

Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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ABSTRACT

The purpose of this research is to synthesize zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent. This research used cetyltrimethylammonium bromide surfactant to invent the high porosity, surface area, acidity and thermal stability of synthesized zeolite. The Silica was extracted by alkaline fusion using sodium hydroxide solution. The synthesis was conducted by hydrothermal process at 100 °C for 7 days, ageing process for 24 h and calcination at 500 °C for 5 h. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950\text{--}1050\text{ cm}^{-1}$ and $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$, double ring at $520\text{--}570\text{ cm}^{-1}$, pore opening at $300\text{--}370\text{ cm}^{-1}$. Vibration of --OH as silanol group or water was indicated by broad absorbance at $3400\text{--}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO_2 quartz and contain 12.23% and 12.19% of Si, 4.17% and 13.18% of Al, respectively. Observation on SEM revealed that the crystal produced using cetyltrimethylammonium bromide was more homogenous and regular in shape.

Keywords: surfactant; cetyltrimethylammonium bromide; synthesis; zeolite; sugarcane bagasse

ABSTRAK

Tujuan penelitian ini adalah membuat zeolit dari ampas tebu menggunakan setiltrimetilamonium bromida sebagai agen pengarah struktur. Dalam penelitian ini surfaktan setiltrimetilamonium bromida digunakan untuk meningkatkan porositas, luas permukaan, keasaman dan stabilitas termal. Silika diekstrak dari abu ampas tebu melalui penambahan alkali yaitu NaOH 6 M. Sintesis dilakukan secara hidrotermal pada 100 °C selama 7 hari, ageing selama 24 jam dan kalsinasi pada 500 °C selama 5 jam. Variasi rasio silikat/aluminat (v/v) adalah 1, 15 and 25, sedangkan variasi konsentrasi surfaktan 5×10^{-4} M, 1×10^{-3} M dan 1×10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang $950\text{--}1050\text{ cm}^{-1}$, $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$. Vibrasi double ring pada $520\text{--}570\text{ cm}^{-1}$, pore opening $300\text{--}370\text{ cm}^{-1}$. Vibrasi gugus --OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada $3400\text{--}3450\text{ cm}^{-1}$. Data XRD menunjukkan bahwa produk sintesis mempunyai kristalinitas tinggi. Pada rasio Silikat/Aluminat 1 menghasilkan zeolite tipe sodalit sedangkan rasio 15 dan 25 menghasilkan NaP1 dan SiO_2 kuarsa. Pada rasio Si/Al 1 dan 25 berturut-turut mempunyai Si sebesar 12,23% dan 12,19% serta Al sebesar 4,17% and 13,18%. Hasil SEM menunjukkan bahwa produk sintesis yang menggunakan setiltrimetilamonium bromida mempunyai bentuk yang lebih homogen dan teratur.

Kata Kunci: surfaktan; setiltrimetilamonium bromida; sintesis; zeolit; ampas tebu

INTRODUCTION

Currently, there has been an attempt to utilize the large amount of bagasse ash, such as the residue from an in-line sugar industry and the bagasse-biomass fuel in electric generation industry [1]. Sugarcane bagasse is a solid waste produced in large amount from sugar mills. Sugarcane milling industry produced 35–40% of bagasse and Indonesia has large potential to produce

bagasse. Production of sugarcane in Indonesia was 53,612,133 tons [2]. The compositions of sugarcane product are 52.9% of liquid waste, 32% of bagasse, 4.5% of molasses, 7.05% of sugar and 0.1% ash. Generally sugarcane bagasse contained of 52.67% water, 55.89% organic carbon, 0.25% total nitrogen, 0.16% P_2O_5 and 0.38% K_2O . When this sugarcane bagasse is burned under controlled conditions, it also gives ash having amorphous silica. After sugarcane

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bagasse converted to ashes, the content of silica (SiO_2) to be 64.65% [3]. According to Norsurayaa et al. [4] the SiO_2 content in sugarcane bagasse ash from sugarcane juice industries in Shah Alam, Malaysia much higher, that was 88.19%. The consideration of a large number of sugarcane bagasse and the content of silica caused the development of new procedures for its productive reuse such as using as a source of silica. In the recent decades, many efforts are being carried out to explore other potential applications such as an adsorbent, fertilizers and cement mixture. So, the accumulation of waste and environment pollution can be avoided.

There are many methods applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources for synthesis of various zeolites. Synthesis of NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8-10], from illite-smectite [11], from aluminum solid waste [12]. NaX and NaY [13] NaY was synthesized from rice husk silica [14] and bentonite [15]. Synthesis of zeolite as micro-mesoporous materials has been developed continuously. As micro-mesoporous materials zeolite was expected possess ordered meso-structure, porous, high surface area, acidity and thermal stability. There are some variables which should be controlled in synthesis processes such as the ratio of Si/Al, temperature and time in hydrothermal treatment and the presence of structure directing agent (SDA). The most versatile variable can be used to influence the product of synthesis was structure directing agent, because they influenced on the nucleation and crystallization processes during the formation the framework of zeolite. Organic molecules as structure directing agent must have high chemical stability in order to resist in the hydrothermal condition of the crystallization process and must be soluble in synthesis medium [16].

Selection the properties of directing agent such as size, shape, flexibility and hydrophilicity can lead to the framework of zeolites have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD (N^1, N^1, N^3, N^3 -tetramethylpropane-1,3-diamine) and HMPD ($N^1, N^1, N^3, N^3, 2,2$ -hexamethylpropane-1,3-diamine) as linear and branch molecule [17]. Directing agent also used to control the growth of crystal [18].

Based on previously studies information, there have never been conducted researches using

cetyltrimethylammonium bromide (CTAB) surfactants as directing agent in synthesis of zeolite from sugarcane bagasse ash. Therefore, in this study, we report the using of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis of zeolite from sugarcane bagasse ash as source of silica. It would examine how the effect of the CTAB as directing agent on shape of crystalline and crystallinity of the product. As a comparison, zeolite from sugarcane bagasse ash in the absence of structure directing agent was also synthesized.

The important breakthroughs resulting from this research that this research is uplifting a low cost preparation of NaP1 zeolite from low cost materials. The results of this study are expected to increase the economic value of bagasse waste into high-value material, because these products had many potential applications such as an adsorbent, catalyst and fertilizers. In addition, this study also to contributing to the field of material synthesis with renewable resources. Finally by using bagasse as raw material to make zeolite then the accumulation of waste in the environment can be reduced.

EXPERIMENTAL SECTION

Materials

In this research sugarcane bagasse collected from sugarcane industry in the region of Klaten, Central Java. First step, Sugarcane bagasse was burned then ashed in furnace at 700 °C during 4 h. Furthermore, extraction by alkaline fusion step, 96 g of sugarcane bagasse ash was reacted with about 300 mL 6 M NaOH solution. The mixture was stirred at room temperature for 24 h and filtered. The supernatant was sodium silicate solution. Determination of the content of SiO_2 , Al_2O_3 and others oxide was conducted by AAS. NaOH pellets, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The sample functional group is determined by FTIR spectrometer Shimadzu. The diffraction patterns of the samples were measured by X-ray diffractometer (XRD) Rigaku Multiplex with Cu $K\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$) at generator voltage 40 kV and current 40 mA. The simultaneous scanning electron microscope (SEM) and Energy-Dispersive X-ray (EDX) were performed using JEOL JSM 6510/LV/A/LA. The BET surface areas, pore volume and average pore size of samples were determined from N_2 adsorption isotherms at liquid nitrogen temperature (-195.7 °C)

using a Quantachrome NovaWin2, Quantachrome Instrument version 2.2. The thermal gravimetric analysis (TGA) analysis of samples was determined using LINSEIS STA Platinum Series, Platinum evaluation V1.0.138.

Procedure

An early step was preparation of sodium aluminate solution from dilution of 20 g sodium hydroxide in 100 mL aquadest. The solution was boiled, then 8.5 g aluminium hydroxide was added into solution until homogenous solution achieved. For preparation of zeolite, a number of sodium silicate supernatant was mixed with sodium aluminate solution and cetyltrimethylammonium bromide surfactant (cationic surfactant) solution. The compositions of precursor and surfactant are provided in Table 1. The mixture was stirred at specific duration until gel formed, the process was continued by hydrothermal treatment in Teflon container at 100 °C during 7 days (168 h), then aging process at room temperature for 24 h. The final steps, the precipitates were filtered, washed, dried, and calcined at 500 °C for 5 h. The product was characterized by FTIR and XRD. The provision code in this research was given in Table 1.

RESULT AND DISCUSSION

In this research, content of silica and alumina in sugarcane bagasse ash is Si 70.82% and Al 1.04% respectively, this shows that the method of extraction with alkali NaOH 6 M is effective to extract Si and Al. Thus, sugarcane bagasse ash is a potential source of silica.

The success of zeolite synthesis is demonstrated by the fingerprint absorption of FTIR from the synthesized material. It was investigated synthesis of zeolite on various of composition of sodium silicate and sodium aluminate 1, 15 and 25 (v/v). The comparison between the properties of synthesized product before and after modified with surfactant as structure directing agent is needed to find out of the influence. The FTIR spectra of synthesis product were given in Fig. 1, 2, and 3.

It has been known that every type of zeolite has two kind of specific infrared vibration, they are internal and external linkage of tetrahedral (TO_4). In all products, there can be observed the internal linkage, there is asymmetric stretching vibration of TO_4 ($T = \text{Si}$ or Al) and appears at 950–1250 cm^{-1} wavenumber, whereas the symmetric stretching vibration at 650–720 and 500 cm^{-1} . Furthermore, the wavenumber at 1050–1150 cm^{-1} was asymmetric stretching vibration, 750–820 cm^{-1} is symmetric stretching vibration in the external linkage. All

Table 1. The composition of precursor and surfactant

Sample Code	Ratio of Precursor (v/v)		Surfactant CTAB (M)
	Sodium silicate	Sodium aluminate	
A	1	1	0
A1	1	1	5×10^{-4} (1/2 cmc)
A2	1	1	1×10^{-3} (cmc)
A3	1	1	1×10^{-2} (10 cmc)
B	15	1	0
B1	15	1	5×10^{-4} (1/2 cmc)
B2	15	1	1×10^{-3} (cmc)
B3	15	1	1×10^{-2} (10 cmc)
C	25	1	0
C1	25	1	5×10^{-4} (1/2 cmc)
C2	25	1	1×10^{-3} (cmc)
C3	25	1	1×10^{-2} (10 cmc)

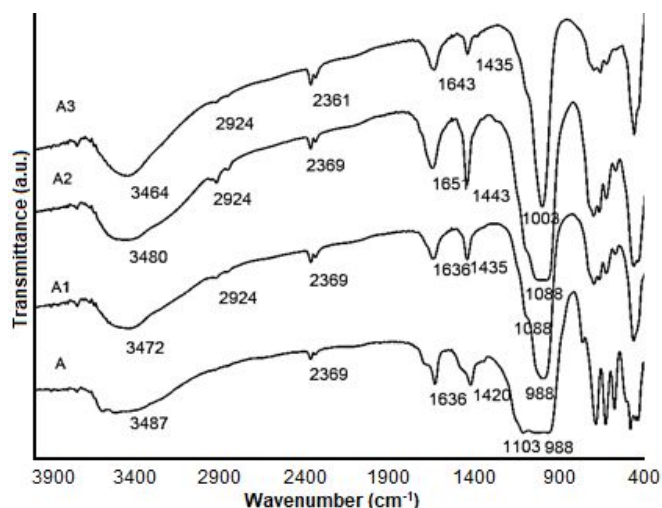


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

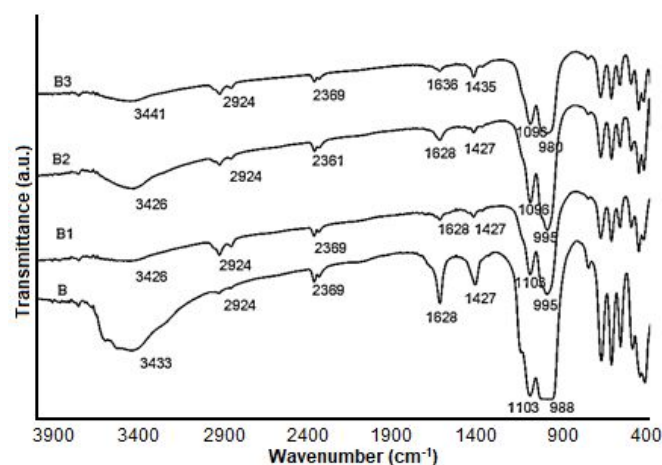


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

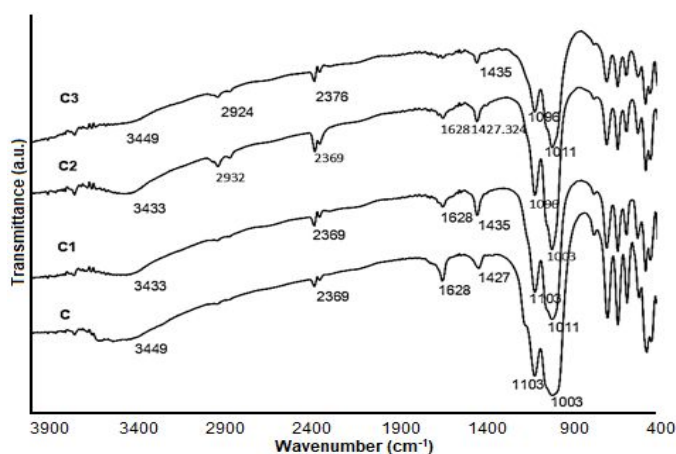


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5×10^{-4} M (C2) with CTAB 1×10^{-3} M (C3) with CTAB 1×10^{-2} M

of spectra were indicating that all the product of synthesis had Si-O-Si or Si-O-Al groups as a specific fingerprint of zeolite type. The absorption at $500\text{--}650\text{ cm}^{-1}$ is double ring [20]. Double ring is specific character of zeolite type, because it is an external linkage between polyhedral. From the FTIR data's, the zeolite was formed as synthesis product for all.

When the cetyltrimethylammonium (CTAB) as cationic surfactant is used as a directing molecule in the synthesis process of zeolite, so it would be interacted electrostatically with the silicate/ $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ anions primary building units. This interaction depends on surfactant concentration. The effect of surfactant presence can be observed on spectra A1, A2, and A3 in Fig. 1, B1, B2, and B3 in Fig. 2, C1, C2, and C3 in Fig. 3. It could be clearly observed that the presence of cetyltrimethylammonium (CTAB) surfactants has significantly affected the intensity and patterns of absorption at the wavenumber $950\text{--}1250$ and $3400\text{--}3500\text{ cm}^{-1}$. The lower absorption intensity of wave number at $3400\text{--}3500\text{ cm}^{-1}$ in the CTAB modified zeolite showed the low levels of silanol groups in the sample, where this corresponds to the formation of framework of zeolite. Besides that, the stronger bonding of Si-O-Si and Si-O-Al in zeolite framework characterized by increasing of intensity absorption bands at wave numbers $950\text{--}1250\text{ cm}^{-1}$ after the addition of CTAB.

In the FTIR spectra of zeolite modified by CTAB surfactant, no new absorption was observed. The spectra have similarity to the absorption region of zeolite which synthesized without surfactant. It was showed that the calcination treatment at 600°C to the modified zeolite by CTAB has removed and decomposed the CTAB as structure directing and all undesired organic compounds.

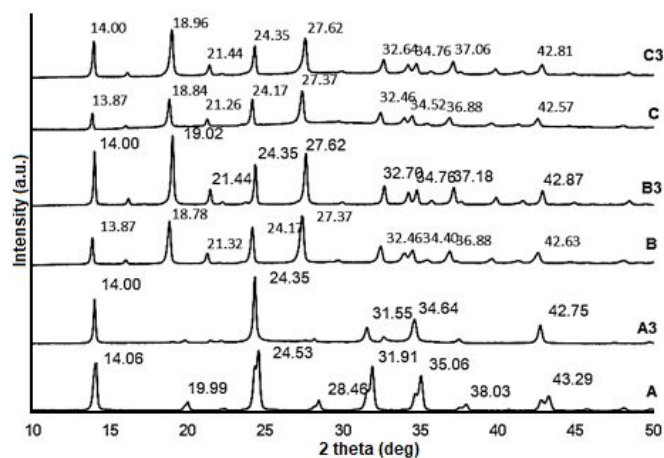


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Thus the shape and pore size of modified zeolite was expected homogeneously.

Fig. 4 shows the pattern of diffractogram XRD of synthesis product. Inorganic material has different shape and structure crystal, due to the diffractogram pattern was characteristic.

The intensity of diffractogram indicated the crystal quality of material. It could investigate the kind of synthesis product by CTAB different with synthesis product without CTAB. In addition, the composition of precursor also affected the kind of product. The synthesis product by ratio of Si/Al (v/v) 1 without CTAB was sodalite, because the XRD pattern has similarity to the sodalite standard in Treacy [21]. The sodalite is plain type of zeolite. The same product was obtained on ratio Si/Al (v/v) 1 used CTAB as structure directing. Although the product is the same but it is observed in XRD that the using of CTAB makes the peak of the diffractogram sharper and the product more pure.

If the ratio of Si/Al (v/v) increased to 15 or 25, the synthesis product was NaP1 zeolite. The presence of CTAB surfactant as structure directing was not affected on the type zeolite but on the main peak of diffractogram. The shifting of 2θ and the changing intensity of peaks at sodalite and NaP1 were known from the Fig. 4. All of the peaks on NaP1 diffractogram NaP1 as synthesis product modified by CTAB shifted to higher angle. Overall the intensity of peaks increased, thus it was concluded that the product modified by CTAB more crystalline.

Fig. 5 is SEM photograph corresponding to the synthesis product. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears

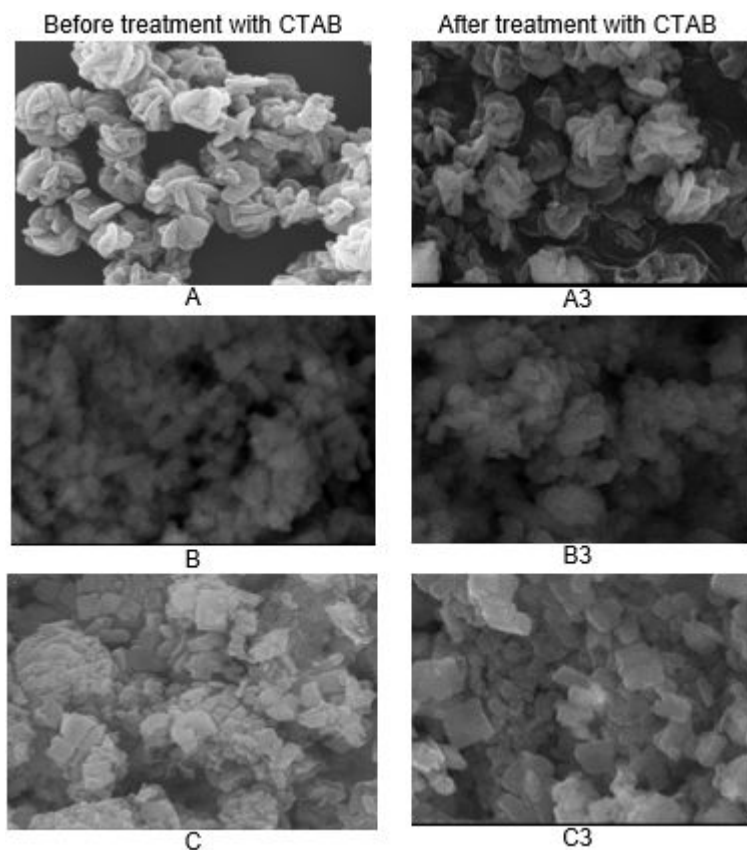


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

Element	Wt (%)					
	A1	A3	B1	B3	C1	C3
O	48.03	45.78	53.14	53.09	53.20	42.59
Na	5.25	17.96	12.65	14.05	13.80	18.49
Mg		00.25				0.34
Al	3.72	18.97	10.81	11.47	11.52	17.25
Si	3.26	17.04	11.18	11.69	11.90	16.6
S		-	1.68	1.74	1.63	3.57
K		-	1.67	0.44	0.55	1.16
Ca				0.16		

Description: (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

that the materials had regular spherical crystal and homogenous. At ratio Si/Al (v/v) 15 and 25 the materials had the same type, rectangular shape with thin platy crystal, however at ratio Si/Al (v/v) 15 has smaller in size. It showed different shape to the ratio Si/Al (v/v) 1. It could be investigated that the shape and size of granular/crystal depends on the composition of precursor, it means the ratio of Si/Al. The use of surfactant CTAB as a directing agent affects the size and homogeneity of the product particles. In SEM it appears that the crystalline product of synthesis with

CTAB has a more porous, regular and firm form crystal. The data's from SEM consistent with the result to the XRD, that the using of surfactant CTAB produce a more crystalline materials.

Meanwhile the chemical composition of products synthesis in Table 2. Sample A1 and A3 are sodalite as the product of synthesis. For sample B1, B3, C1, and C3 are NaP1. The entire product showed that ratio of Si/Al almost 1, these are consistent with the BET result that this product is micropore material. The use of surfactant CTAB as a pore directing agent increases the

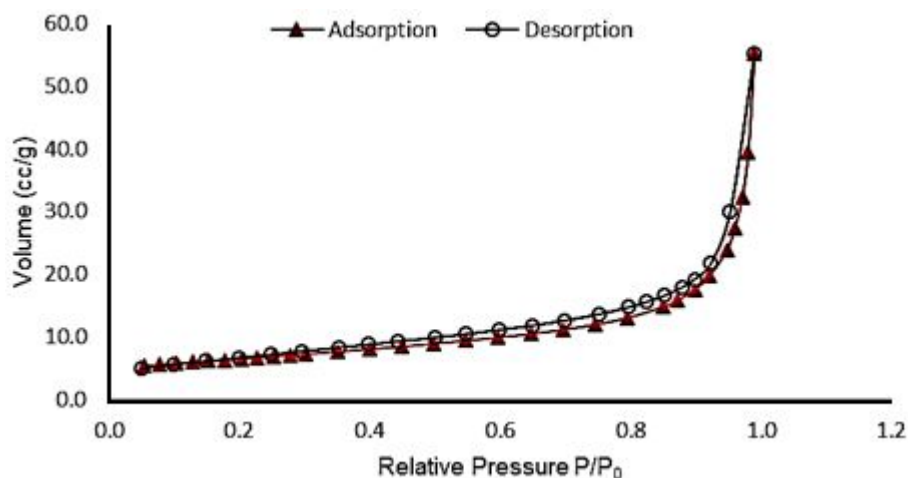


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

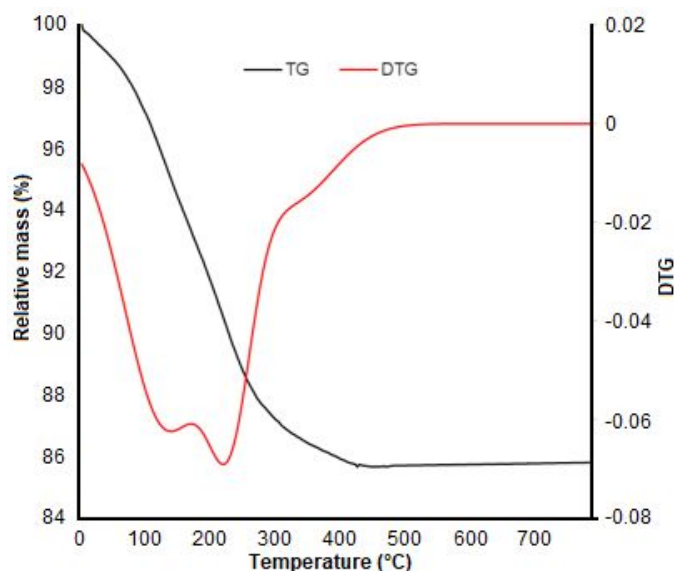


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

number of ions silicate and aluminate that interacted to form the product, so the using of surfactant is needed in this process. This is appropriate to the Wang [22] that the surfactants such as PEG (polyethylene glycol) and CTAB play an important role during the formation of nano crystal IM-5 zeolite.

BET (Brunauer–Emmett–Teller) method is used for multi molecular adsorption on solid surface, so the surface area of a material can be known with a surface area gauge that uses the BET method principle. The measurement of surface area with BET model used nitrogen as adsorbate. This measurement is based on isothermal adsorption data nitrogen at a temperature of 77.35 K. Isothermal adsorption with BET principle is a type physical isotherms. BET analysis is given in Fig. 6. The isotherm could be classified as type I of Brunauer's

classification showing closed hysteresis loops. The calculated surface area of the prepared zeolite is relatively low 22.062 m²/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56×10^{-2} cm³/g and average pore radius was determined as 7.737 Å. It means the NaP1 of this research has a micropore structure.

The TGA (thermogravimetric analysis) graph for the synthesized zeolite from sugarcane bagasse ash is given in Fig. 7. From the figure, it can be seen that the sample is lost the weight at temperature 120 °C, it means the loosely bond or free water molecules from product zeolite cage. Then the reducing weight also occurred at 270- which is due to the decomposition of the CTAB surfactant residues on the product zeolite surface.

CONCLUSION

In this study zeolite were successfully obtained from sugarcane bagasse ash by hydrothermal method at 100 °C for 168 h. The type of zeolite was determined by the ratio of Si/Al in term the SiO₄⁴⁻ and AlO₄⁵⁻ which formed the zeolite framework. The ratio Si/Al 1 (v/v) produced sodalite, while the ratio Si/Al 15 and 25 produced NaP1. The presence of CTAB surfactant micelles in synthesis zeolite from sugarcane bagasse ash could improve the homogeneity and crystallinity of the product zeolite. It means the product has homogeneous and orderly structure. But the introduction of CTAB micelles in the synthesis failed in terms of generating the mesopores structure because both of sodalite and NaP1 still had micropores structure.

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