZA and synthetic zeolites are affected by reaction temperature and reaction time in the range between 75-95°C and 1-3 h respectively. The above results indicate that the highest reaction temperature and time in the range (ZA31, 95°C in 3 h) resulted in the successful synthesis of zeolite A. The compositions were compared with the reference zeolite A in Table 2. The increasing reaction temperature and reaction time ensured the better value of the mole ratio of Na₂O/Al₂O₃ (ZA = 1.05), ZA31 = 1.07) and SiO₂/Al₂O₃ (ZA = 2, ZA31 = 2.10), which is the indices of the zeolite A framework.

The results of the mean of Pore Volume (PV) from three methods are reported in Table 3. By varying two variables simultaneously and obtaining multiple measurements under the same experimental conditions, the experimental responds are randomized.

Table 2: The mole ratio of reference zeolite A and synthetic zeolites.

Samples	Mole ratio of samples	
	Na ₂ O/Al ₂ O ₃	SiO ₂ /Al ₂ O ₃
ZA ^a	1.05	2.00
ZA31	1.07	2.10
ZA32	1.11	2.15
ZA33	1.13	2.20
ZA21	1.14	2.16
ZA22	1.16	2.18
ZA23	1.20	2.52
ZA11	1.15	2.21
ZA12	1.28	2.45
ZA13	1.36	2.60

^a: Reference zeolite A from Thai Silicate Chemicals CO., LTD in the sodium ion form (Na-A zeolite, CODE: SMC-P) All three ANOVA tables show a significant interaction between reaction temperature and reaction time on the pore volume (Sig.<0.05)

Table 3: The mean of 3 individual values of pore volume from three methods

Samples	Mean of pore volume (cc g ⁻¹)		
	BJH method	DH method	DR method
ZA	0.0223	0.0247	0.00680
ZA31	0.0205	0.0231	0.00650
ZA32	0.0183	0.0218	0.00510
ZA33	0.0165	0.0193	0.00470
ZA21	0.0147	0.0162	0.00390
ZA22	0.0122	0.0154	0.00310
ZA23	0.0095	0.0114	0.00280
ZA11	0.0087	0.0099	0.00250
ZA12	0.0061	0.0072	0.00230
ZA13	0.0053	0.0059	0.00189

The normality of the respond data is assumed. They are analyzed by using SPSS statistical software. The results of applying the analysis of variance are shown in Table 4.

All three ANOVA tables show a significant interaction between reaction temperature and reaction time on the pore volume (Sig.<0.05).

The results of gas adsorption studies conducted utilizing a gas of 99.5 % CO₂ with 99.9 % N₂ carrier on samples were shown in Table 5. Carbon dioxide (CO₂) adsorption for ZA31, ZA21 and ZA11 showed that the amount of CO₂ adsorbed increased progressively with reaction time of synthesis zeolite. The total amount of CO₂ adsorbed followed the order of ZA31>ZA21>ZA11.

The capacity of CO₂ adsorbed by ZA was 0.1183 cc g⁻¹. While that of ZA31 was 0.1023 cc g⁻¹, which was better agreed with ZA as compared with other sample (Table 5). These results show that the synthetic zeolite could absorb the CO₂, but the adsorption capacity of reaction time these result confirmed by pore volume analysis, showing the increase pore volume with increased reaction temperature and reaction time.

Table 4: ANOVA tables from BJH, DH and DR method respectively versus reaction temperature and reaction time

Dependent variable: BJH					
Source	Type III Sum of Squares	DF	Mean square	F	Sig
Tests of between-subjects effects					
Corrected model	0.001	8	0.000	604.809	0.000
Intercept	0.004	1	0.004	28624.214	0.000
Time	0.001	2	0.000	2131.992	0.000
Temp	0.000	2	0.000	274.969	0.003
Time * Temp	0.000	4	0.000	6.137	0.003
Error	0.000	18	0.000		
Total	0.005	27			
Corrected total	0.001	26			

a: $R^2 = 0.996$ (Adjusted $R^2 = 0.995$)

Dependent variable: DH					
Source	Type III Sum of squares	DF	Mean square	F	Sig
Tests of between-subjects effects					
Corrected model	0.001 ^a	8	0.000	483.028	0.000
Intercept	0.006	1	0.004	23328.495	0.000
Time	0.001	2	0.000	1752.440	0.000
Temp	0.000	2	0.000	166.954	0.000
Time * Temp	0.000	4	0.000	6.358	0.002
Error	0.000	18	0.000		
Total	0.007	27			
Corrected total	0.001	26			

a: $R^2 = 0.995$ (Adjusted $R^2 = 0.993$)

Dependent variable: BJH					
Source	Type III sum of squares	DF	Mean square	F	Sig
Tests of between-subjects effects					
Corrected model	0.000 ^a	8	0.000	81.623	0.000
Intercept	0.000	1	0.004	4191.584	0.000
Time	0.000	2	0.000	280.558	0.000
Temp	0.000	2	0.000	37.442	0.003
Time * Temp	0.000	4	0.000	4.247	0.014
Error	0.000	18	0.000		
Total	0.000	27			
Corrected total	0.000	26			

a: $R^2 = 0.973$ (Adjusted $R^2 = 0.961$)

Table 5: The values of pore volume for all samples

Samples	The total amount of CO ₂ adsorbed (cc g ⁻¹)
ZA	0.1183
ZA31	0.1023
ZA21	0.0965
ZA11	0.0894

DISCUSSION

The temperature of a silicate solution also affects the polymerization of silicate anion in the solution. The reaction temperature and reaction time depends on the distribution of silicate anions in aluminosilicate solution. Therefore, the samples of the synthetic zeolite at different reaction temperature were subjected to chemical analysis. The trends were in close agreement, showing that the mole ratio of framework (SiO₂/Al₂O₃ and Na₂O₃/Al₂O₃) in the solid sample with an increase in the temperature and time, compared to the corresponding ratio of the reference zeolite as shown in Fig. 5.

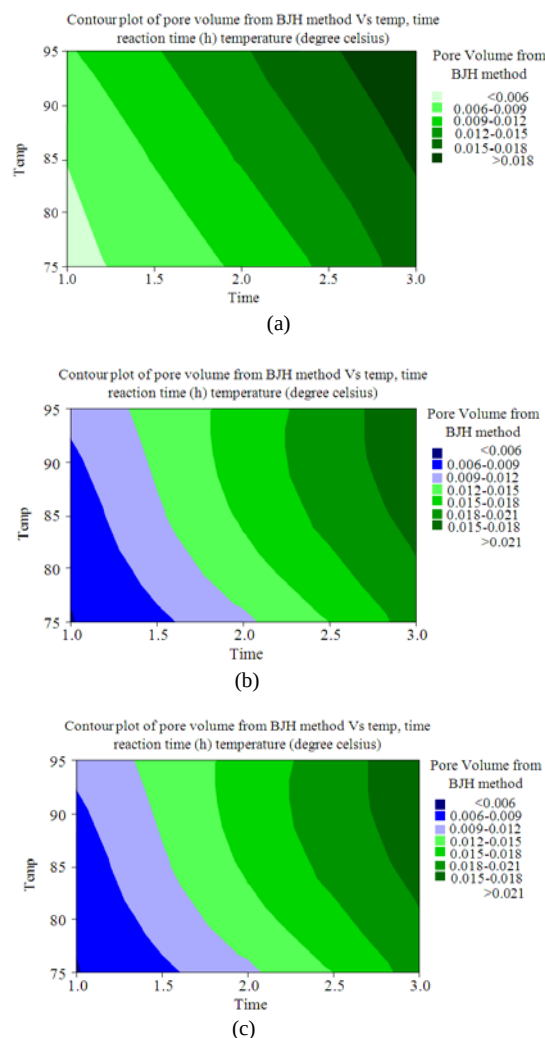


Fig. 4: Contour Plots of Pore Volume from BJH, DH and DR method respectively versus temp, time

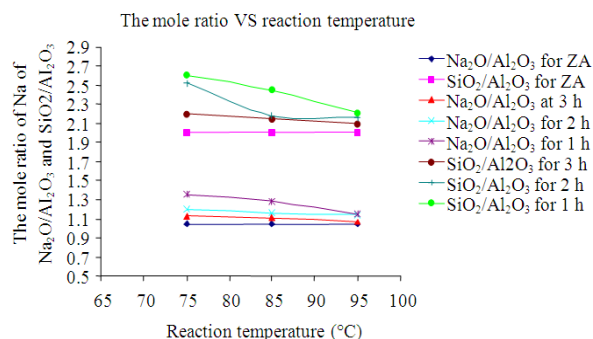


Fig. 5: The mole ratios versus reaction temperature of synthesis zeolites vary reaction time.

The contour plots from Fig. 4 show that the optimum conditions for containing the highest pore volume are at reaction time of 3 h and reaction temperature of 95°C. It can be seen that, the PV of ZA31 for all methods were higher than other synthesis zeolites and the PV of ZA31 is close to ZA. These results indicated that the SA of synthetic zeolites increases progressively with increases reaction temperature and reaction time this could be the results of more perfect structure of ZA31 as compared to the other synthetic zeolites. However, the BJH and DH method measured cumulative adsorption surface area; combination of mesopore volume and micro porosity. While the DR methods were the analysis for micro porosity. Therefore, the pore volume of the BJH and DH method were higher than the DR method.

One arrives at the conclusion that the data obtained from all methods of pore volume of ZA31 sample and ZA sample were quite similar. This indicates the presence of similar structural units and the formation of identical chemical moieties in the ZA31. This also tallies with the results obtained from XRF.

CONCLUSION

The present investigation has demonstrated that zeolite A can synthesize within 1-3 h at 75-95°C conditions from by-product of aluminium etching process. The chemical composition of by-product of aluminium etching process showed that it could be utilized as a very proper raw material for synthesis zeolites. There was a large amount of Al_2O_3 content, which is one of the main compositions of synthetic zeolite A; namely, Al and Si. While, Si and Na composition can be adjusted by sodium metasilicate. The chemical composition analysis performed for

reference zeolite A and synthetic zeolites indicated that higher synthesis reaction temperature and higher reaction time resulted in the more successful synthesis of zeolite A. This relatively rapid transformation of the amorphous aluminosilicate species into a zeolite a material under these conditions. Due to what?, these conditions generally yield more condensed phase species and that at lower reaction temperature and lower reaction time (such as 75-85°C); there were the short chain molecule of $\equiv \text{Si-O}^-$ which contributes to parts of the amorphous zeolite. This relatively rapid transformation of the amorphous aluminosilicate species into a zeolite a material under this conditions.

Zeolites of different pore volume, silica/alumina ratio and the total amount of CO_2 adsorbed were obtained by changing the reaction temperatures and reaction time. The best quality zeolite within the range of this experiment was obtained at the following conditions: $\text{SiO}_2/\text{Al}_2\text{O}_3$, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ reaction ratios of 2; reaction temperature, 95°C for 3 h by hydrogel process. The chemical composition confirms that the main product was zeolite A. The synthetic zeolite had the mole ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ of 1.07 and $\text{SiO}_2/\text{Al}_2\text{O}_3$ of 2.1 while the reference zeolite A had the mole ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ of 1.05 and $\text{SiO}_2/\text{Al}_2\text{O}_3$ of 2.00. The pore volume of synthetic zeolite also increased with reaction temperature and reaction time.

From the experiments, all three ANOVA tables show a significant interaction between reaction temperature and reaction time on the pore volume and the optimum conditions for containing the highest pore volume are at reaction temperature of 95°C and reaction time of 3 h.

Therefore, it can be concluded that zeolite synthesis of by product from alkaline etching of the aluminum industry have the major analysis agree well with those of reference zeolite A. Adsorption test with CO_2 , yield the best result with ZA31 which is the zeolite synthesis at 95°C of 3 h, with the capacity of 0.1023 cc g^{-1} .

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