Daftar Lampiran C1

1. Hasil penilaian sejawat sebidang atau peer review karya ilmiah
2. Tabel of conten jurnal “Indonesian Journal of Chemistry” Vol 18, No.1 (2018)
3. Bukti tim editor dari lebih 4 negara
4. Abstrak artikel dari 2 negara ke 1
5. Abstrak artikel dari 2 negara ke 2
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7. Bukti jurnal terindeks SCOPUS
8. Bukti abstrak artikel yang bersangkutan di SCOPUS
9. Bukti impac faktor (SJR) Scimago Journal & Country Rank
10. Artikel lengkap yang bersangkutan
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12. Bukti korespondensi (https://doc-pak.undip.ac.id/3073/2/C1_Kores.pdf)

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
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Judul Jurnal Ilmiah (Artikel)	:	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent			
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Status Pengusul	:	Penulis Utama			
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		b.	Nomor ISSN	:	1411-9420 (print); 2460-1578 (on-line)
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		d.	Penerbit	:	Gadjah Mada University
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☐ Jurnal Ilmiah Nasional Tidak Terakreditasi

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b. Ruang lingkup dan kedalaman pembahasan (30%)	12,00			12
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	12,00			12
d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)	12,00			11,6
Total = (100%)	**40,00**			**39,6**
Penulis Utama : 0,6 x 39,6 = 23,76				

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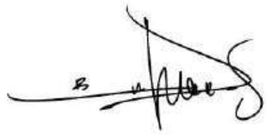
Semarang, 30 Maret 2020
Reviewer 1



Dr. Bambang Cahyono
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LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
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f. Ruang lingkup dan kedalaman pembahasan (30%)	12,00			12
g. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	12,00			12
h. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)	12,00			11
Total = (100%)	**40,00**			**39**
Penulis Utama: 0,6 x 39 = 23,4				

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   Lengkap dan sesuai dengan yang dipersyaratkan jurnal internasional.
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4. **Kelengkapan unsur dan kualitas terbitan:**
   Kualitas terbitan baik dan masuk dalam Q3, hanya ada beberapa gambar yang kurang jelas. Nilai 11

Semarang, 23 Maret 2020
Reviewer 2

Drs. Gunawan, M.Si, Ph.D
NIP.196408251991031001
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LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH : JURNAL ILMIAH

Judul Jurnal Ilmiah (Artikel) : Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
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Hasil Penilaian *Peer Review* :

Komponen Yang Dinilai	**Nilai Reviewer**		**Nilai Rata-rata**
	Reviewer I	**Reviewer II**	
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b. Ruang lingkup dan kedalaman pembahasan (30%)	12	12	12
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	12	12	12
d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)	11,6	11	11,3
Total = (100%)	**39,6**	**39**	**39,3**
Penulis Utama (rata-rata): 0,6 x 39,3 = 23,58			

Semarang, 30 Maret 2020

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Abstrak artikel 2 negara ke 1

Activated Carbon from Spent Brewery Barley Husks for Cadmium Ion Adsorption from Aqueous Solution

https://doi.org/10.22146/ijc.22422

Ilesanmi Osasona(1*), Kayode Aiyedatiwa(2), Jonathan Johnson(3), Oluwabamise Lekan Faboya(4)

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(3) Afe Babalola University, Ado - Ekiti
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Abstract

This study investigated the feasibility of using acid activated carbon prepared from brewery spent barley husks for the adsorption of cadmium from aqueous solution. The effects of operation parameters such as pH, contact time, adsorbent dosage, concentration and temperature were verified. The amount of cadmium adsorbed increased with increase in solution pH, initial solution concentration and with the amount of adsorbent dosed. A time of 5 minutes was required for attainment of equilibrium. The equilibrium data obtained were analysed using both Langmuir and Freundlich isotherm models and the data were better described by Langmuir model with correlation coefficient of 0.9183. The thermodynamic parameters revealed that the removal of cadmium by the activated carbon was exothermic and spontaneous. Thus, activated carbon obtained from brewery spent barley husk can be employed as an economically viable low-cost adsorbent for removing cadmium from aqueous solution.

Keywords

Cadmium;adsorbent; thermodynamic; equilibrium

Full Text:

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Abstrak artikel 2 negara ke 2

Improvement of Cycling Performance of $Na_{2/3}Co_{2/3}Mn_{1/3}O_2$ Cathode by PEDOT/PSS Surface Coating for Na Ion Batteries

https://doi.org/10.22146/ijc.24893

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Abstract

The surface-modified $Na_{2/3}Co_{2/3}Mn_{1/3}O_2$ is coated with a conductive Poly (3,4-Ethylene dioxy thiophene)-poly (styrene sulfonate) (PEDOT/PSS) polymer, and their resulting electrochemical properties were investigated as Na-ion battery cathode. The surface-modified $Na_{2/3}Co_{2/3}Mn_{1/3}O_2$ cathode material exhibits a high discharge capacity and good rate capability due to enhanced electron transport by surface PEDOT/PSS. The presence of PEDOT/PSS surface layer suppresses the growth of a resistive layer, while the dissolution of transition metals of the active cathode materials is inhibited as well. The resulting surface-modified $Na_{2/3}Co_{2/3}Mn_{1/3}O_2$ shows superior cycling performance, which is much stable than the pristine one as being the Na-ion battery cathode.

Keywords

Sodium ion battery; PEDOT/PSS; cathode; surface coating

Full Text:

References

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

https://doi.org/10.22146/ijc.22197

Sriatun Sriatun(1*), Taslimah Taslimah(2), Linda Suyati(3)

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(*) Corresponding Author

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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 were homogenous and regular in shape.

Keywords

surfactant; cetyltrimethylammonium bromide; synthesis; zeolite; sugarcane bagasse

Full Text:

References

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Sriatun, Taslimah, Suyati, L.

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Abstract

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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.

SciVal Topic Prominence

Topic: Coal Fly Ash | Zeolite | Kaolin

Prominence percentile: 96.330

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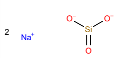

cetyltrimethylammonium

Author keywords

Cetyltrimethylammonium bromide | Sugarcane bagasse | Surfactant | Synthesis | Zeolite

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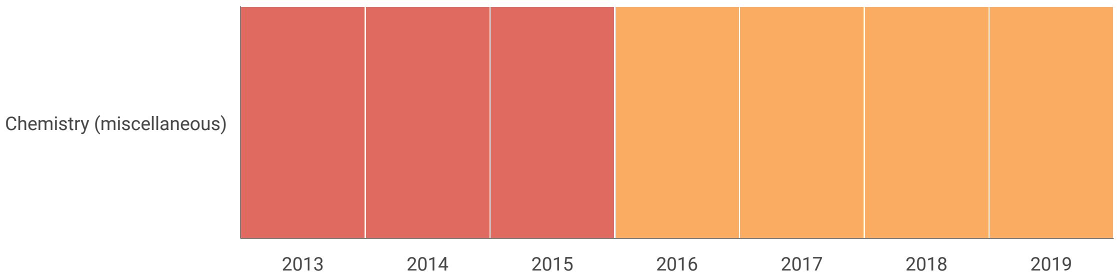

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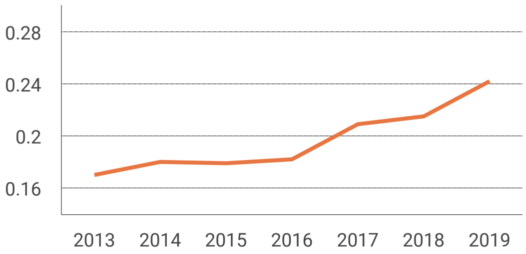

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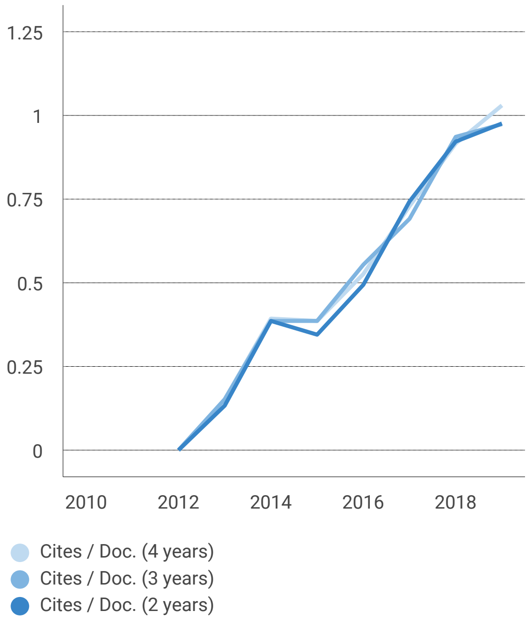

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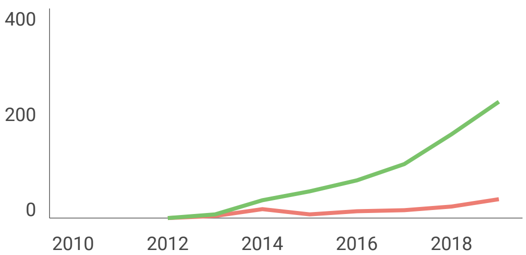

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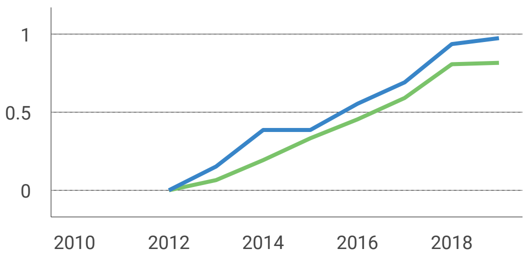

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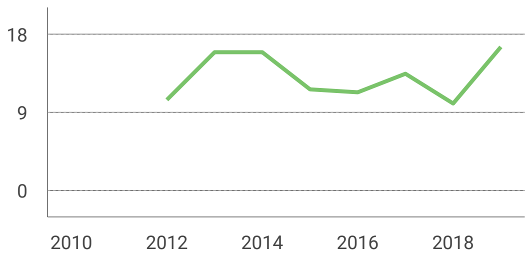

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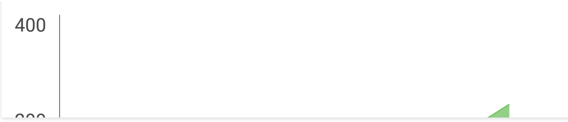

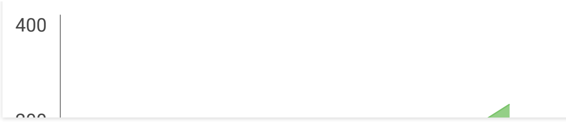

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Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300–370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 NaA [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 linier 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, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 = 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	$5x10^{-4}$ (1/2 cmc)
A2	1	1	$1x10^{-3}$ (cmc)
A3	1	1	$1x10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5x10^{-4}$ (1/2 cmc)
B2	15	1	$1x10^{-3}$ (cmc)
B3	15	1	$1x10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5x10^{-4}$ (1/2 cmc)
C2	25	1	$1x10^{-3}$ (cmc)
C3	25	1	$1x10^{-2}$ (10 cmc)

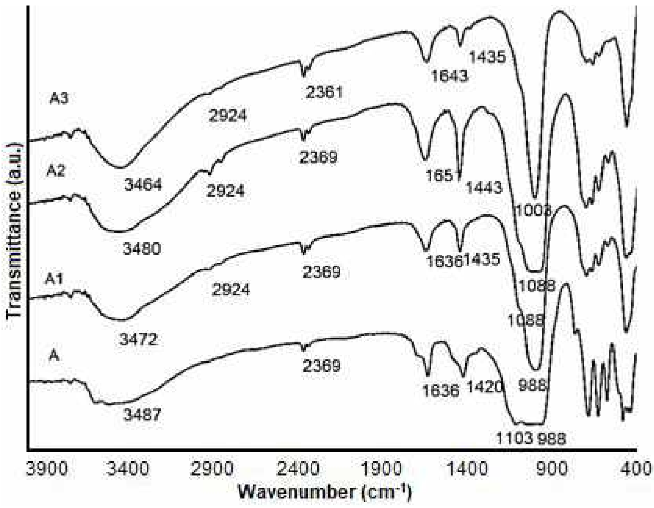

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB $5x10^{-4}$ M (A2) with CTAB $1x10^{-3}$ M (A3) with CTAB $1x10^{-2}$ M

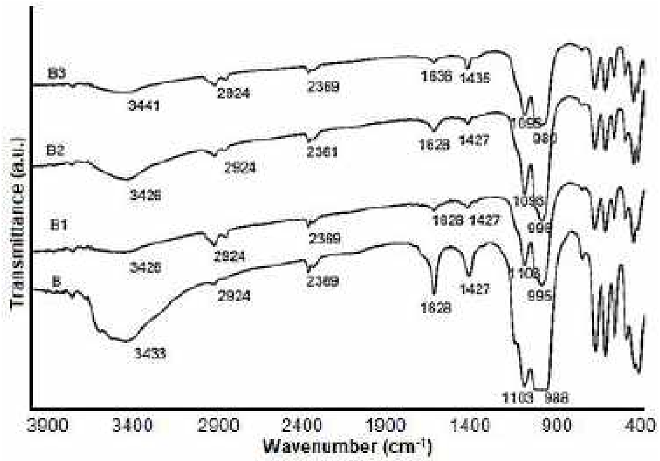

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB $5x10^{-4}$ M (B2) with CTAB $1x10^{-3}$ M (B3) with CTAB $1x10^{-2}$ M

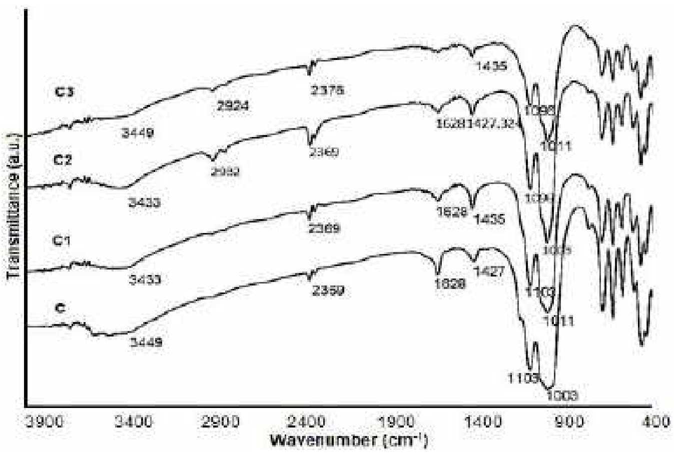

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5x10^{-4} M (C2) with CTAB 1x10^{-3} M (C3) with CTAB 1x10^{-2} M

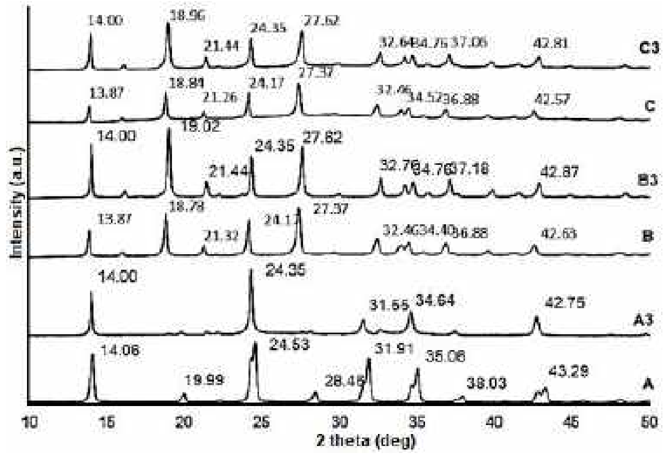

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1x10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1x10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1x10^{-2} M

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 500–650 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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

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

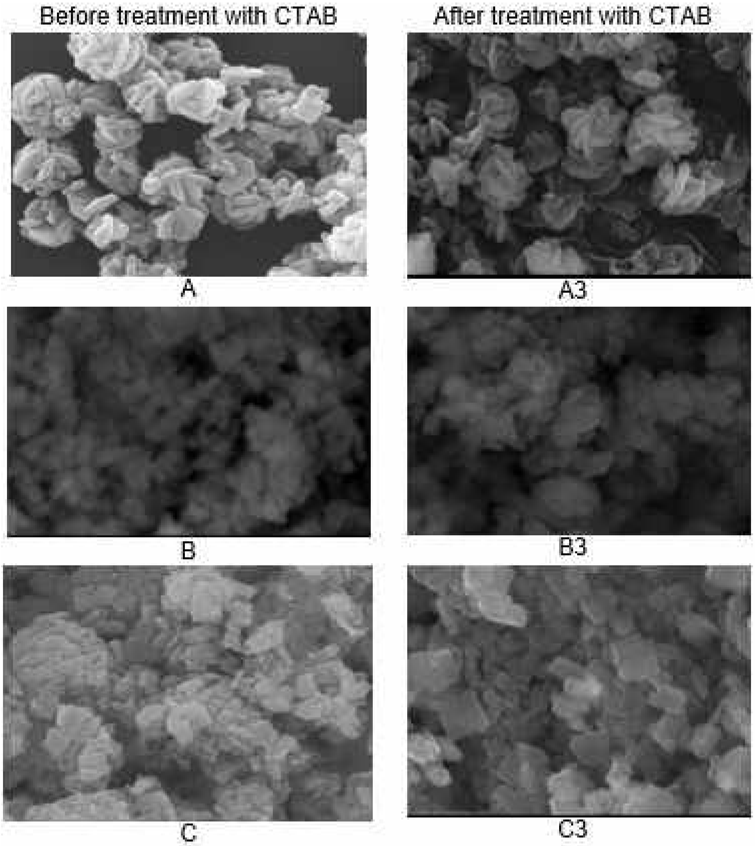

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ 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

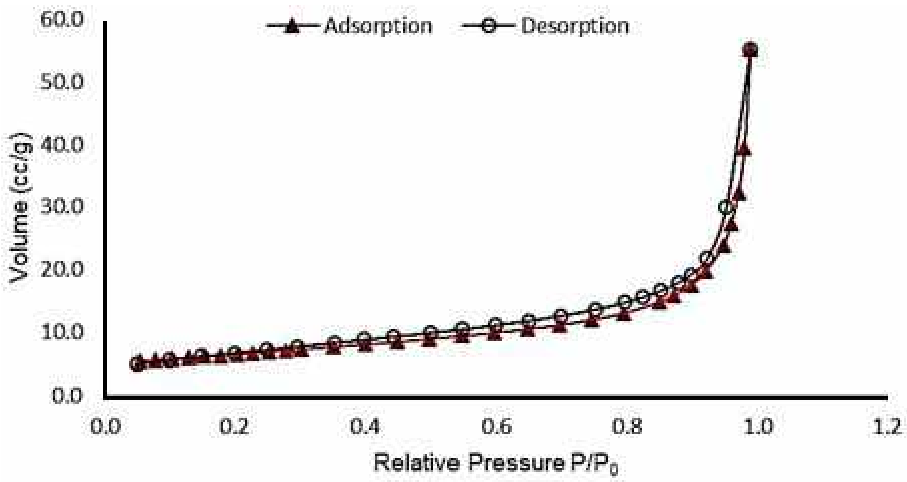

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

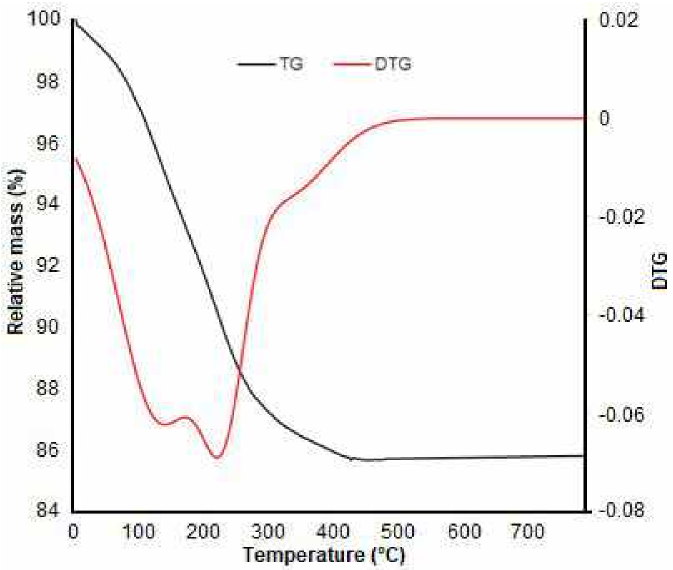

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56x10^{-2} cm^3/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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300–370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 NaA [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 linier 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, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 = 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) Sodium silicate	Sodium aluminate	Surfactant CTAB (M)
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)

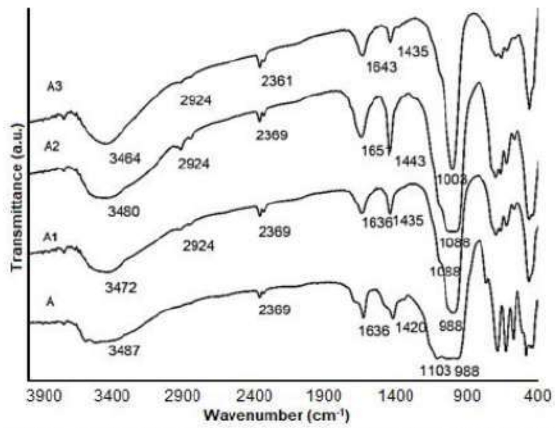

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

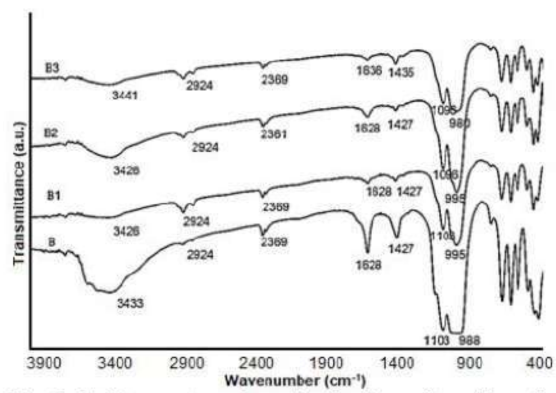

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

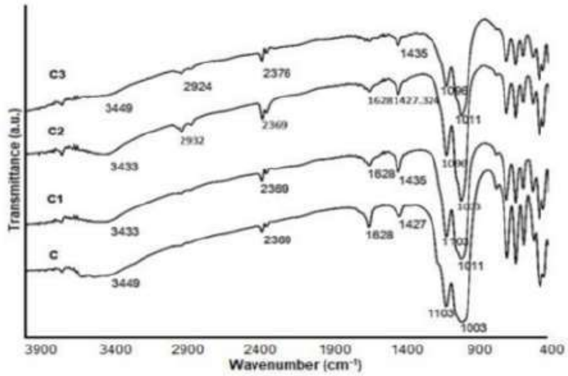

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5x10^{-4}$ M (C2) with CTAB $1x10^{-3}$ M (C3) with CTAB $1x10^{-2}$ M

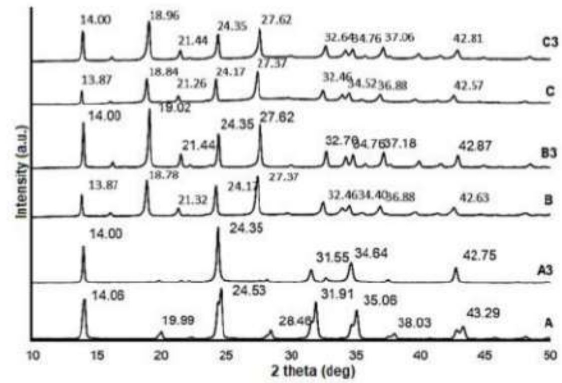

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 500–650 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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

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

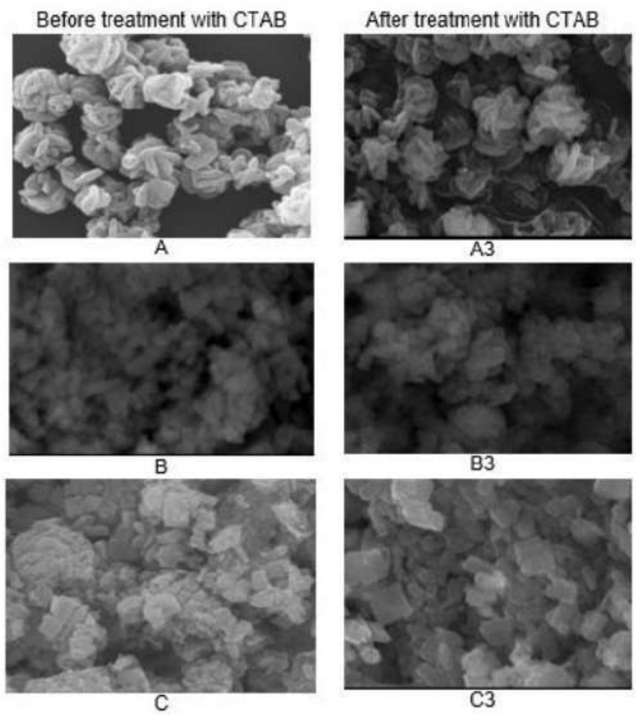

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ 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

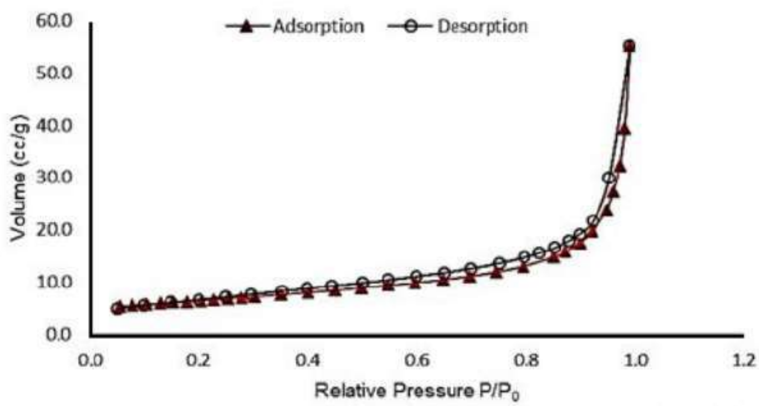

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

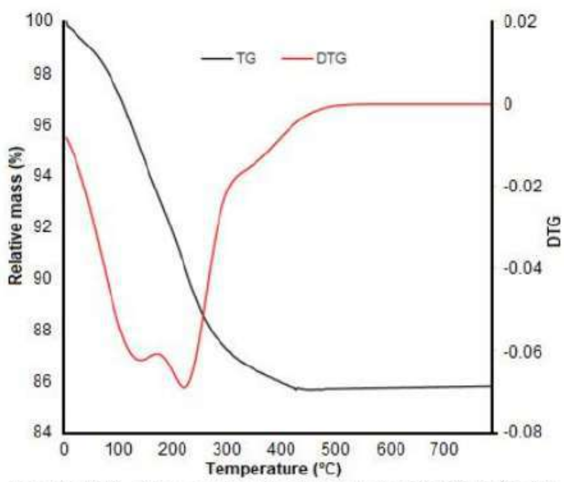

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56x10^{-2} cm^3/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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

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ABSTRACT

The effect of surfactant in synthesis of zeolite from sugarcane bagasse ash was studied. 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 and calcination at 500˚C for 5 hours. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5.10^{-4} M, 10^{-3} M and10^{-2} M. The result showed that all of products have strong absorbance at 950-1050 cm^{-1} and 620-690 cm^{-1}, 420-460 cm^{-1}, double ring at 520-570 cm^{-1}, pore opening at 300-370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400-3450 cm^{-1}. The XRD diffractograms 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 mixture of 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 were homogenous and regular in shape.

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

ABSTRAK

Telah dilakukan kajian pengaruh surfaktan setiltrimetilamonium pada sintesis zeolite dari ampas tebu. Dalam penelitian ini surfaktan setiltrimetilamonium 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 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, 10^{-3} M dan 10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950-1050 cm^{-1}, 620-690 cm^{-1}, 420-460 cm^{-1}. *Vibrasi double ring pada 520-570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400-3450 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 mempunyai bentuk yang lebih homogen dan teratur pada rasio silikat/Aluminat 1dan 25.*

Kata kunci: *surfaktan, setiltrimetilamonium bromida, sintesis, zeolit, ampas tebu*

INTRODUCTION

Currently, there have been various attempts 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, with sugarcane annual production of Indonesia was 53,612,133 tons [2], so Indonesia has large potential to produce bagasse. 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 residue is burned under controlled conditions, it will produce ash containing amorphous silica of about 64.65% [3]. According to Aida and Dewanti [4] the SiO_2 content in sugarcane bagasse ash from Tasik Madu sugar mills Industry slightly higher, that was 70.97%. Considering large number of sugarcane bagasse and the high content of silica, triggers the development of new procedures for its productive reuse, such as using as a source of silica. In the recent decades, many efforts have been 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 can be 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, condensation polymerization under hydrothermal condition may take place [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources to synthesis of various zeolite. For examples, NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8, 9, 10], from illite-smectite [11], from aluminum solid waste [12]. Purnomo *et al.* [13] was synthesized NaX and NaA. Meanwhile NaY from rice husk silica was conducted by Jatuporn *et al.* [14] and from bentonite by Faghihian and Godazandeha [15]. Synthesis of zeolite as micro-mesoporous materials has been developed continously. Micro-mesoporous materials zeolite was expected possess ordered mesostructure, porous, high surface area, acidity and thermal stability. There are some variables 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)/template. The most versatile variable to influence the product of synthesis was SDA, because they influenced on the nucleation and crystallization processes during the formation the framework of zeolite. The organic molecule as SDA must have high chemical stability in order to resist in the hydrothemal condition of the crystallization process and must be soluble in synthesis medium [16].

Selection the properties of SDA such as size, shape, flexibility and hydrophilicity can lead to the framework of zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier and branch molecule [17]. In addition SDA also used to control the growth of crystal [18].

In this study, we report the influence of cetyltrimethylammonium bromide as structure directing agent in synthesis zeolite using sugarcane bagasse ash as source of silica.

EXPERIMENTAL SECTION

Materials

In this research sugarcane bagasse collected from sugarcane industry in the region of Klaten, Central of Java. First step, sugarcane bagasse was burned then the char was ashed in furnace at 700^{o}C during 5 hours. Futhermore, 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 hours and filtered. The supernatant was sodium silicate solution. Determination of SiO_2, Al_2O_3 dan others oxide was conducted by AAS. NaOH pellets, $Al(OH)_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The equipment for analysis were used in this research: FTIR spectrometer Nicolet Avatar 360 IR, X-Ray Diffractometer (XRD) Shimadzu, Scanning Electron Microscope (SEM) FEI Inspect S50, Energy-Dispersive X-ray (EDX) spectroscopy EDAX AMETEX.

Procedure

An early step was preparation of sodium aluminate solution from dillution of 20 g sodium hydroxide in 100 mL aquadest. The solution was boiled, then 8,5 g aluminium hydroxide was added into solution to form homogenous solution. The next step is preparation of zeolite. The predetermined amount of sodium silicate supernatant was mixed with sodium aluminate solution and cetyltrimethylammonium bromide surfactant (cationic surfactant) solution. The compositions of precursor and concentration of 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^{o}C during 7 days, then aging process at room temperature for 24 hours. The final steps, the precipitates were filtered, washed , dried and calcined at 500^{o}C for 5 hours. The products were characterized by FTIR and XRD. The provision code in this research was given in Table 1.

RESULTS AND DISCUSSION

The aim of the ashing of sugarcane bagasse at 700^{o}C is removing the organic compounds and changing the inorganic compounds to metal oxide such as SiO_2, Al_2O_3, MgO, CaO, Fe_2O_3 and others. The content of silica and alumina the sugarcane bagasse ash is Si 70.82% and Al 1.04% respectively. This result is much higher than the results of the extraction silica by acid treatment, where silica content is only reached 21.053% at 700^{o}C [19]. It means that the extraction by alkaline is better than acid, although the silica contents of bagasse and its ash are varied depending on the type of soil and harvesting.

The purposes of temperature adjusting at 100°C for 168 h on hydrothermal process was to control the rate of nucleation and crystallization. The crystallization times (1, 2,3,4,6 and 8 hours) on formation of zeolite A under 373 K, time of hydrothermal process significantly influenced the crystallinity degree [20]. Actually, in this process the silica alumina framework was formed. The crystallinity of zeolite increased with the increasing of hydrothermal time. Meanwhile, ageing time during 24 hours after hydrothermal process for arranged and established the framework structure of zeolite.

Generally, the framework of zeolite had a negative charge, so it was neutralized by some cation. In this case, the surfactant such as cetyltrimethylammonium was used for structure directing of material, so the product would have different size and shape of pore and more homogenous. Cetyltrimethylamonium as cationic surfactant would have electrostatic interaction with the negative charge in silicate aluminate framework from zeolite.

Cetyltrimethylammonium, TPAOH and PPDA surfactant were used as structure directing agents in synthesis zeolite [21]. The cetyltrimethylammonium (CTA) is cationic surfactant, the presence of cationic surfactant in solution would react rapidly with silicate $[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ ions primary building units. Furthermore, they occurred condensation polymerization and formed zeolite embryo. Surfactant as SDA determined the type of polyhedral as which built the structure of zeolite completely. The illustration of mechanism given in Figure 1.

Finally, the CTA^+ surfactant as SDA was disappeared from the zeolite structure by calcination at 500°C during 5 hours. The surfactant would be decomposed into gas, and leaving pores of materials.

Characterizations of product

Synthesis of zeolite on various of composition of sodium silicate and sodium aluminate 1, 15 and 25 (v/v) has been investigated. The comparison between the properties of synthesized product before and after modified with surfactant is necessary to find out of the effect of modification. The FTIR spectra of products given in Figure 2a, 2b and 2c.

It well known that every type of zeolite has two kind of specific infrared vibration, they are internal and external linkage of tetrahedral (TO_4). In the internal linkage, there is asymmetric stretching vibration of TO_4 (T = Si or Al) and appears at 950-1250 cm^{-1} wavenumber, whereas the symetric stretching vibration at 650-720 cm^{-1} and 500 cm^{-1}. Furthermore, the wavenumber at 1050-1150 cm^{-1} was asymmetric stretching vibration, 750-820 cm^{-1} is symetric stretching vibration in the external linkage. All of spectra indicated that all the products had Si-O-Si or Si-O-Al groups as a specific fingerprint of zeolite type, but the presence of CTA surfactant did not affect significantly on the function group of product. The absorption at 300-420 cm^{-1} is pore opening and 500-650 cm^{-1} is double ring [22]. Double ring is specific character of zeolite type, because it was an external lingkage between polyhedral. From the FTIR data, the zeolite was successfully formed as product.

The zeolite which was modified by CTA surfactant has similarity to the absorption region of zeolite synthesized without surfactant. It was showed that the calcination treatment at 500^{o}C to the modified zeolite by CTA has removed and decomposed the CTA as structure directing agent and all undesired organic compounds. Thus the shape and pore size of modified zeolite was expected to be homogenous.

Figure 3 shows the pattern of XRD diffractogram of product synthesis. Every inorganic material has specific shape and structure crystal, as characterized by the diffractogram patterns.

The instensity of diffractogram indicated the crystal quality of material. It could be observed that type of product synthesized by CTA is different with that synthesized without CTA. In addition, the composition of precursor also affected the type of product. The product obtained from synthesis by ratio of Si/Al (v/v) 1 using CTA as structure directing agent was sodalite, it is a plain type of zeolite. The same product was obtained on ratio Si/Al (v/v) 1 without CTA. If the ratio of Si/Al (v/v) increased to 15 or 25, the product synthesis was NaP1 zeolite. The presence of CTA surfactant as structure directing did 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 Figure 3. All of the peaks on NaP1 diffractogram which synthesized by CTA shifted to higher angle. Overall the intensity of peaks increased, thus it was concluded that the product modified by CTA was more crystalline.

Figure 4 is SEM photograph corresponding to the product synthesis by ratio Si/Al (v/v) 1 as sodalite and 25 as NaP1. Both of the materials had regular crystal and homogenous. It can be observed that the shape and size of granular/crystal depends on the composition of precursor, it means the ratio of Si/Al. Meanwhile the chemical composition of the synthesized zeolite are shown in Table 2. Sample A3 that is synthesized zeolite with ratio Si/Al (v/v) 1 and concentration of CTA 1.10^{-2} M was sodalite that containing 17.96 % Na, 18.97% Al and 17.04% Si. For sample C3 with ratio Si/Al 25 (v/v) was NaP1contained 18.49 % Na, 17.25% Al 16.60% Si. The decreasing of silica in the product NaP1 was caused calcination treatment at 500^{o}C. The remainder of silica was transformed to be SiO_2 quartz.

Conclusion

The presence of CTA surfactant as structure directing at concentration 10 times CMC of CTA did not affect the type zeolite, but shifted slightly the 2 theta to higher angle. The zeolite modified by CTA are more crystalline than that obtained without CTA.

Acknowledgments

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Table 1. The composition of precursor and concentration of surfactant

Sample Code	Ratio of Precursor (v/v)		Concentration of 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)

Table 2. The chemical composition of synthesized zeolite

Element	***Sample A3***		***Sample C3***	
	Wt %	***At %***	***Wt %***	***At %***
O	45.78	57.66	42.59	54.87
Na	17.96	15.74	18.49	16.57
Mg	00.25	00.20	0.34	0.29
Al	18.97	14.17	17.25	13.18
Si	17.04	12.23	16.6	12.19
S	-	-	3.57	2.3
K	-	-	1.16	0.61

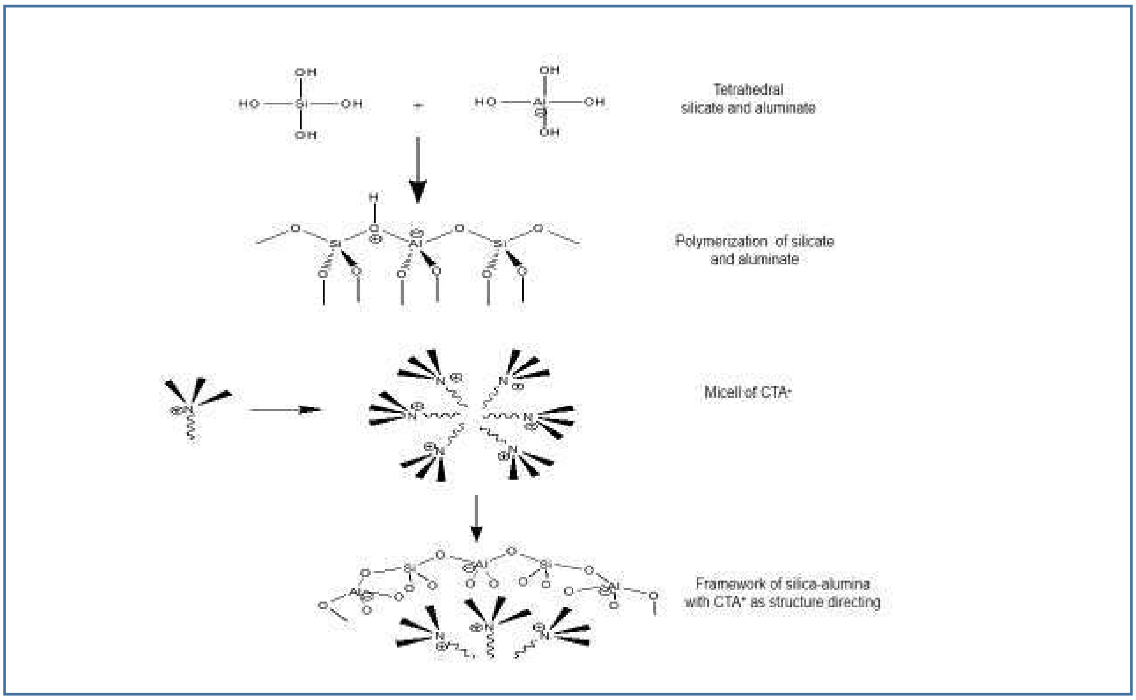

Figure 1. The mechanism of zeolite framework formation by CTA^{+} as structure directing

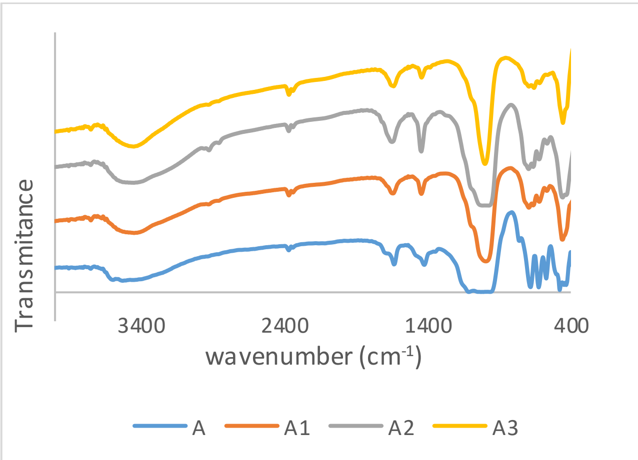

Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTA surfactant (A1) with CTA 5.10^{-4}M (A2) with CTA 1.10^{-3} M (A3) with CTA 1.10^{-2} M

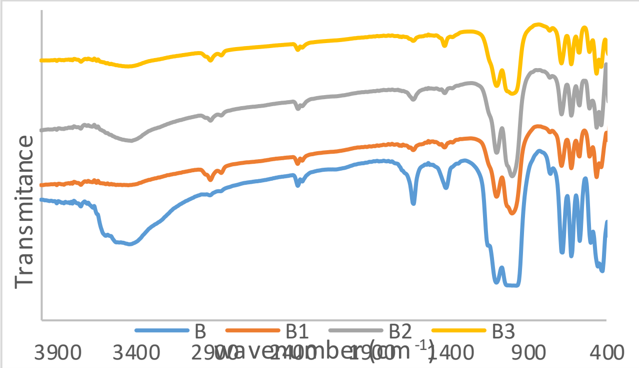

Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTA surfactant (B1) with CTA 5.10^{-4}M (B2) with CTA 1.10^{-3} M (B3) with CTA 1.10^{-2} M

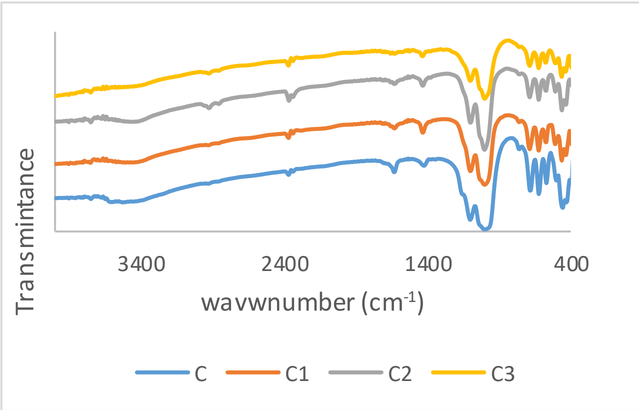

Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTA surfactant (C1) with CTA 5.10^{-4}M (C2) with CTA 1.10^{-3} M (C3) with CTA 1.10^{-2} M

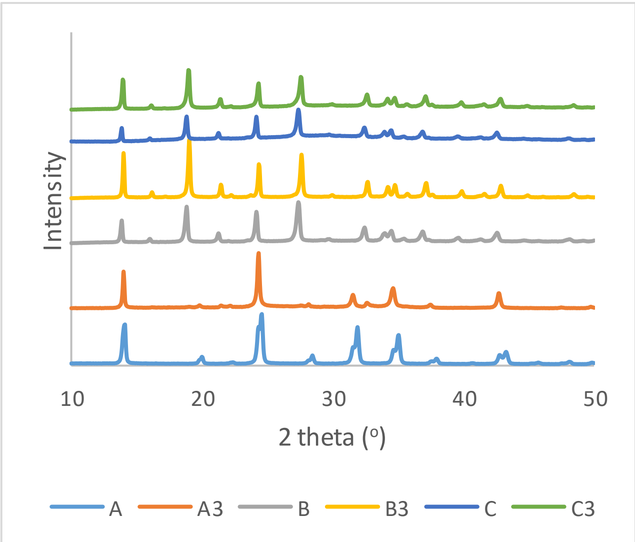

Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTA (A3) Ratio Si/Al 1 with CTA 1.10^{-2}M (B) Ratio Si/Al 15 without CTA (B3) Ratio Si/Al 15 with CTA 1.10^{-2}M (C) Ratio Si/Al 25 without CTA (C3) Ratio Si/Al 25 with CTA 1.10^{-2}M

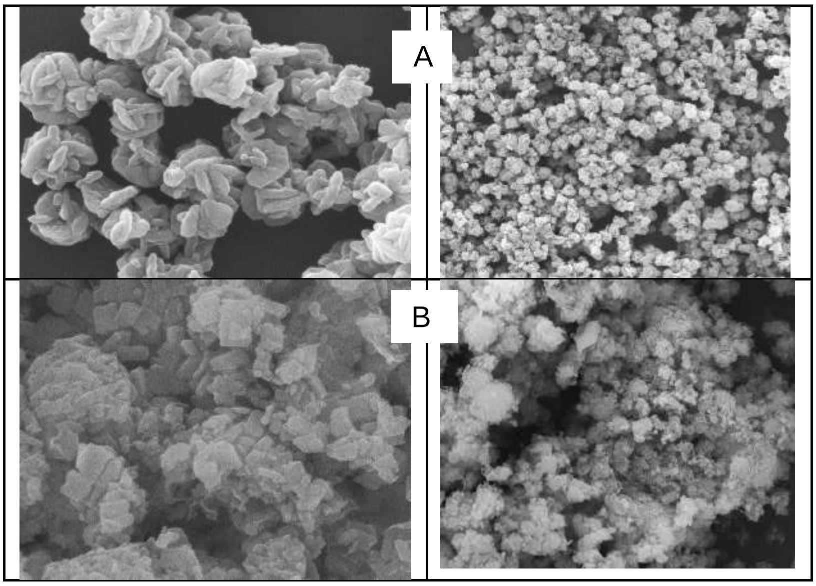

Figure 4. The SEM image of synthesized zeolite (A) Ratio Si/Al (v/v) 1 (B) Ratio Si/Al (v/v) 25

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The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

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ABSTRACT

The effect of surfactant in synthesis zeolite from sugarcane bagasse ash was studied. 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 hours and calcination at 500˚C for 5 hours. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5.10^{-4} M, 10^{-3} M and10^{-2} M. The result showed all of product have strong absorbance at 950-1050 cm^{-1} and 620-690 cm^{-1}, 420-460 cm^{-1}, double ring at 520-570 cm^{-1}, pore opening at 300-370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400-3450 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 was known that the crystal shape of product on ratio Si/Al 1 and 25 with concentration of cetyltrimethylammonium 10^{-2} M was homogenous and orderly.

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

ABSTRAK

Telah dilakukan kajian pengaruh surfaktan setiltrimetilamonium pada sintesis zeolite dari ampas tebu. Dalam penelitian ini surfaktan setiltrimetilamonium 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, 10^{-3} M dan 10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950-1050 cm^{-1}, 620-690 cm^{-1}, 420-460 cm^{-1}. *Vibrasi double ring pada 520-570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400-3450 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 mempunyai bentuk yang lebih homogen dan teratur pada rasio silikat/Aluminat 1dan 25.*

Kata kunci: *surfaktan, setiltrimetilamonium bromida, sintesis, zeolit, ampas teb*

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 producted 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 bagasse converted to ashes, the content of silica (SiO_2) to be 64.65% [3]. According [4] the SiO_2 content in sugarcane bagasse ash from Tasik Madu sugar mills Industry slightly higher, that was 70.97%. 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 applicated 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 to synthesis of various zeolite. Synthesis of NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8, 9, 10], from illite-smectite [11], from aluminum solid

waste [12]. NaX and NaA [13] NaY was synthesized from rice husk silica [14] and bentonite [15]. Synthesis of zeolite as micro-mesoporous materials has been developed continously. As micro-mesoporous materials zeolite was expected possess ordered mesostructure, 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 molecul as structure directing agent must have high chemical stability in order to resist in the hydrothemal 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 zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier and branch molecule [17]. Directing agent also used to control the growth of crystal [18].

Based on previously studies information, there has never been conducted researches using CTAB surfactants as directing agent in synthesis of zeolite from sugarcane bagasse ash. Therefore, in this study, we report the influence of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis zeolite using 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 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 waste bagasse into material

into high-value material, because this 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 of Java. First step, Sugarcane bagasse was burned then ashed in furnace at 700^{o}C during 4 hours. Futhermore, 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 hours and filtered. The supernatant was sodium silicate solution. Determination the content of SiO_2, Al_2O_3 dan others oxide by AAS. NaOH pellets, $Al(OH)_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The equipment were used in this research: stirrer, bar magnetic stirrer, oven, furnace, grinder were used on synthesis process. FTIR spectrometer Nicolet Avatar 360 IR, X-Ray Diffractometer (XRD) Rigaku Multiplex, Scanning Electron Microscope (SEM) FEI Inspect S50, Energy-Dispersive X-ray (EDX) spectroscopy EDAX AMETEX,

Procedure

An early step was preparation of sodium aluminate solution from dillution of 20 g sodium hydroxide in 100 mL aquadest. The solution was boiled, then 8,5 g alumunium hydroxide was added into solution until homogenous solution achieved. The next step is preparation 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 spesific duration until gel formed, the process was continued by hydrothermal treatment in teflon container at 100°C during 7 days, then aging process at rom temperature for 24 hours. The final steps, the precipitates were filtering, washing , drying and calcinating at 500°C for 5 hours. The product characterized by FTIR and XRD. The provision code in this research was given in table 1.

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)

See in article vol .17 how tables are formatted

RESULTS AND DISCUSSION

The aim of the ashing of sugarcane bagasse at 700°C is removing the organic compounds and changing the inorganic compounds to metal oxide such as SiO_2, Al_2O_3, MgO, CaO, Fe_2O_3 and others. The content of silica and alumina the sugarcane bagasse ash is Si 70.82% and Al 1.04% respectively.

The purposes of temperature adjusting at 100°C for 168 h on hydrothermal process controlled the rate of nucleation and crystallization. The effect of crystallization times (1, 2,3,4,6 and 8 hours) on formation of zeolite A under 373 K has been known that the long time of hydrothermal process influenced the crystallinity degree [19]. Actually, in this process the silica alumina framework was formed. The crystallinity of zeolite increased with the increasing of hydrothermal time. Meanwhile, ageing time during 24 hours after hydrothermal process for arranged and established the framework structure of zeolite.

Several factors that influence in the synthesis process of zeolite are the type of precursor, reactant, volume of surfactant, temperature of reaction, aging time and organic template. Generally, the framework of zeolite had a negative charge, so it was neutralized by some cation. In this case, the surfactant such as cetyltrimethylammonium was used for structure directing of material, so the product would have different size and shape of pore and more homogenous. Cetyltrimethylamonium as cationic surfactant would be interacted electrostatic with the negative charge in silicate aluminate framework from zeolite.

Cetyltrimethylammonium, TPAOH and PPDA surfactant were used as structure directing agents in synthesis zeolite [20]. The cetyltrimethylammonium (CTA) is cationic surfactant, the presence of cationic surfactant in solution would be reacted rapidly with silicate/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ ions primary building units. Furthermore, they occurred condensation polymerization and formed zeolite embryo. Surfactant as structure directing determined the type of polyhedral as which built the structure of zeolite completely. The illustration of mechanism given in Figure 1.

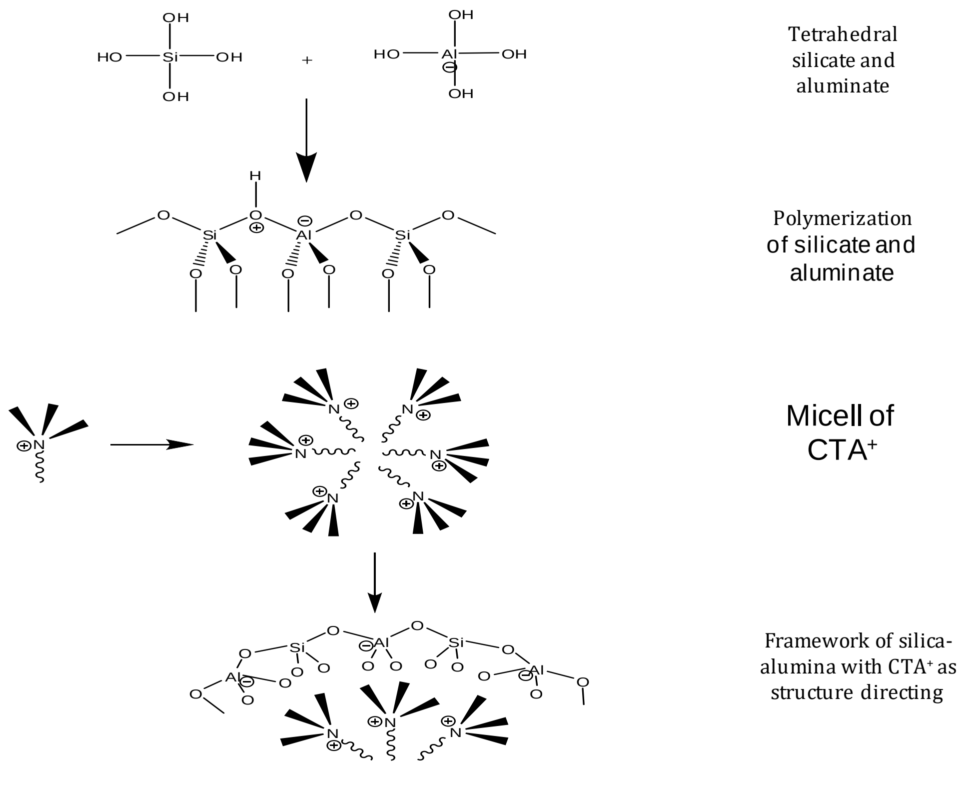

Figure 1. The mechanism of zeolite framework formation by CTA^+ as structure directing (the letters in figure are too small, unreadable)

Finally, the CTAB surfactant as structure directing was disappeared from the zeolite structure by calcination at 500°C during 4 hours. The surfactant would be decomposed to be gas, and leaved hole. This hole was pore of materials. Above explanation is discussion, not result

Characteristics~~zations~~ of products

It was investigated synthesis 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 is needed to find out of the influence. The FTIR spectra of product synthesis given in Figure 2a, 2b and 2c.

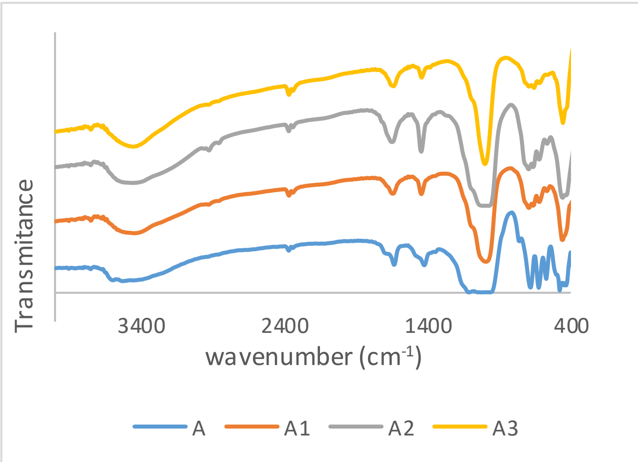

Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4}M (A2) with CTAB 1.10^{-3} M (A3) with CTAB 1.10^{-2} M
Should the curves be color? The lines and scale are not clear/contract

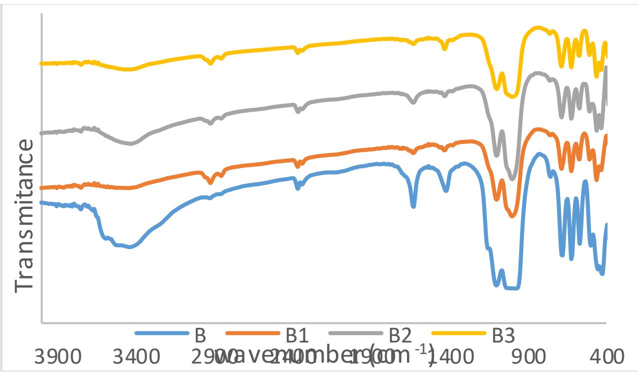

Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4}M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M

Should the curves be color? The lines and scale are not clear/contract

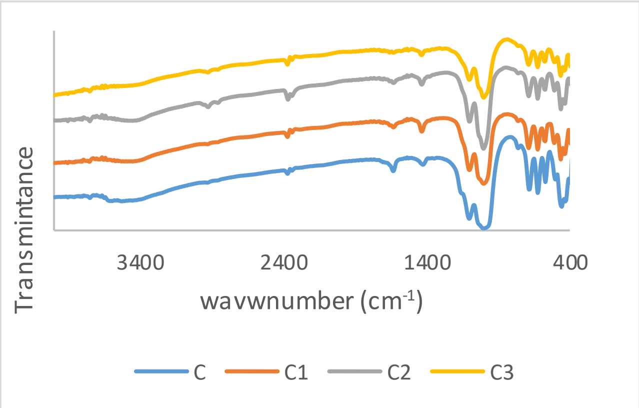

Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5.10^{-4}M (C2) with CTAB 1.10^{-3} M (C3) with CTAB 1.10^{-2} M Should the curves be color? The lines and scale are not clear/contract. a.u. should be used for transparence unit.

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 the internal linkage, there is asymmetric stretching vibration of TO_4 (T = Si or Al) and appears at 950-1250 cm^{-1} wavenumber, whereas the symetric stretching vibration at 650-720 cm^{-1} and 500 cm^{-1}. Furthermore, the wavenumber at 1050-1150 cm^{-1} was asymmetric stretching vibration, 750-820 cm^{-1} is symetric stretching vibration in the external linkage. All 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 300-420 cm^{-1} is pore opening and 500-650 cm^{-1} is doublé ring [21]. Doublé ring is specific character of zeolite type, because it is an external lingkage between polyhedral. From the FTIR datas, the zeolite was formed as product synthesis. The presence of Cetyltrimethylammonium surfactants has significantly affected the intensity and patterns of absorption at the wavenumber 950-1250 cm^{-1} and 3400-3500 cm^{-1}. The lower absorption intensity of wave number at 3400-3500 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 zeolit. Beside 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 950-1250 cm^{-1} after the addition of CTAB.

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

Figure 3 shows the pattern of diffractogram XRD of product synthesis. Inorganic material has different shape and structure crystal, due to the diffractogram pattern was characteristic.

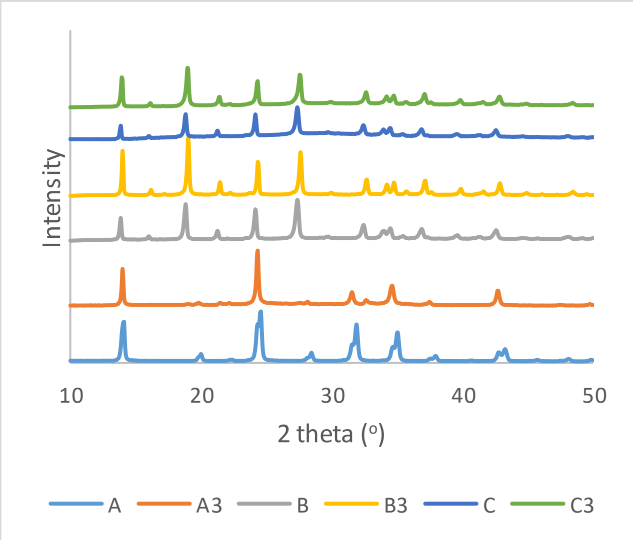

Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2}M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2}M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB1.10^{-2}M

Should the curves be color? The lines and scale are not clear/contract

The instensity of diffractogram indicated the crystal quality of material. It could investigated the kind of product synthesis by CTAB different with product synthesis without CTAB. In addition, the composition of precursor also affected the kind of product. The product synthesis by ratio of Si/Al (v/v) 1 without CTAB was sodalite. 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 product synthesis 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 Figure 3. All of the peaks on NaP1 diffractogram NaP1 as product synthesis 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.

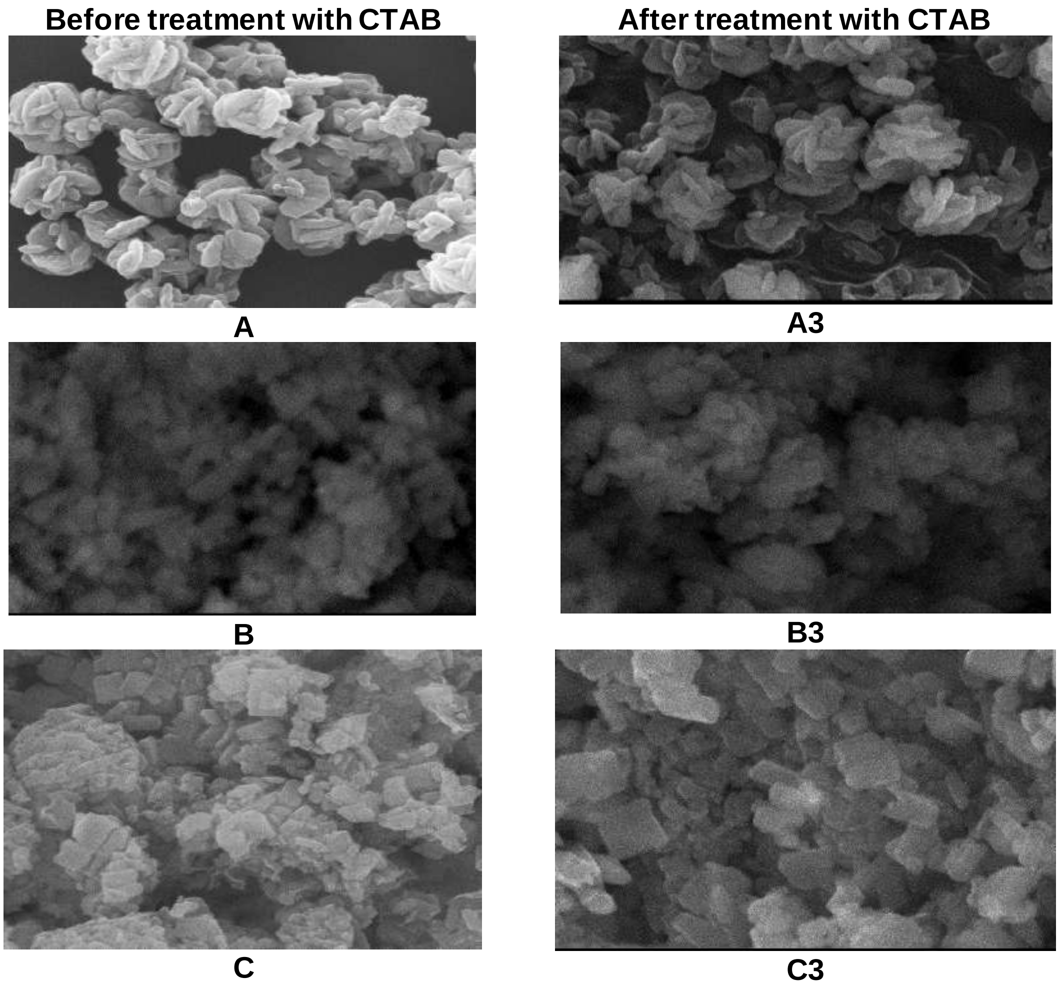

Figure 4. The SEM image of synthesized zeolite

Figure 4 is SEM photograph corresponding to the product synthesis. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears 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 diffrerent 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 datas 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. All of the product showed that ratio of Si/Al almost 1, these is consistent with the BET result that this product is micropore material. The use of surfactant CTAB as a pore directing agent increases the number of ions silicate and aluminate that interacted to form the product.

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		

See the published article in Vol 17 to format Table

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 Figure 4. 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 13.508 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be $5.28x10^{-3}$ cm^3/g and average pore radius was determined as 2.261°A. It means the NaP1 of this research have a micropore structure.

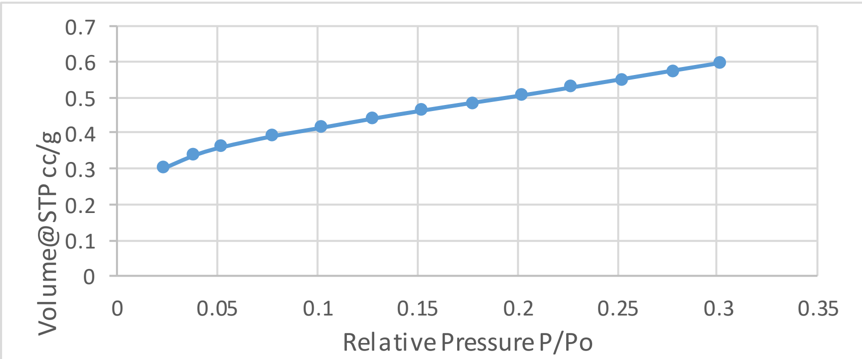

Figure 4. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

Should the curves be color? See the published article in Vol 17 to format curve (without grill).

The TGA (thermogravimetric analysis) graph for the synthesized zeolite from sugarcane bagasse ash is given in Figure 5. From the figure, it can be seen that the sample is lost the weight at temperature 100°C, it means the loosely bond or free water molecules from product zeolite cage. Then the reducing weight also occurred at 270-400°C wich is due to the decomposition of the CTAB surfactant resides on the product zeolite surface.

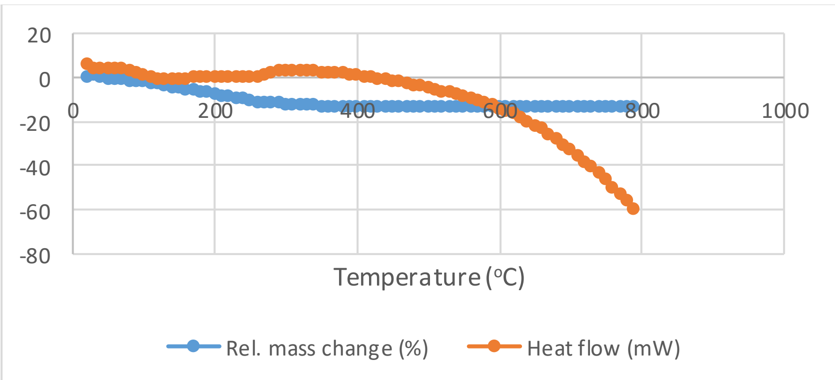

Figure 5. Thermogram of the synthesized product ratio Si/Al (v/v) 1

Should the curves be color? See the published article in Vol 17 to format curve (without grill).

Conclusion

The presence of CTAB surfactant as structure directing at concentration 10 times CMC on synthesis zeolite from sugarcane bagasse ash improves the porosity and crystallinity of the product zeolite, but in this reseach the product still has micropore structure. Too short conclusion

Acknowledgments Should be mentioned which specific grant and contract number.

This research is financially supported by DRPM DIKTI

REFFERENCES **Apply new style as used in IJC articles vol. 17. Cite literatures that can be assessed on line. Avoid to cite unpublished documents such as skripsi ect.**

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LAMPIRAN 6

Bagian-bagian yang diperbaiki/direvisi atas comment reviewer (Round 1)

IMPROVEMENT OF ARTICLE

Title: The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

REVIEWER	COMMENT	DESCRIPTION OF IMPROVEMENT
Reviewer A	The results are great and worth to be published in this journal	
	There are some issues need to be addressed: 1. The novelty of this research has not been described in detail, what advantage this study from previous similar studies, is there any important breakthroughs resulting from this research?	Description of the virtues and advantage of this study has been added at the last paragraph on page 3. Based on previously studies information, there has never been conducted researches using CTAB surfactants as directing agent in synthesis of zeolite from sugarcane bagasse ash. Therefore, in this study, we report the influence of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis zeolite using 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 the product. As a comparison, zeolite from sugarcane bagasse ash in the absence of structure directing agent was also synthesized.
	Is there any important breakthroughs resulting from this research?	Yes 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 waste bagasse into material into high-value material, because this 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.
	2. Several additional data are necessarily required to support author's arguments within the manuscript (e.g. XRD and BET).	XRD datas has been presented in Figure 3, but not all synthesis products are analyzed by XRD. Only products synthesized without CTAB and with CTAB concentrations of 0.01 M. This concentration is a highest concentration, hat is 10 times to CMC so it is expected to provide information quite different from before. The explanation about the data is given at pages 9-10. For BET data was added in page 11. The description is given at pages 10-11.

Reviewer B	Novelty aspect of this research is not explicitly described in this article. In addition, the data presented in this article is also relatively less significant to support the conclusion.	The novelty aspect was described more explicit. In this article has been added several data such as SEM for other samples, TGA and BET to support in discussion and conclusion.
	Characterization data needs to be enriched by other characterization data such as DTA and BET data.	Several analyzes (TGA and BET) to characterize the product have been added to support the discussion and conclusions.

LAMPIRAN 7

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Sriatun Sriatun <sriatun@live.undip.ac.id>
to Nuryono

To. Mr. Nuryono
Editor in Chief
Indonesian Journal of Chemistry

Thank you Mr. Nuryono, for the opportunity given to me to
synthesis of zeolite from sugarcane bagasse ash".
At the last time that given, I re-submitted the article that I h
in Indonesian Journal of Chemistry (IJC). Once again than

Best regards

Sriatun

Laboratory of Inorganic Chemistry,
Dept of Chemistry FSM Undip

--
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2 Attachments

LAMPIRAN 8

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Nuryono Nuryono <nuryono_mipa@ugm.ac.id>
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Dear Mrs sriatun sriatun:

We have reached a decision regarding your submission to Indonesian J of Chemistry, "The Effect of cetyltrimethylammonium bromide surfactant synthesis of zeolite from sugarcane bagasse ash".

Our decision is: Revisions Required
The comments of reviewers can be read below or/and in attachment

The Revised article has to be submitted within one week after receiving email.

Best regards,

Nuryono Nuryono
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The Revised article has to be submitted within one week after receiving email.

Best regards,

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Attachment comment reviewer

Comment reviewer pada artikel

The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

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ABSTRACT

The effect of surfactant in synthesis zeolite from sugarcane bagasse ash was studied. 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 hours and calcination at 500˚C for 5 hours. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5.10^{-4} M, 10^{-3} M and10^{-2} M. The result showed all of product have strong absorbance at 950-1050 cm^{-1} and 620-690 cm^{-1}, 420-460 cm^{-1}, double ring at 520-570 cm^{-1}, pore opening at 300-370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400-3450 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 was known that the crystal shape of product on ratio Si/Al 1 and 25 with concentration of cetyltrimethylammonium 10^{-2} M was homogenous and orderly.

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

ABSTRAK

Telah dilakukan kajian pengaruh surfaktan setiltrimetilamonium pada sintesis zeolite dari ampas tebu. Dalam penelitian ini surfaktan setiltrimetilamonium 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, 10^{-3} M dan 10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950-1050 cm^{-1}, 620-690 cm^{-1}, 420-460 cm^{-1}. *Vibrasi double ring pada 520-570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400-3450 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 mempunyai bentuk yang lebih homogen dan teratur pada rasio silikat/Aluminat 1dan 25.*

Kata kunci: *surfaktan, setiltrimetilamonium bromida, sintesis, zeolit, ampas teb*

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 producted 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 bagasse converted to ashes, the content of silica (SiO_2) to be 64.65% [3]. According [4] the SiO_2 content in sugarcane bagasse ash from Tasik Madu sugar mills Industry slightly higher, that was 70.97%. 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 applicated 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 to synthesis of various zeolite. Synthesis of NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8, 9, 10], from illite-smectite [11], from aluminum solid

waste [12]. NaX and NaA [13] NaY was synthesized from rice husk silica [14] and bentonite [15]. Synthesis of zeolite as micro-mesoporous materials has been developed continously. As micro-mesoporous materials zeolite was expected possess ordered mesostructure, 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 molecul as structure directing agent must have high chemical stability in order to resist in the hydrothemal 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 zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier and branch molecule [17]. Directing agent also used to control the growth of crystal [18].

Based on previously studies information, there has never been conducted researches using CTAB surfactants as directing agent in synthesis of zeolite from sugarcane bagasse ash. Therefore, in this study, we report the influence of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis zeolite using 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 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 waste bagasse into material

into high-value material, because this 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 of Java. First step, Sugarcane bagasse was burned then ashed in furnace at 700^oC during 4 hours. Futhermore, 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 hours and filtered. The supernatant was sodium silicate solution. Determination the content of SiO_2, Al_2O_3 dan others oxide by AAS. NaOH pellets, $Al(OH)_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The equipment were used in this research: stirrer, bar magnetic stirrer, oven, furnace, grinder were used on synthesis process. FTIR spectrometer Nicolet Avatar 360 IR, X-Ray Diffractometer (XRD) Rigaku Multiplex, Scanning Electron Microscope (SEM) FEI Inspect S50, Energy-Dispersive X-ray (EDX) spectroscopy EDAX AMETEX,

Procedure

An early step was preparation of sodium aluminate solution from dillution of 20 g sodium hydroxide in 100 mL aquadest. The solution was boiled, then 8,5 g alumunium hydroxide was added into solution until homogenous solution achieved. The next step is preparation 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 spesific duration until gel formed, the process was continued by hydrothermal treatment in teflon container at 100°C during 7 days, then aging process at rom temperature for 24 hours. The final steps, the precipitates were filtering, washing , drying and calcinating at 500°C for 5 hours. The product characterized by FTIR and XRD. The provision code in this research was given in table 1.

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)

See in article vol .17 how tables are formatted

RESULTS AND DISCUSSION

The aim of the ashing of sugarcane bagasse at 700°C is removing the organic compounds and changing the inorganic compounds to metal oxide such as SiO_2, Al_2O_3, MgO, CaO, Fe_2O_3 and others. The content of silica and alumina the sugarcane bagasse ash is Si 70.82% and Al 1.04% respectively.

The purposes of temperature adjusting at 100°C for 168 h on hydrothermal process controlled the rate of nucleation and crystallization. The effect of crystallization times (1, 2,3,4,6 and 8 hours) on formation of zeolite A under 373 K has been known that the long time of hydrothermal process influenced the crystallinity degree [19]. Actually, in this process the silica alumina framework was formed. The crystallinity of zeolite increased with the increasing of hydrothermal time. Meanwhile, ageing time during 24 hours after hydrothermal process for arranged and established the framework structure of zeolite.

Several factors that influence in the synthesis process of zeolite are the type of precursor, reactant, volume of surfactant, temperature of reaction, aging time and organic template. Generally, the framework of zeolite had a negative charge, so it was neutralized by some cation. In this case, the surfactant such as cetyltrimethylammonium was used for structure directing of material, so the product would have different size and shape of pore and more homogenous. Cetyltrimethylamonium as cationic surfactant would be interacted electrostatic with the negative charge in silicate aluminate framework from zeolite.

Cetyltrimethylammonium, TPAOH and PPDA surfactant were used as structure directing agents in synthesis zeolite [20]. The cetyltrimethylammonium (CTA) is cationic surfactant, the presence of cationic surfactant in solution would be reacted rapidly with silicate/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ ions primary building units. Furthermore, they occurred condensation polymerization and formed zeolite embryo. Surfactant as structure directing determined the type of polyhedral as which built the structure of zeolite completely. The illustration of mechanism given in Figure 1.

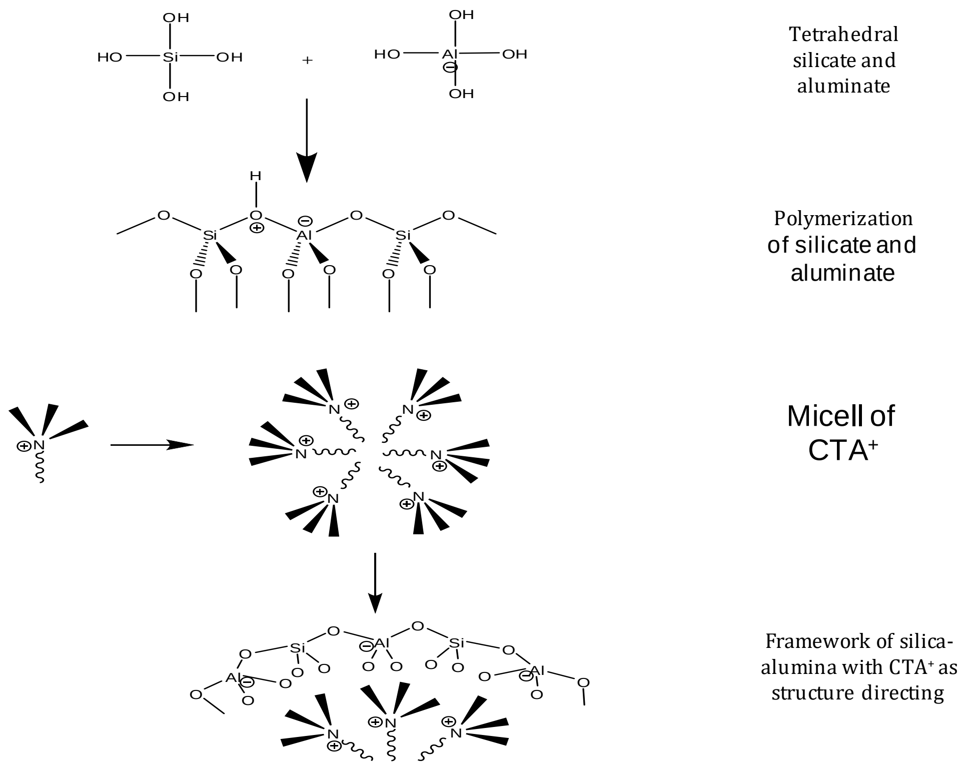

Figure 1. The mechanism of zeolite framework formation by CTA^+ as structure directing (the letters in figure are too small, unreadable)

Finally, the CTAB surfactant as structure directing was disappeared from the zeolite structure by calcination at 500°C during 4 hours. The surfactant would be decomposed to be gas, and leaved hole. This hole was pore of materials. Above explanation is discussion, not result

Characteristics~~zations~~ of products

It was investigated synthesis 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 is needed to find out of the influence. The FTIR spectra of product synthesis given in Figure 2a, 2b and 2c.

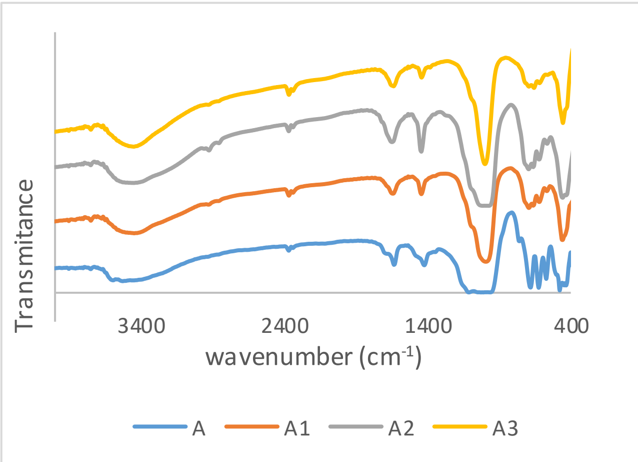

Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4}M (A2) with CTAB 1.10^{-3} M (A3) with CTAB 1.10^{-2} M
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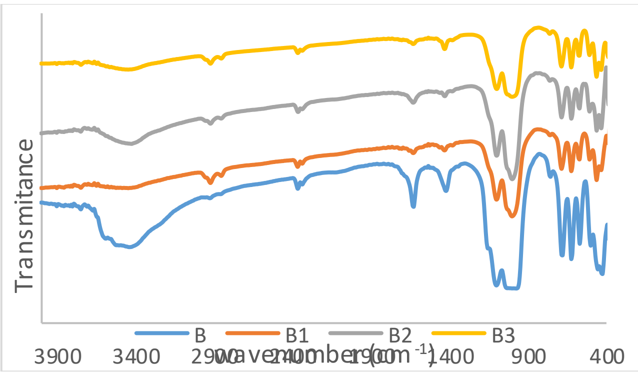

Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4}M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M

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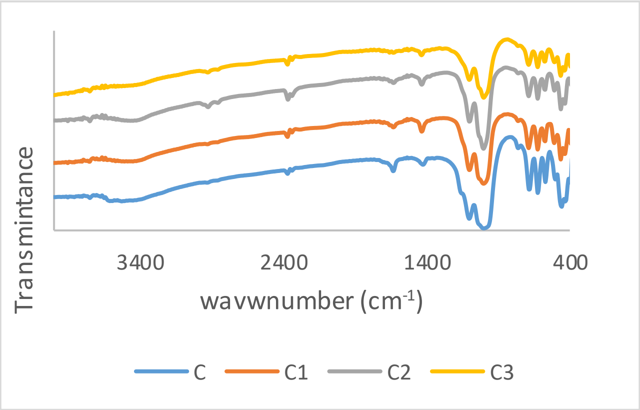

Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5.10^{-4}M (C2) with CTAB 1.10^{-3} M (C3) with CTAB 1.10^{-2} M Should the curves be color? The lines and scale are not clear/contract. a.u. should be used for transparence unit.

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 the internal linkage, there is asymmetric stretching vibration of TO_4 (T = Si or Al) and appears at 950-1250 cm^{-1} wavenumber, whereas the symetric stretching vibration at 650-720 cm^{-1} and 500 cm^{-1}. Furthermore, the wavenumber at 1050-1150 cm^{-1} was asymmetric stretching vibration, 750-820 cm^{-1} is symetric stretching vibration in the external linkage. All 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 300-420 cm^{-1} is pore opening and 500-650 cm^{-1} is doublé ring [21]. Doublé ring is specific character of zeolite type, because it is an external lingkage between polyhedral. From the FTIR datas, the zeolite was formed as product synthesis. The presence of Cetyltrimethylammonium surfactants has significantly affected the intensity and patterns of absorption at the wavenumber 950-1250 cm^{-1} and 3400-3500 cm^{-1}. The lower absorption intensity of wave number at 3400-3500 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 zeolit. Beside 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 950-1250 cm^{-1} after the addition of CTAB.

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

Figure 3 shows the pattern of diffractogram XRD of product synthesis. Inorganic material has different shape and structure crystal, due to the diffractogram pattern was characteristic.

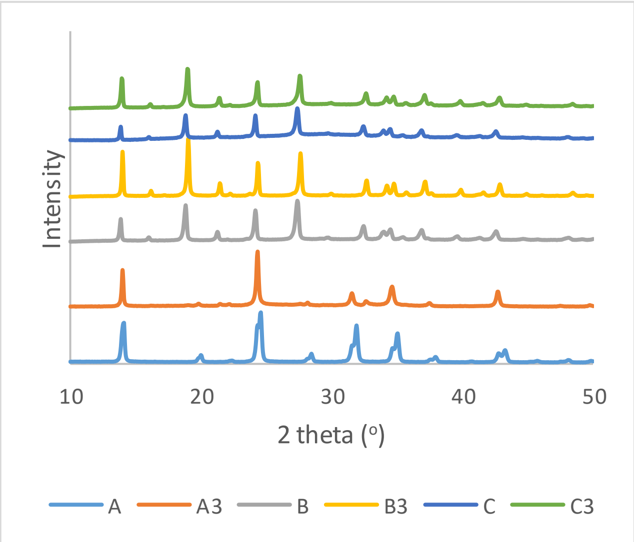

Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2}M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2}M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB1.10^{-2}M

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The instensity of diffractogram indicated the crystal quality of material. It could investigated the kind of product synthesis by CTAB different with product synthesis without CTAB. In addition, the composition of precursor also affected the kind of product. The product synthesis by ratio of Si/Al (v/v) 1 without CTAB was sodalite. 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 product synthesis 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 Figure 3. All of the peaks on NaP1 diffractogram NaP1 as product synthesis 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.

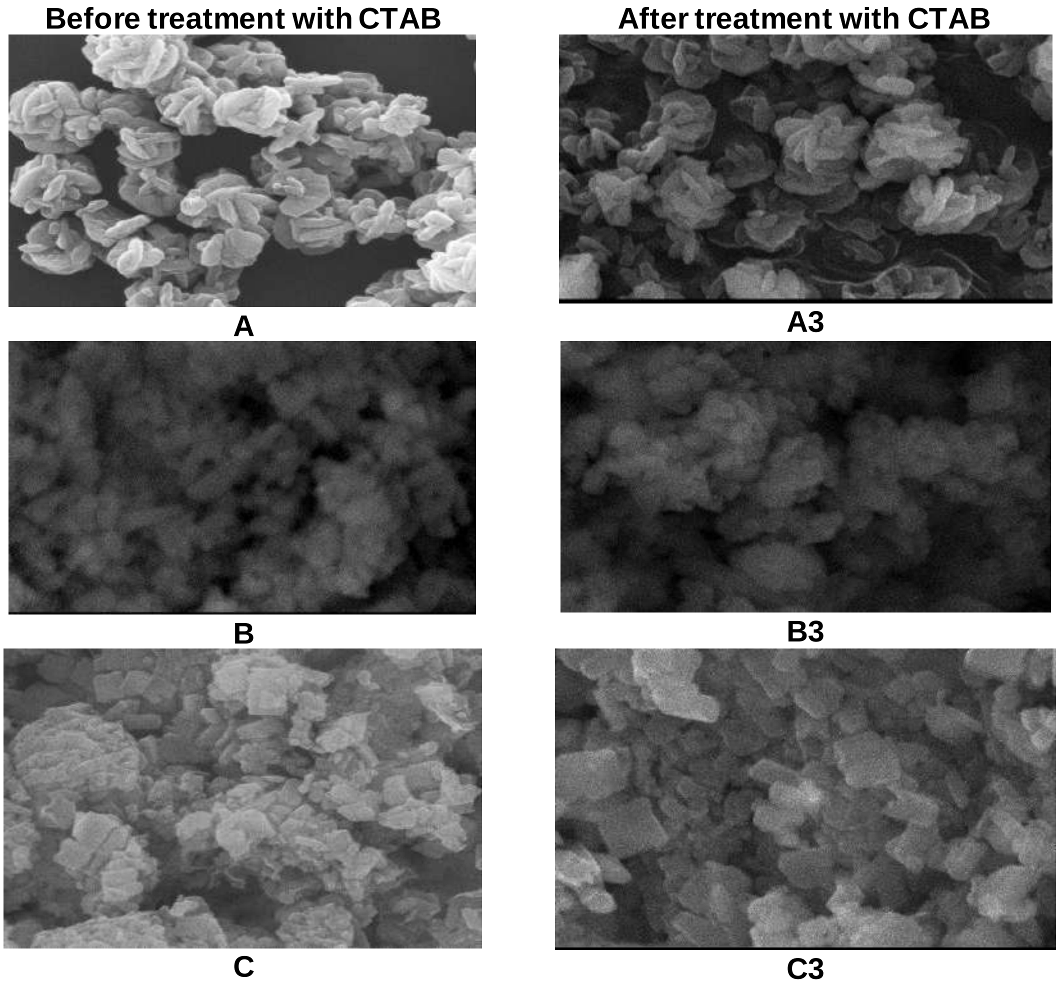

Figure 4. The SEM image of synthesized zeolite

Figure 4 is SEM photograph corresponding to the product synthesis. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears 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 diffrerent 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 datas 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. All of the product showed that ratio of Si/Al almost 1, these is consistent with the BET result that this product is micropore material. The use of surfactant CTAB as a pore directing agent increases the number of ions silicate and aluminate that interacted to form the product.

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		

See the published article in Vol 17 to format Table

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 Figure 4. 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 13.508 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 5.28x10^{-3} cm^3/g and average pore radius was determined as 2.261°A. It means the NaP1 of this research have a micropore structure.

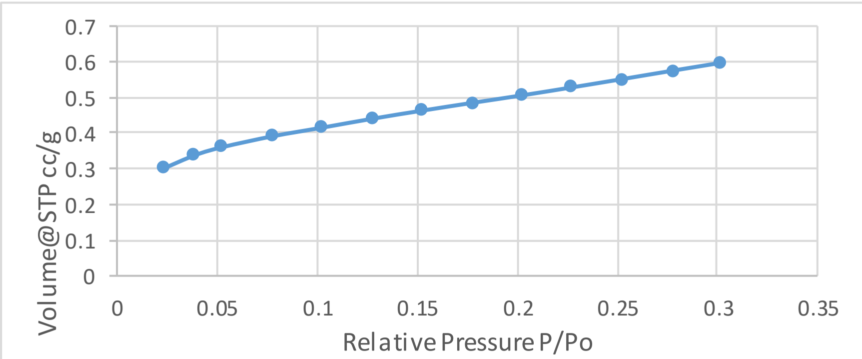

Figure 4. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

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The TGA (thermogravimetric analysis) graph for the synthesized zeolite from sugarcane bagasse ash is given in Figure 5. From the figure, it can be seen that the sample is lost the weight at temperature 100°C, it means the loosely bond or free water molecules from product zeolite cage. Then the reducing weight also occurred at 270-400°C wich is due to the decomposition of the CTAB surfactant resides on the product zeolite surface.

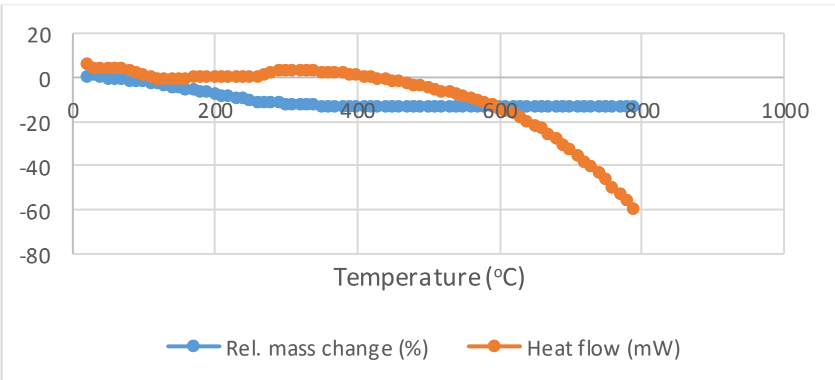

Figure 5. Thermogram of the synthesized product ratio Si/Al (v/v) 1

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Conclusion

The presence of CTAB surfactant as structure directing at concentration 10 times CMC on synthesis zeolite from sugarcane bagasse ash improves the porosity and crystallinity of the product zeolite, but in this reseach the product still has micropore structure. Too short conclusion

Acknowledgments **Should be mentioned which specific grant and contract number.**

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REFFERENCES **Apply new style as used in IJC articles vol. 17. Cite literatures that can be assessed on line. Avoid to cite unpublished documents such as skripsi ect.**

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LAMPIRAN 9

Daftar perbaikan/revisi per bagian

IMPROVEMENT OF ARTICLE

Title: The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

REVIEWER	COMMENT	DESCRIPTION OF IMPROVEMENT
	Marked on words/part	
Reviewer	**Part: abstract** homogenous and orderly	The words have been corrected : more homogeneous and orderly
	Part: instrumentation The equipment were used in this research: stirrer, bar magnetic stirrer, oven, furnace, grinder were used on synthesis process. FTIR spectrometer Nicolet Avatar 360 IR, X-Ray Diffractometer (XRD) Rigaku Multiplex, Scanning Electron Microscope (SEM) FEI Inspect S50, Energy-Dispersive X-ray (EDX) spectroscopy EDAX AMETEX,	Description of instrumentation has been corrected. The sample functional group is determined by FTIR spectrometer Nicolet Avatar 360 IR. The diffraction patterns of the samples were measured by X-ray diffractometer (XRD) Rigaku Multiplex with Cu Kα radiaton (λ = 1.54184 A°) 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 N2 adsorption isotherms at liquid nitrogen temperature (-195,7 °C) using a Quantachrome NovaWin, Quantachrome Instrument version 10.01. The thermal gravimetric analysis (TGA) analysis of samples were determined using LINSEIS STA PlatinumSeries, Platinum evalustion V1.0.138.
	Part: Procedure The next step is	The words have been corrected : The next steps were Description: Because at that stage consists of several steps that must be plural, then followed “were” because it happened (past)

Part: Procedure

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)

See in article vol .17 how tables are formatted

The Table 1 has been corrected according to the format of the article Vol. 17 of IJC

Table 1. The composition of precursor and surfactant

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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)

Part: Result and discussion

has been

The word has been omitted and replaced with : was

Figure 1. The mechanism of zeolite framework formation by CTA^+ as structure directing (the letters in figure are too small, unreadable)

The letters in Figure 1 has been enlarged

Finally, the CTAB surfactant as structure directing was disappeared from the zeolite structure by calcination at $500^{o}C$

This section has been described according to the study results obtained

	during 4 hours. The surfactant would be decomposed to be gas, and leaved hole. This hole was pore of materials. Above explanation is discussion, not result	
	Characteristics~~zations~~ of products	The word has been corrected: Characteristics of products
	Figure 2a., Figure 2b.Figure 2c.and Figure 3 Should the curves be color? The lines and scale are not clear/contract	The curves do not should be colored. So, the curves have been corrected become colorless and to distinguish each line has the corresponding encoded
	Table 2. See the published article in Vol 17 to format Table	The Table 1 has been corrected according to the format of the article Vol. 17 of IJC
	Figure 4 and Figure 5 Should the curves be color? See the published article in Vol 17 to format curve (without grill).	The curves do not should be colored. So, the curves have been corrected become colorless and gridlines have been omitted
	Part: Conclusion Too short conclusion	The conclusion has been added with some information from discussion
	Section: Acknowledgments Should be mentioned which specific grant and contract number.	Acknowledgments have been corrected. - Specific grant and contract number have been mentioned in this acknowledgments
	Section: REFFERENCES Apply new style as used in IJC articles vol. 17. Cite literatures that can be assessed on line. Avoid to cite unpublished documents such as skripsi ect.	The literary citation has been corrected - Citation used the style in the article IJC vol. 17 [1] Srinivasan, R. and Sathiya, K., (2010), Experimental Study on Bagasse Ash in Concrete, *International Journal for Service Learning in Engineering,* 5(2), 60-

		66. - Unpublished documents have been omitted and replaced by journals. [4] Aida, N. and Dewanti, L., (2010), *Pembuatan Silika Gel Dari Abu Ampas Tebu Dengan Proses Ekstraksi Basa (NaOH) dan Sol Gel*, D3 Teknik Kimia Institut Teknologi Surabaya, Surabaya. [18] Primaroni, O., Sriatun and Nuryanto, R., (2013), Sintesis Zeolit dari Fly Ash PLTU Ombilin: Pengaruh Variasi Konsentrasi Surfaktan Cetyltrimethylammonium Bromide (CTAB) Terhadap Kristalinitas Zeolit, *Prosiding Seminar Nasional Kimia dan Pendidikan Kimia 2013* [20] Yusri, S., (2012), *Synthesis and Characterization of Mesoporous ZSM-5 Zeolite Using Secondary template and Preliminary Study of Methane Oxidation Catalysis,* Skripsi, Kimia FMIPA UI. Substituted by: [4] Norsurayaa, S., Fazlenaa, H., Norhasyimi R., 2016, Sugarcane Bagasse as a Renewable Source of Silica to Synthesize Santa Barbara Amorphous-15 (SBA-15), *Procedia Engineering*, 148, 839 – 846 [18] Hasan, F., Singh, R., Li, G., Zhao, D., Webley, P.A., 2012, Direct synthesis of hierarchical LTA zeolite *via* a low crystallization and growth rate technique in presence of cetyltrimethylammonium bromide, *Journal of Colloid and Interface Science*, 382,1–12 [20] Wang, L., Yang, W., Xin, C., Ling, F., Sun, W., Fang, X., Yang, R., 2012, Synthesis of nano-zeolite IM-5 by

		hydrothermal method with the aid of PEG and CTAB, *Materials Letters*, 69,16–19

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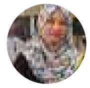

Sriatun Sriatun <sriatun@live.undip.ac.id>
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To. Mr. Nuryono
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Indonesian Journal of Chemistry

Thank you Mr. Nuryono, for the second opportunity improv
sugarcane bagasse ash".
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published in Indonesian Journal of Chemistry (IJC). Once a

Best regards

Sriatun

Laboratory of Inorganic Chemistry,
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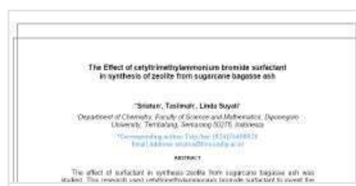

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Thank you for your contribution to Indonesian Journal of Chemistry.

Best regards,

Nuryono Nuryono
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The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

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REVIEW COPYEDITING

The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash

***Sriatun[1], Taslimah[1], Linda Suyati[1]**

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ABSTRACT

The effect of surfactant in synthesis zeolite from sugarcane bagasse ash was studied. 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 hours and calcination at 500˚C for 5 hours. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5.10^{-4} M, 10^{-3} M and10^{-2} M. The result showed all of product have strong absorbance at 950-1050 cm^{-1} and 620-690 cm^{-1}, 420-460 cm^{-1}, double ring at 520-570 cm^{-1}, pore opening at 300-370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400-3450 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 was known that the crystal shape of product on ratio Si/Al 1 and 25 with concentration of cetyltrimethylammonium 10^{-2} M was more homogeneous and orderly.

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

ABSTRAK

Telah dilakukan kajian pengaruh surfaktan setiltrimetilamonium pada sintesis zeolite dari ampas tebu. Dalam penelitian ini surfaktan setiltrimetilamonium 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, 10^{-3} M dan 10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950-1050 cm^{-1}, 620-690 cm^{-1}, 420-460 cm^{-1}. *Vibrasi double ring pada 520-570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400-3450 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 mempunyai bentuk yang lebih homogen dan teratur pada rasio silikat/Aluminat 1dan 25.*

Kata kunci: *surfaktan, setiltrimetilamonium bromida, sintesis, zeolit, ampas teb*

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 producted 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 bagasse converted to ashes, the content of silica (SiO_2) to be 64.65% [3]. According [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 applicated 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 to synthesis of various zeolite. Synthesis of NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8, 9, 10], from illite-smectite [11], from aluminum solid

waste [12]. NaX and NaA [13] NaY was synthesized from rice husk silica [14] and bentonite [15]. Synthesis of zeolite as micro-mesoporous materials has been developed continously. As micro-mesoporous materials zeolite was expected possess ordered mesostructure, 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 molecul as structure directing agent must have high chemical stability in order to resist in the hydrothemal 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 zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier and branch molecule [17]. Directing agent also used to control the growth of crystal [18].

Based on previously studies information, there has never been conducted researches using CTAB surfactants as directing agent in synthesis of zeolite from sugarcane bagasse ash. Therefore, in this study, we report the influence of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis zeolite using 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 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 waste bagasse into material

into high-value material, because this 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 of Java. First step, Sugarcane bagasse was burned then ashed in furnace at 700^{o}C during 4 hours. Futhermore, 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 hours and filtered. The supernatant was sodium silicate solution. Determination the content of SiO_2, Al_2O_3 dan others oxide by AAS. NaOH pellets, $Al(OH)_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentations

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 Kα radiaton (λ = 1.54184 A^{o}) 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 N2 adsorption isotherms at liquid nitrogen temperature (-195,7 °C) using a Quantachrome NovaWin, Quantachrome Instrument version 10.01. The thermal gravimetric analysis (TGA) analysis of samples were determined using LINSEIS STA PlatinumSeries, Platinum evalustion V1.0.138.

Procedure

An early step was preparation of sodium aluminate solution from dillution of 20 g sodium hydroxide in 100 mL aquadest. The solution was boiled, then 8,5 g alumunium hydroxide was added into solution until homogenous solution achieved. The next steps were preparation 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 spesific 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 hours. The final steps, the precipitates were filtering, washing , drying and calcinating at 500°C for 5 hours. The product characterized by FTIR and XRD. The provision code in this research was given in Table 1.

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)

RESULTS AND DISCUSSION

The aim of the ashing of sugarcane bagasse at 700°C is removing the organic compounds and changing the inorganic compounds to metal oxide such as SiO_2, Al_2O_3, MgO, CaO, Fe_2O_3 and others. The content of silica and alumina the sugarcane bagasse ash is Si 70.82% and Al 1.04% respectively.

The purposes of temperature adjusting at 100°C for 168 h on hydrothermal process controlled the rate of nucleation and crystallization. The effect of crystallization times (1, 2,3,4,6 and 8 hours) on formation of zeolite A under 373 K was known that the long time of hydrothermal process influenced the crystallinity degree [19]. Actually, in this process the silica alumina framework was formed. The crystallinity of zeolite increased with the increasing of hydrothermal time. Meanwhile, ageing time during 24 hours after hydrothermal process for arranged and established the framework structure of zeolite.

Several factors that influence in the synthesis process of zeolite are the type of precursor, reactant, volume of surfactant, temperature of reaction, aging time and organic template. Generally, the framework of zeolite had a negative charge, so it was neutralized by some cation. In this case, the surfactant such as cetyltrimethylammonium was used for structure directing of material, so the product would have different size and shape of pore and more homogenous. Cetyltrimethylammonium as cationic surfactant would be interacted electrostatic with the negative charge in silicate aluminate framework from zeolite.

Cetyltrimethylammonium bromide (CTAB) and polyethylene glycol (PEG) surfactant were used as structure directing agents in synthesis zeolite nano-zeolite IM-5 [20]. In this study only used CTAB surfactant with the concentrations less than CMC (Critical Micelle Concentration), equal to CMC and higher than CMC. If the concentrations of surfactant less than CMC the surfactant molecules will be bilayer, but if the concentrations equal or higher than CMC the surfactant molecules will be micelles/sphere. Part of the non-polar group interacts inside the layer if bilayer or the sphere if micelles, while the polar part is the positively charged ammonium part outside. The positively charged of CTAB surfactant in solution would be interacted electrostatically with the with silicate/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ anions primary building units. Furthermore, they occurred condensation polymerization and formed zeolite embryo. Surfactant as structure directing determined the type of polyhedral as which built the structure of zeolite completely. The illustration of mechanism for high concentration of CTAB given in Figure 1.

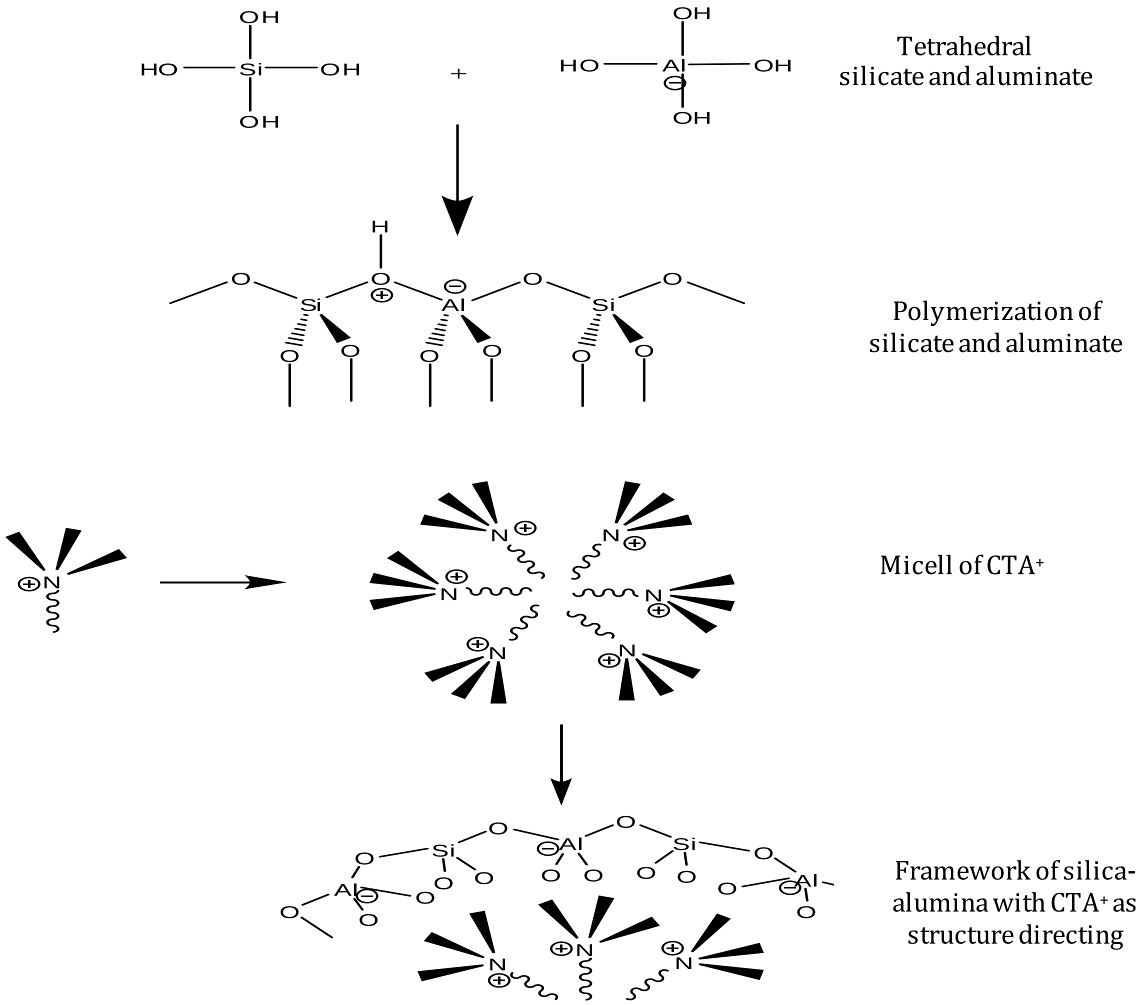


Figure 1. The mechanism of zeolite framework formation by CTA^+ as structure directing

Finally, the CTAB surfactant as structure directing was disappeared from the zeolite structure by calcination at 500°C during 4 hours. The surfactant would be decomposed to be gas, and leaved hole. This hole was pore of materials. A bilayer-shaped of surfactant provided zeolite framework with more Si-OH. Whereas in the micelles-shaped of surfactant produced the zeolite framework Si-O-Si or Si-O-Al was preferred. This can be seen in the FTIR spectra datas on Figure 2a, 2b and 2c. It seems to be discussion, not result. Please revise in order relevant to sub title: result and discussion.

Characteristics of products

It was investigated synthesis 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 is needed to find out of the influence. The FTIR spectra of product synthesis given in Figure 2a, 2b and 2c.

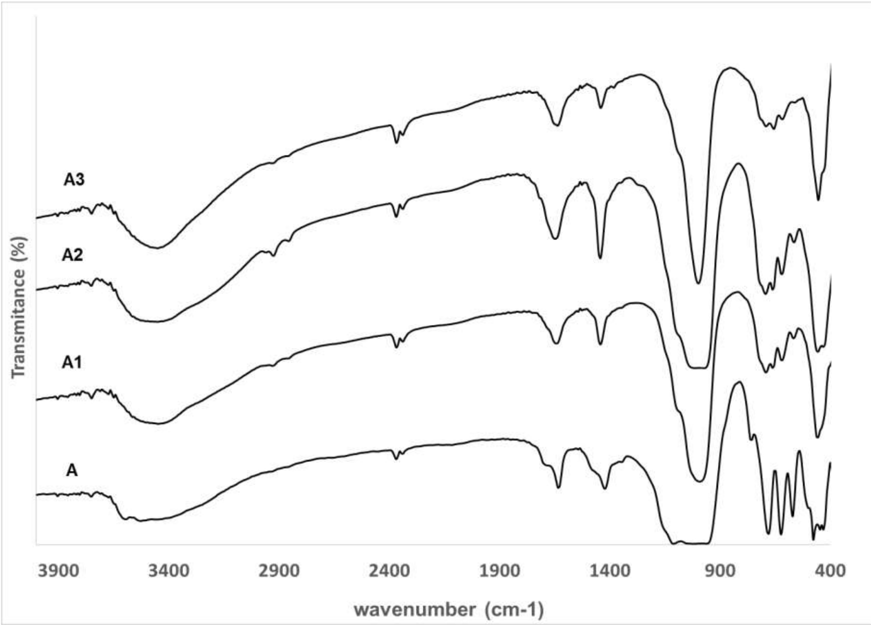


Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4}M (A2) with CTAB 1.10^{-3} M (A3) with CTAB 1.10^{-2} M (scale is too small)

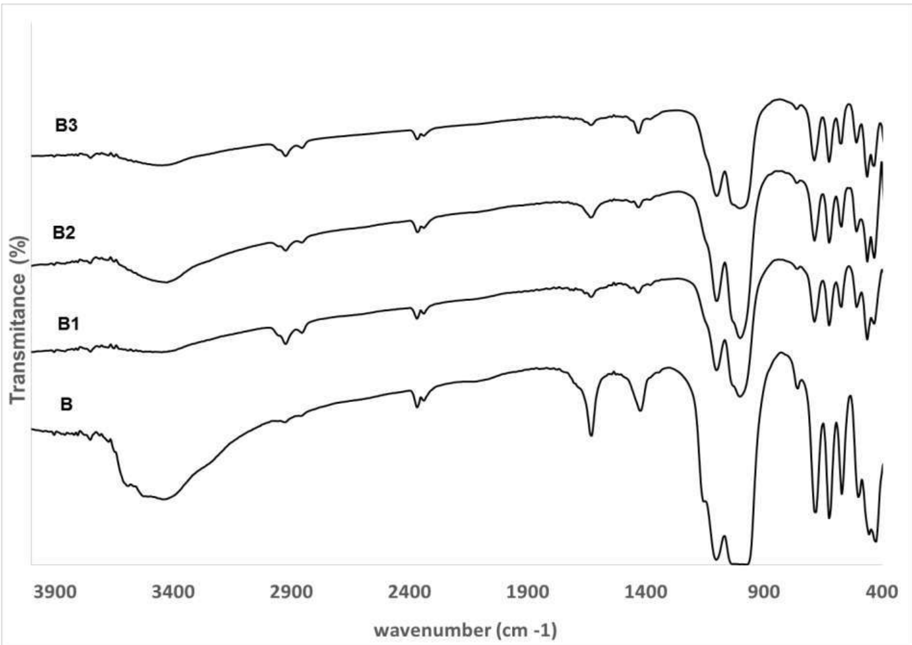


Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4}M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M (scale is too small)

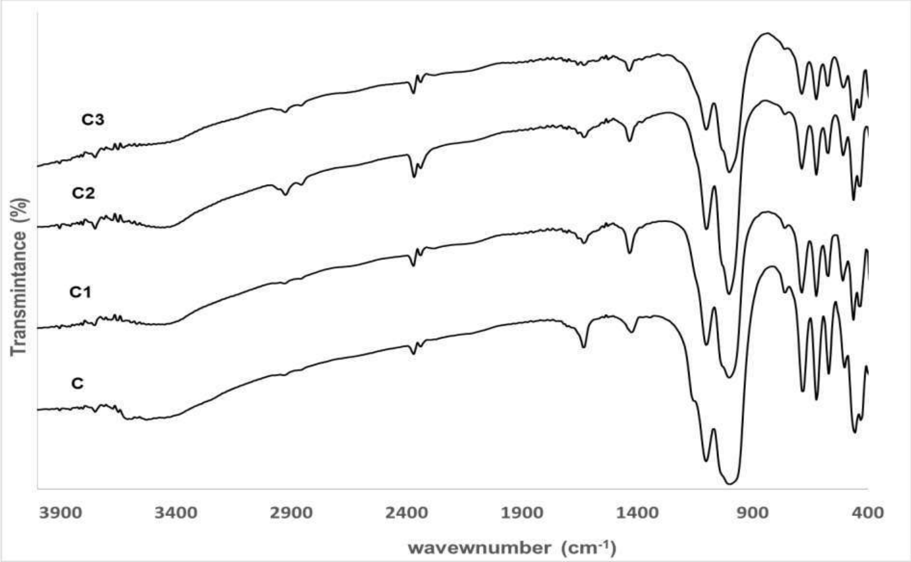


Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5.10^{-4}M (C2) with CTAB 1.10^{-3} M (C3) with CTAB 1.10^{-2} M (scale is too small)

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 the internal linkage, there is asymmetric stretching vibration of TO_4 (T = Si or Al) and appears at 950-1250 cm^{-1} wavenumber, whereas the symetric stretching vibration at 650-720 cm^{-1} and 500 cm^{-1}. Furthermore, the wavenumber at 1050-1150 cm^{-1} was asymmetric stretching vibration, 750-820 cm^{-1} is symetric stretching vibration in the external linkage. All 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 300-420 cm^{-1} is pore opening and 500-650 cm^{-1} is doublé ring [21]. Doublé ring is specific character of zeolite type, because it is an external lingkage between polyhedral. From the FTIR datas, the zeolite was formed as product synthesis. The presence of Cetyltrimethylammonium surfactants has significantly affected the intensity and patterns of absorption at the wavenumber 950-1250 cm^{-1} and 3400-3500 cm^{-1}. The lower absorption intensity of wave number at 3400-3500 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 zeolit.

Beside 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 950-1250 cm^{-1} after the addition of CTAB.

In the FTIR spectra of zeolite modified by CTAB surfactant, no new absorption was observed. The spectra has 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. Thus the shape and pore size of modified zeolite was expected homogenously.

Figure 3 shows the pattern of diffractogram XRD of product synthesis. Inorganic material has different shape and structure crystal, due to the diffractogram pattern was characteristic.

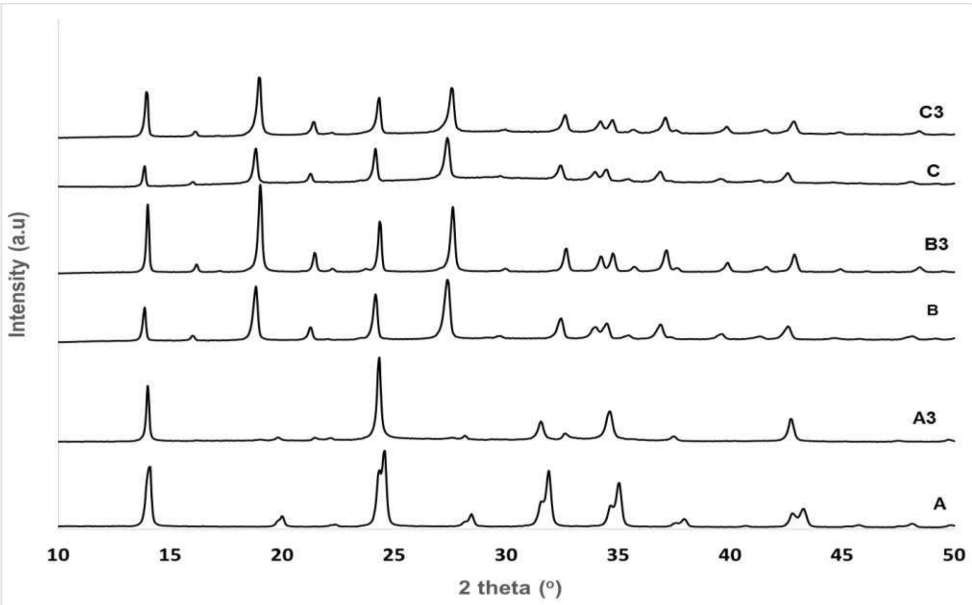


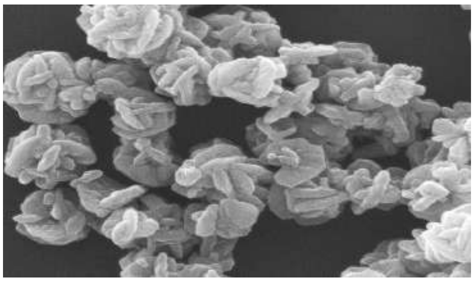
Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2}M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2}M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB1.10^{-2}M

The instensity of diffractogram indicated the crystal quality of material. It could investigated the kind of product synthesis by CTAB different with product synthesis without CTAB. In addition, the composition of precursor also affected the kind of product.

The product synthesis by ratio of Si/Al (v/v) 1 without CTAB was sodalite. 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 product synthesis 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 Figure 3. All of the peaks on NaP1 diffractogram NaP1 as product synthesis 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.

Before treatment with CTAB **After treatment with CTAB**



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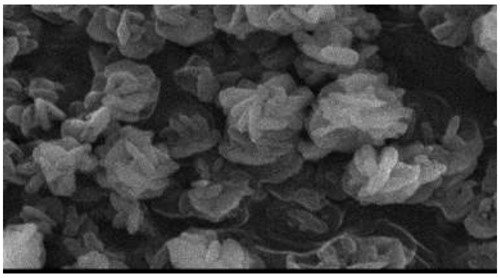

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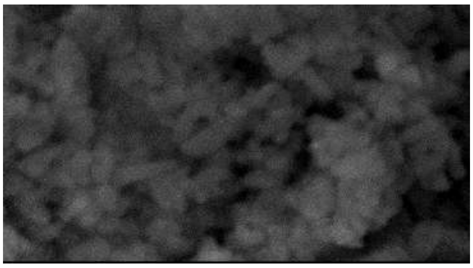

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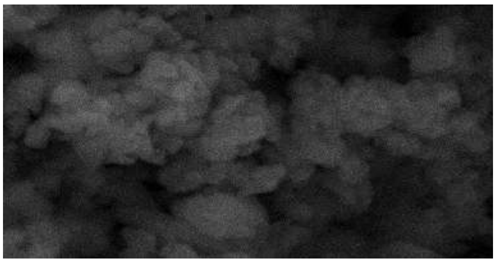

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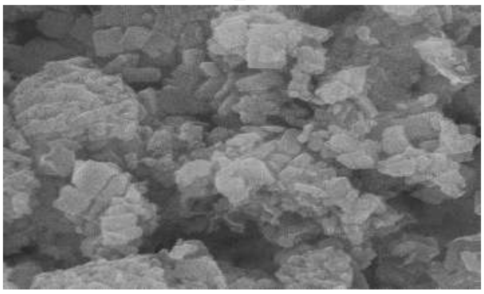

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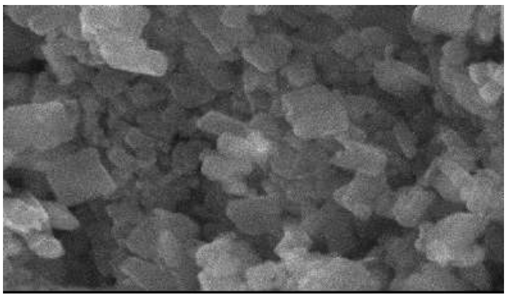

C3

Figure 4. 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 CTAB1.10^{-2}M

Figure 4 is SEM photograph corresponding to the product synthesis. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears 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 diffrerent 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 datas 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. All of the product showed that ratio of Si/Al almost 1, these is consistent with the BET result that this product is micropore material. The use of surfactant CTAB as a pore directing agent increases the number of ions silicate and aluminate that interacted to form the product.

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 CTAB1.10^{-2}M

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 Figure 5. 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 13.508 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 5.28x10^{-3} cm^3/g and average pore radius was determined as 2.261^{o}A. It means the NaP1 of this research have a micropore structure.

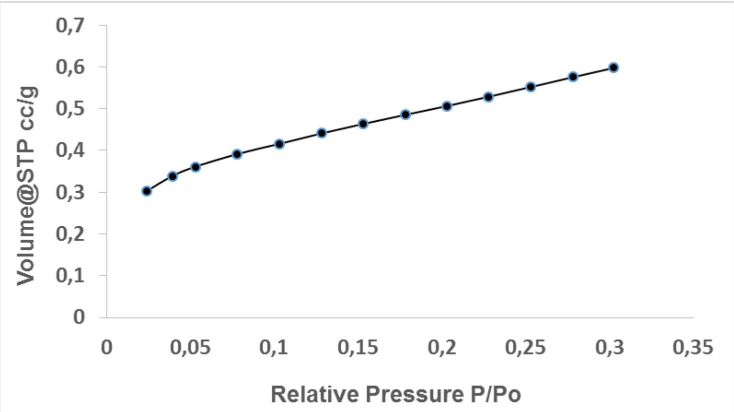

Figure 5. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1 (lines are too tin)

The TGA (thermogravimetric analysis) graph for the synthesized zeolite from sugarcane bagasse ash is given in Figure 6. From the figure, it can be seen that the sample is lost the weight at temperature 100^{o}C, it means the loosely bond or free water molecules from product zeolite cage. Then the reducing weight also occurred at 270-400^{o}C wich is due to the decomposition of the CTAB surfactant resides on the product zeolite surface.

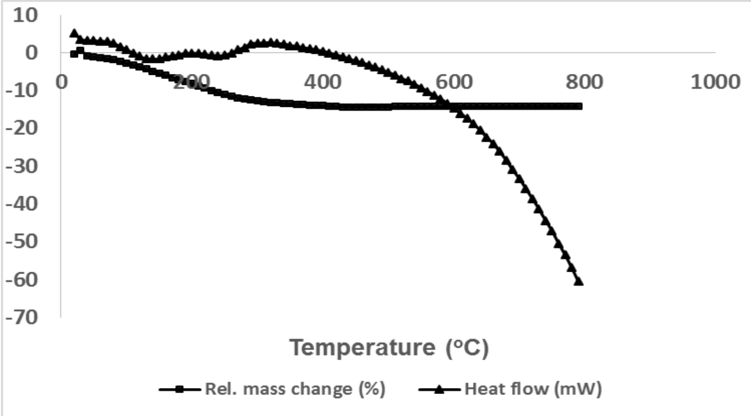

Figure 6. Thermogram of the synthesized product ratio Si/Al (v/v) 1 (there is not legend for x and y axis)

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 SiO_4^{4-} and AlO_4^{5-} 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.

Acknowledgments

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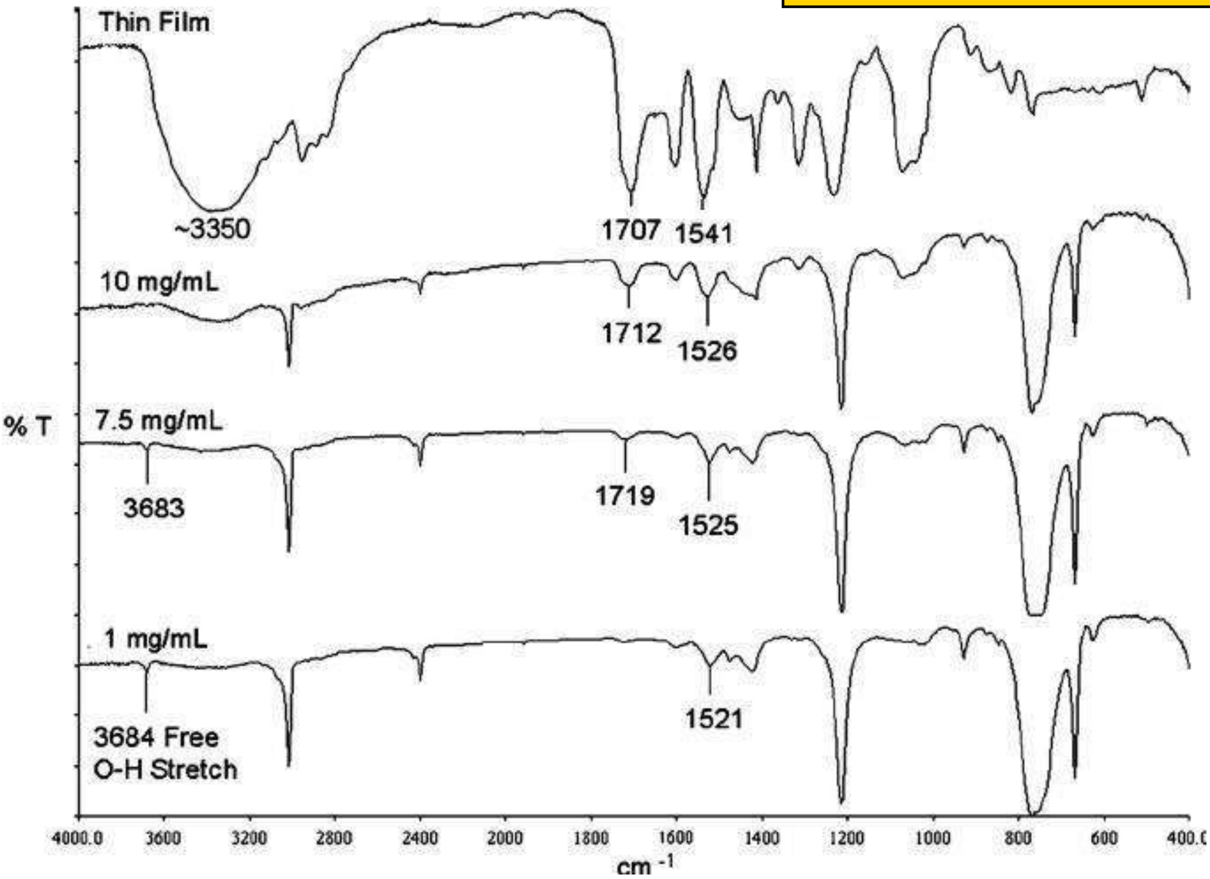

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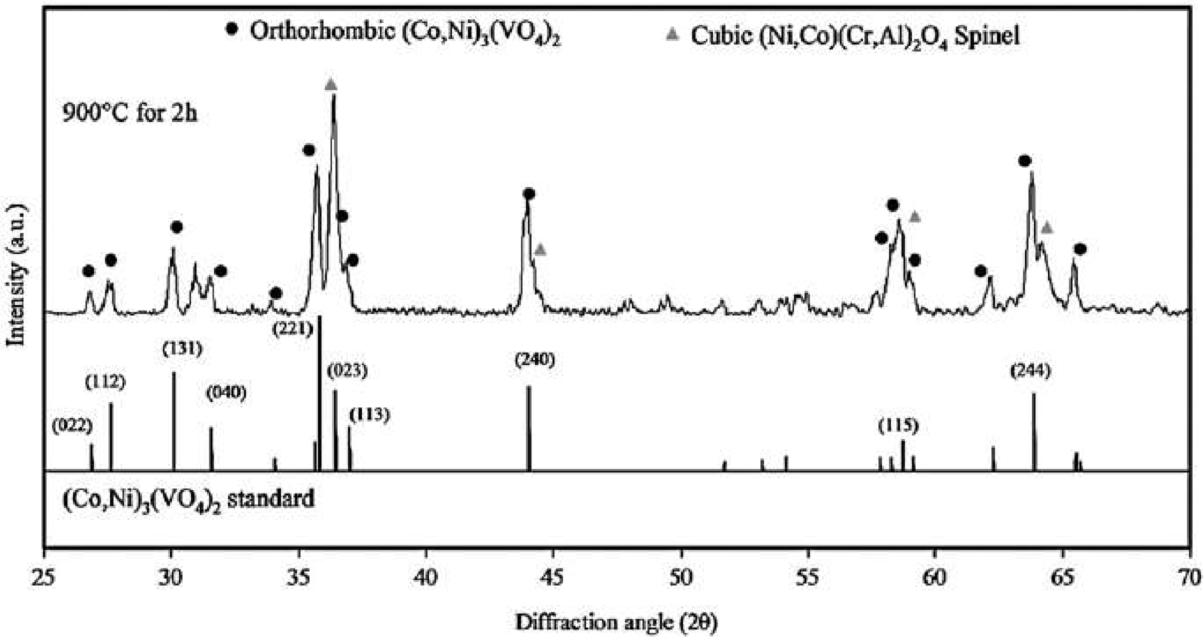

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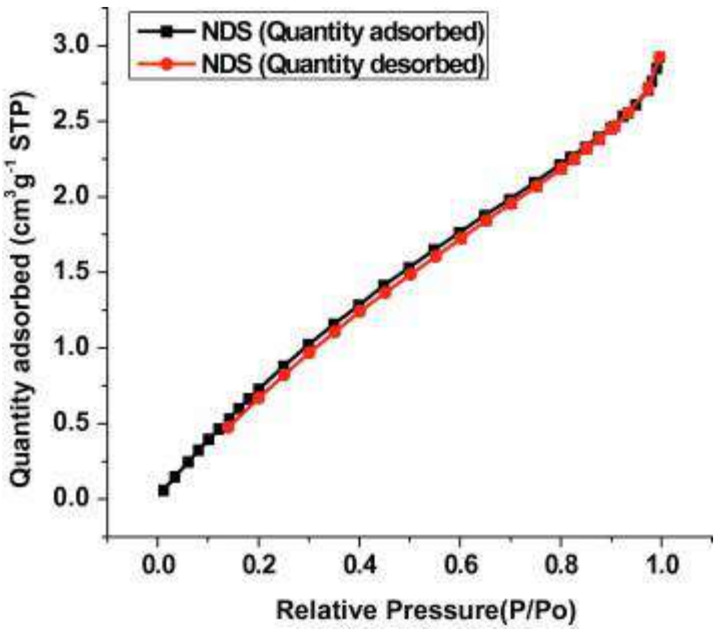

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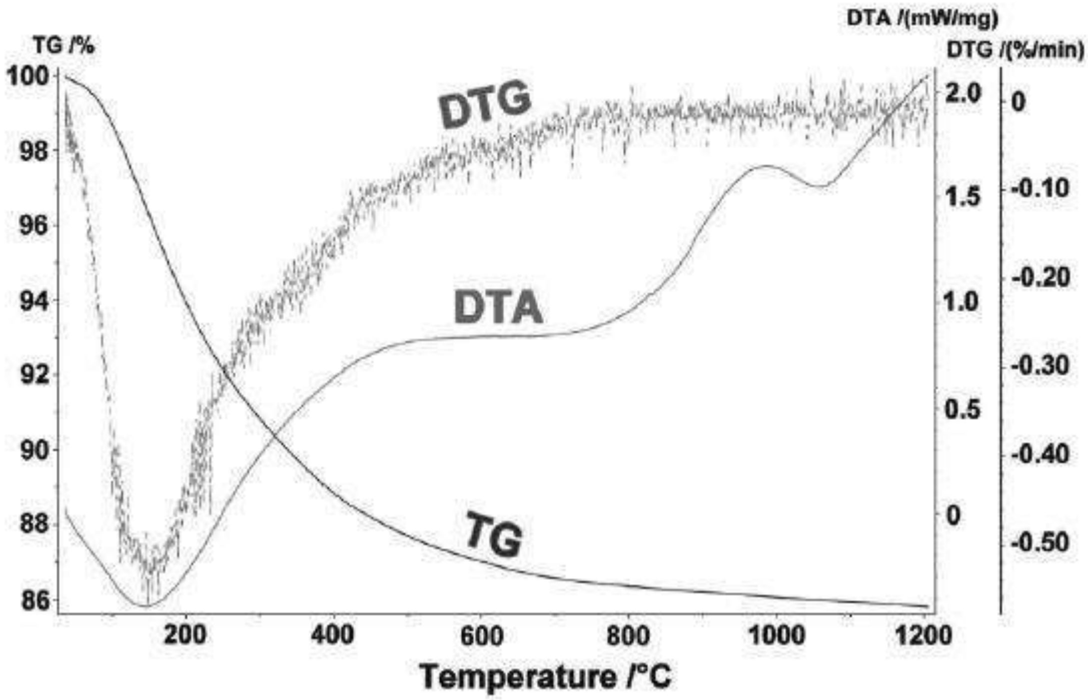

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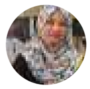

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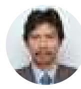

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The Effect of Cetyltrimethylammonium Bromide Surfactant in Synthesis of Zeolite from Sugarcane Bagasse Ash

Sriatun, Taslimah, and Linda Suyati

ABSTRACT

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Judul artikel setelah perubahan

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 hours 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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 were 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 bromide 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 to 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 NaA [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 molecule 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 zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier 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 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 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 waste bagasse 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 of 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 the content of SiO_2, Al_2O_3 and others oxide by AAS. NaOH pellets, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 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 filtering, washing, drying and calcination at 500°C for 5 h. The product 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 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 product synthesis 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 = 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 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 300–420 cm^{-1} is pore opening and 500–650 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 product synthesis for 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	$5x10^{-4}$ (1/2 cmc)
A2	1	1	$1x10^{-3}$ (cmc)
A3	1	1	$1x10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5x10^{-4}$ (1/2 cmc)
B2	15	1	$1x10^{-3}$ (cmc)
B3	15	1	$1x10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5x10^{-4}$ (1/2 cmc)
C2	25	1	$1x10^{-3}$ (cmc)
C3	25	1	$1x10^{-2}$ (10 cmc)

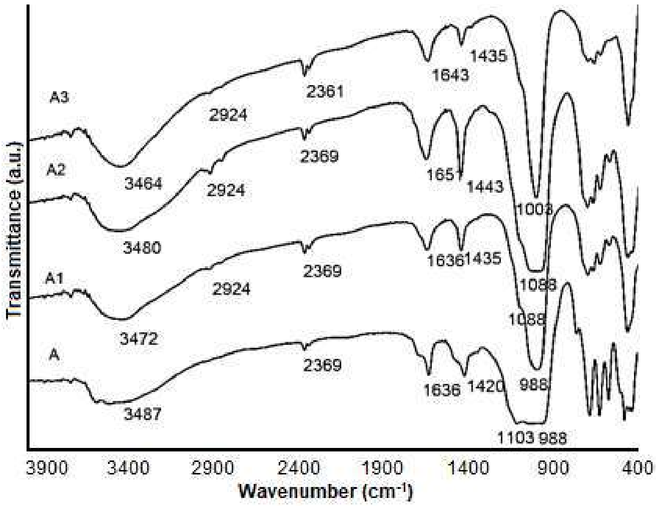

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB $5x10^{-4}$ M (A2) with CTAB $1x10^{-3}$ M (A3) with CTAB $1x10^{-2}$ M

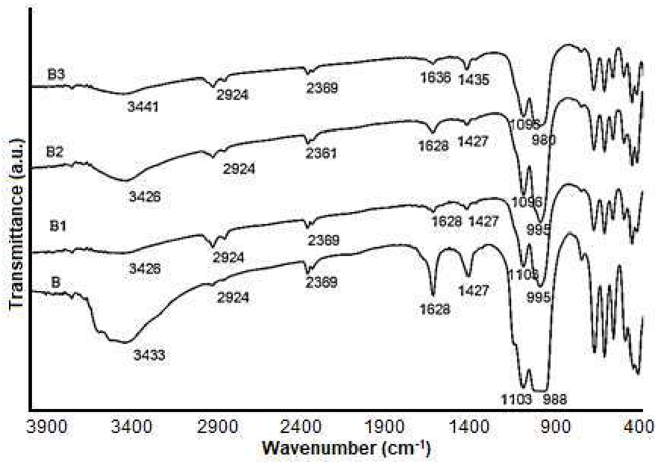

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB $5x10^{-4}$ M (B2) with CTAB $1x10^{-3}$ M (B3) with CTAB $1x10^{-2}$ M

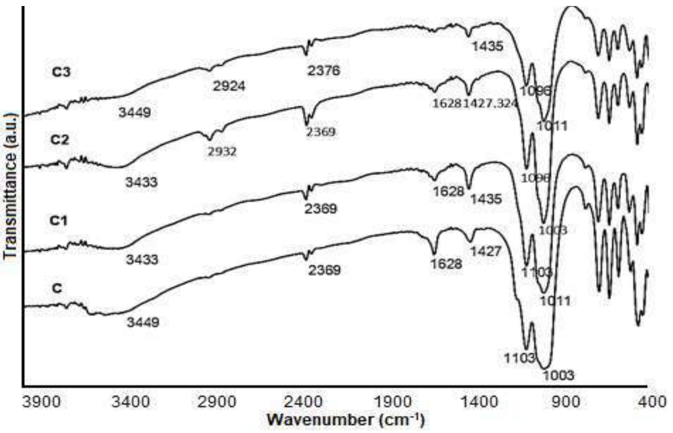

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5x10^{-4}$ M (C2) with CTAB $1x10^{-3}$ M (C3) with CTAB $1x10^{-2}$ M

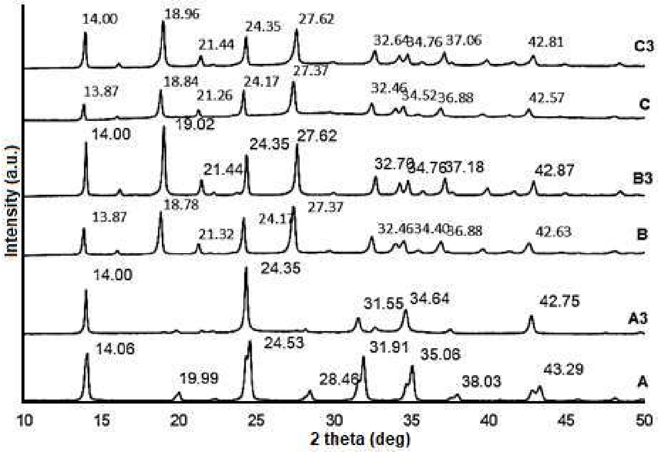

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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. Beside 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

Fig. 4 shows the pattern of diffractogram XRD of product synthesis. 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 product synthesis by CTAB different with product synthesis without CTAB. In addition, the composition of precursor also affected the kind of product. The product synthesis 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 product synthesis 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 product synthesis 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 product synthesis. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears 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

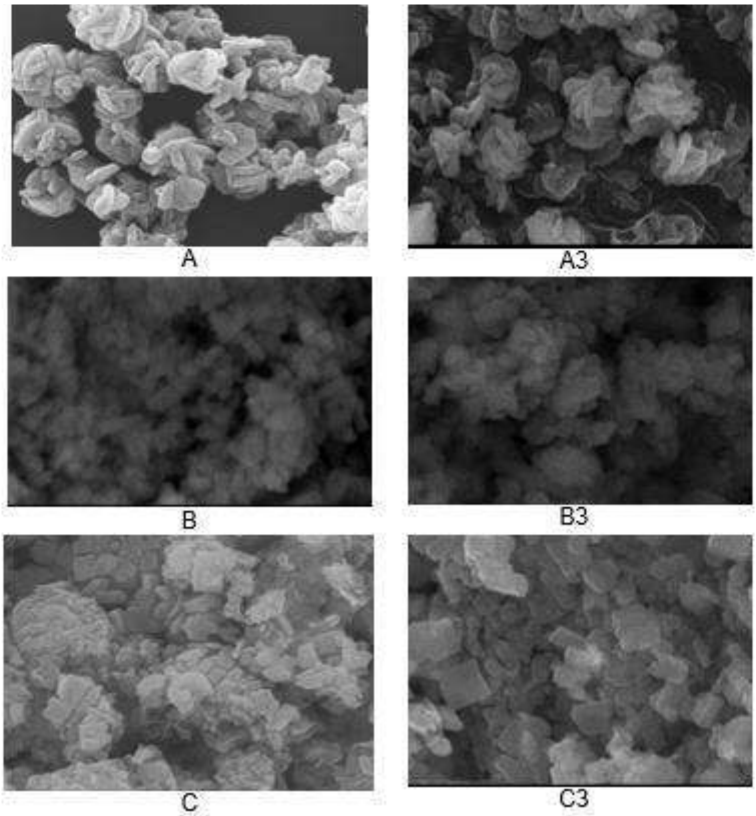

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 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 and CTAB play an important role during the formation of nano crystal IM-5 zeolite.

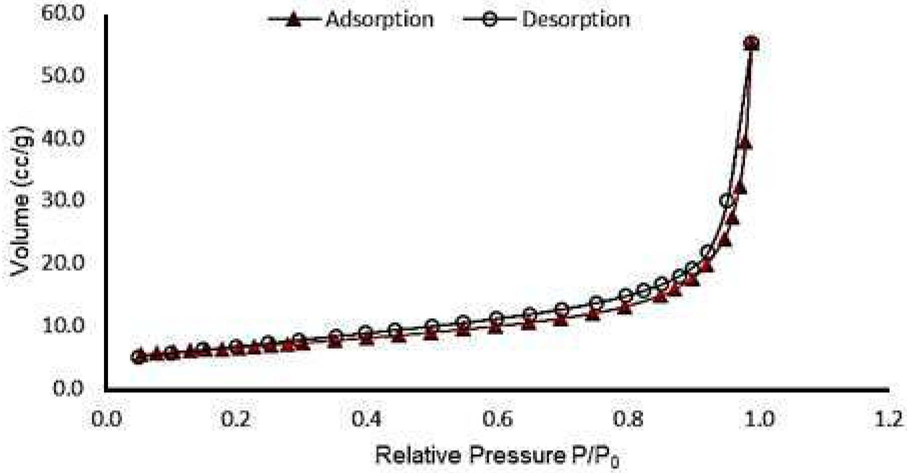

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

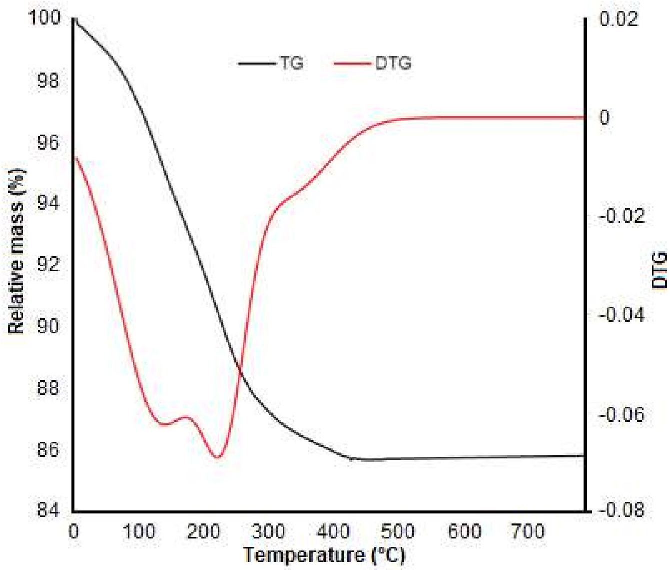

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56x10^{-2} cm^3/g and average pore radius was determined as 7.737°A. It means the NaP1 of this research have 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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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

Sriatun*, Taslimah, and Linda Suyati

Department of Chemistry, Faculty of Science and Mathematics, Diponegoro University, Jl. Prof. Soedarto, SH, Tembalang, Semarang 50275, Indonesia

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 were homogenous and regular in shape.

more ✓ was

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

bromida

ABSTRAK

Tujuan penelitian ini adalah membuat zeolit dari ampas tebu menggunakan setiltrimetilamonium bromida sebagai agen pengarah struktur. Dalam penelitian ini surfaktan setiltrimetilamonium bromide 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 to 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 NaA [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 molecule 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 zeolite have channel with different dimensions. Several investigations have carried out using directing has controlled the morphology of MTT and MFI using TMPD and HMPD as linier 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 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 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 waste bagasse 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 of 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 the content of SiO_2, Al_2O_3 and others oxide by AAS. NaOH pellets, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 filtered, washed, dried and calcined

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 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 filtering, washing, drying and calcination at 500°C for 5 h. The product 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 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 product synthesis 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 = 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 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 300–420 cm^{-1} is pore opening and 500–650 cm^{-1} is

?. no peak less than 400 cm⁻¹

Table 1. The composition of precursor and surfactant

Sample Code	Ratio of Precursor (v/v) Sodium silicate	Sodium aluminate	Surfactant CTAB (M)
A	1	1	0
A1	1	1	$5x10^{-4}$ (1/2 cmc)
A2	1	1	$1x10^{-3}$ (cmc)
A3	1	1	$1x10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5x10^{-4}$ (1/2 cmc)
B2	15	1	$1x10^{-3}$ (cmc)
B3	15	1	$1x10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5x10^{-4}$ (1/2 cmc)
C2	25	1	$1x10^{-3}$ (cmc)
C3	25	1	$1x10^{-2}$ (10 cmc)

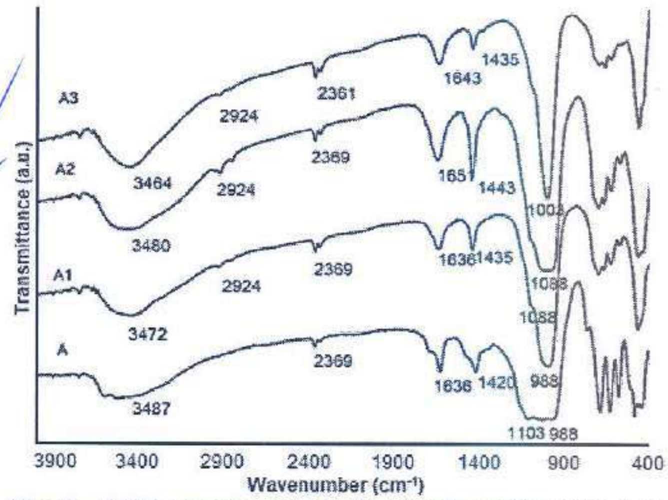

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB $5x10^{-4}$ M (A2) with CTAB $1x10^{-3}$ M (A3) with CTAB $1x10^{-2}$ M

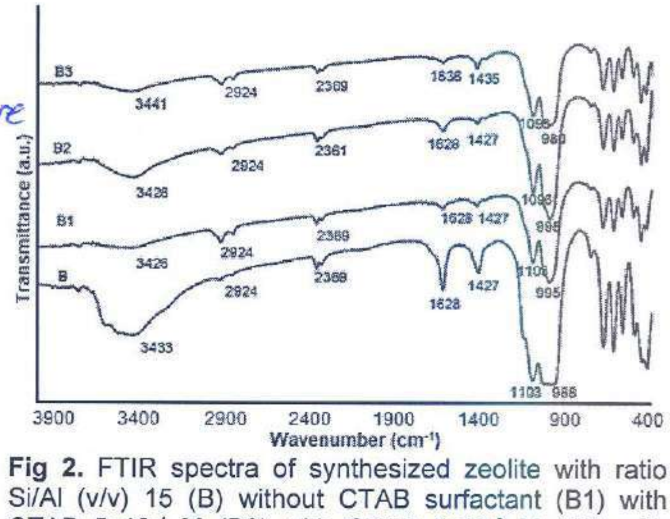

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB $5x10^{-4}$ M (B2) with CTAB $1x10^{-3}$ M (B3) with CTAB $1x10^{-2}$ M

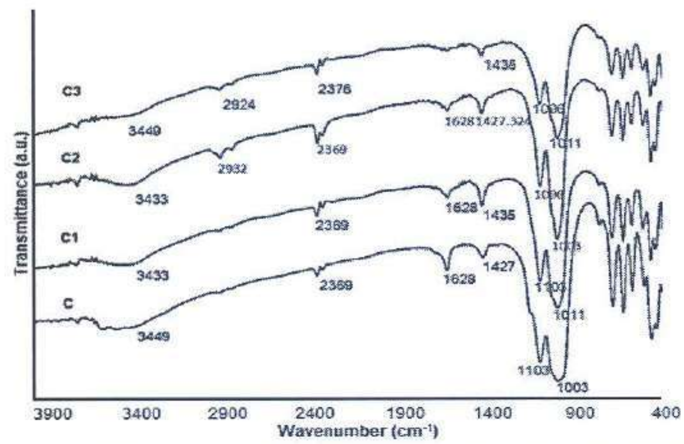

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5x10^{-4}$ M (C2) with CTAB $1x10^{-3}$ M (C3) with CTAB $1x10^{-2}$ M

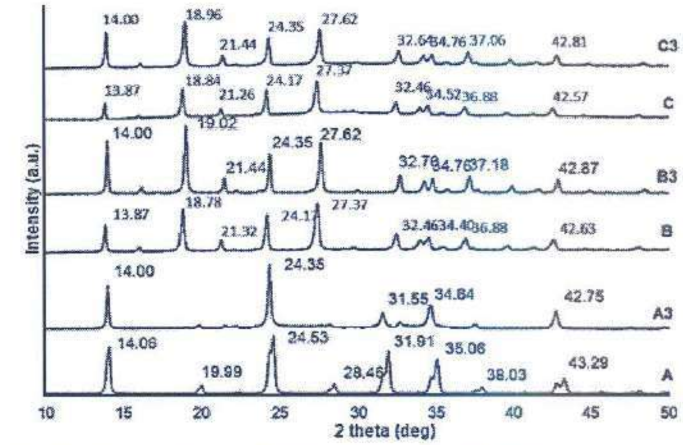

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 product synthesis 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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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. Beside 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

Fig. 4 shows the pattern of diffractogram XRD of product synthesis. 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 product synthesis by CTAB different with product synthesis without CTAB. In addition, the composition of precursor also affected the kind of product. The product synthesis 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 product synthesis 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 product synthesis 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 product synthesis. From the XRD data, the products by ratio Si/Al (v/v) 1 is sodalite and from the SEM it appears 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

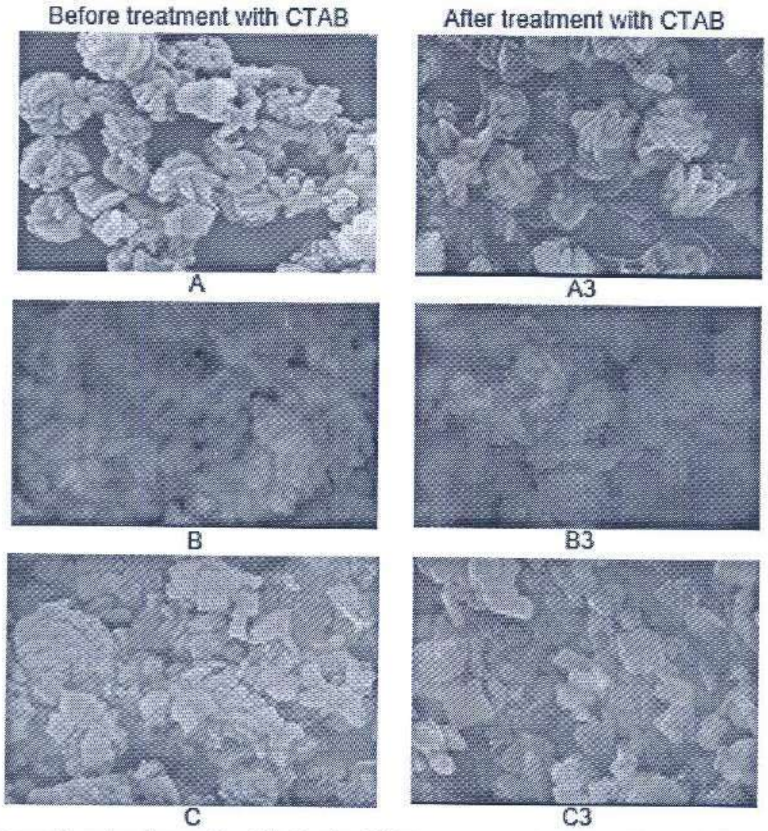

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 number of ions silicate and aluminate that interacted to form the product, so the using of surfactant

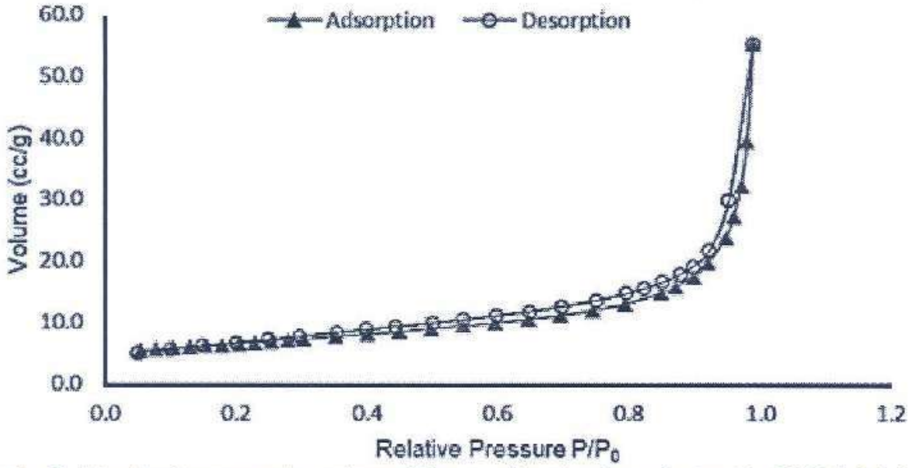

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

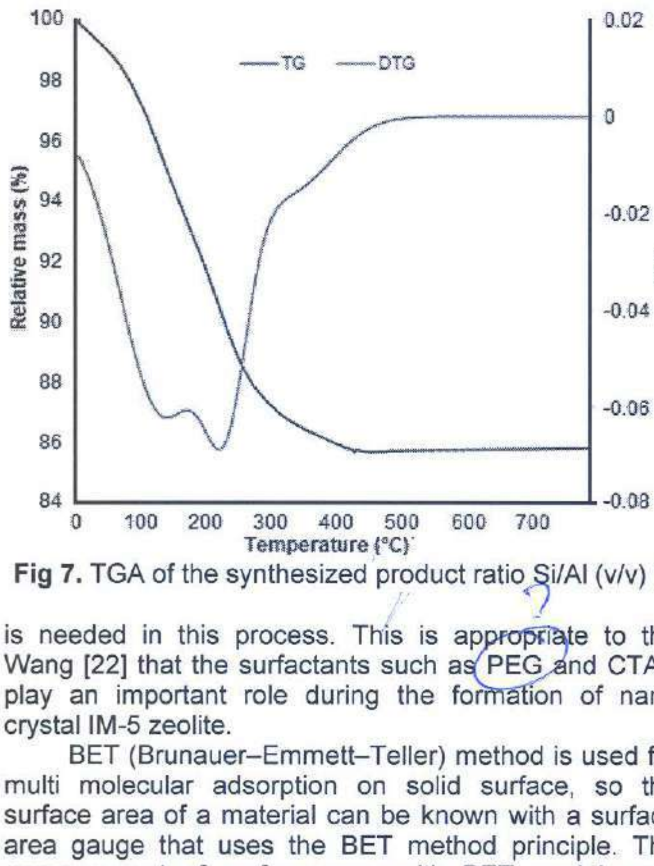

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

is needed in this process. This is appropriate to the Wang [22] that the surfactants such as PEG 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 m^2/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} cm^3/g and average pore radius was determined as 7.737°A. It means the NaP1 of this research have 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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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Synthesis of Zeolite from Sugarcane Bagasse Ash
Using Cetyltrimethylammonium Bromide as Structure Directing Agent

Sriatun, Taslimah, and Linda Suyati

ABSTRACT

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300-370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 NaA [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 linier 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, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 = 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 of spectra were indicating that all the product of synthesis had Si-O-Si or Si-O-Al groups as a specific

Table 1. The composition of precursor and surfactant

Sample Code	Ratio of Precursor (v/v) Sodium silicate	Sodium aluminate	Surfactant CTAB (M)
A	1	1	0
A1	1	1	$5x10^{-4}$ (1/2 cmc)
A2	1	1	$1x10^{-3}$ (cmc)
A3	1	1	$1x10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5x10^{-4}$ (1/2 cmc)
B2	15	1	$1x10^{-3}$ (cmc)
B3	15	1	$1x10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5x10^{-4}$ (1/2 cmc)
C2	25	1	$1x10^{-3}$ (cmc)
C3	25	1	$1x10^{-2}$ (10 cmc)

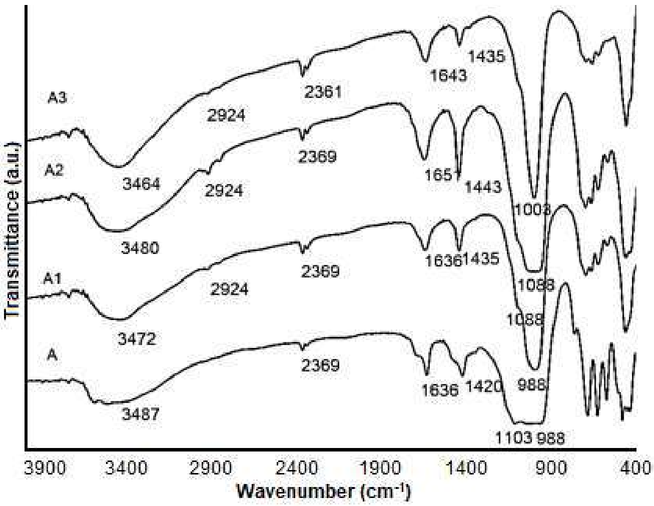

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB $5x10^{-4}$ M (A2) with CTAB $1x10^{-3}$ M (A3) with CTAB $1x10^{-2}$ M

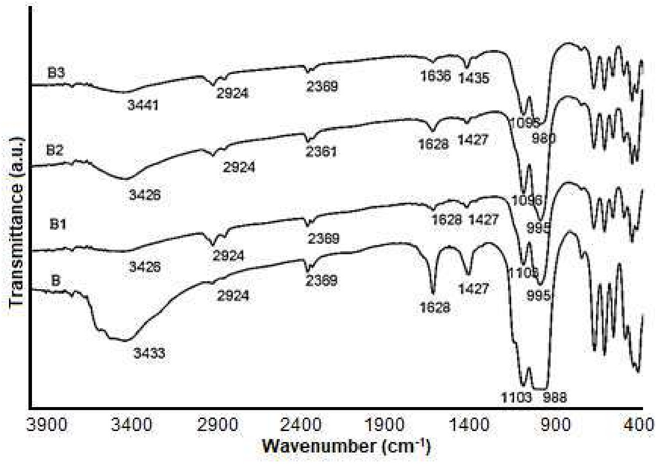

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB $5x10^{-4}$ M (B2) with CTAB $1x10^{-3}$ M (B3) with CTAB $1x10^{-2}$ M

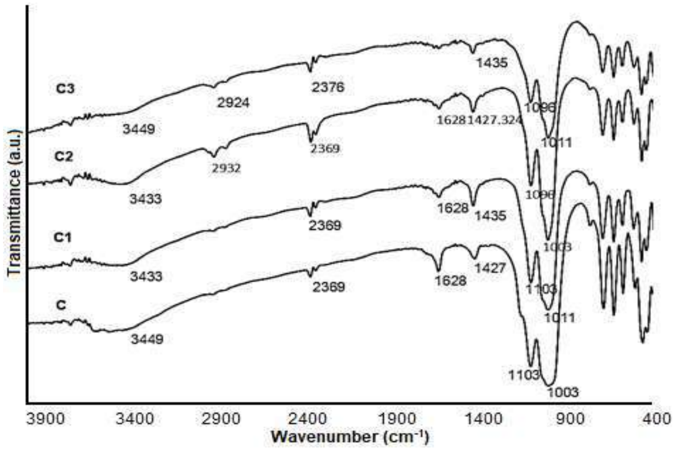

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5x10^{-4}$ M (C2) with CTAB $1x10^{-3}$ M (C3) with CTAB $1x10^{-2}$ M

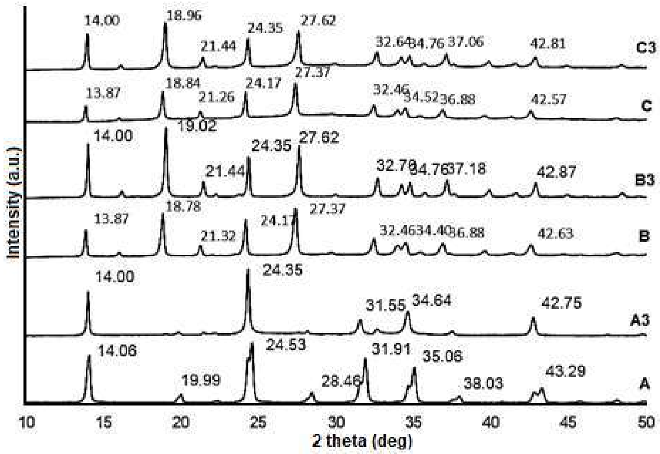

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

fingerprint of zeolite type. The absorption at 500–650 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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

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 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.

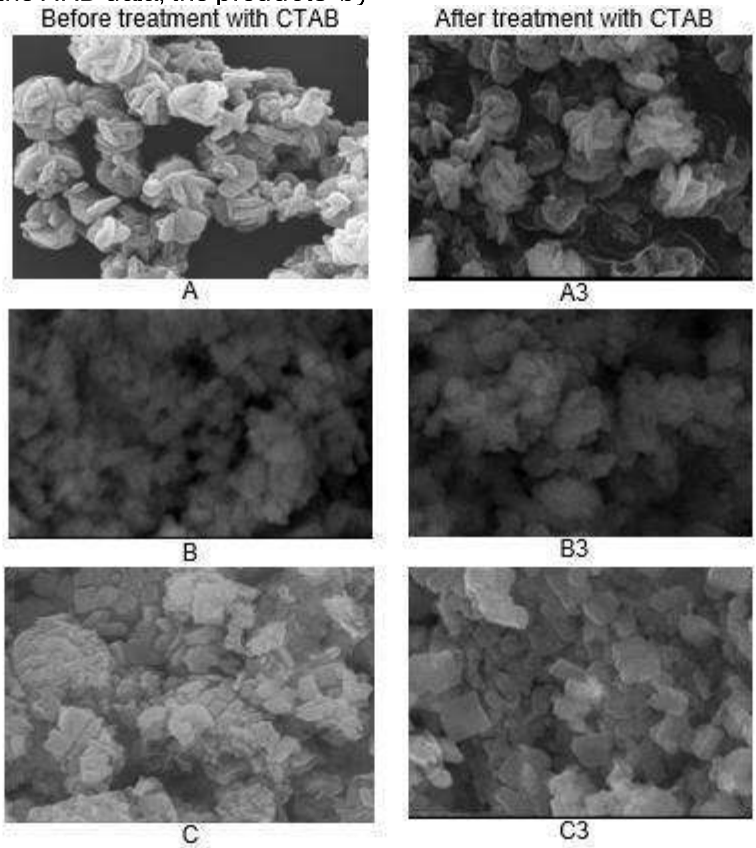

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1x10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1x10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1x10^{-2} M

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 1x10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1x10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1x10^{-2} M

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

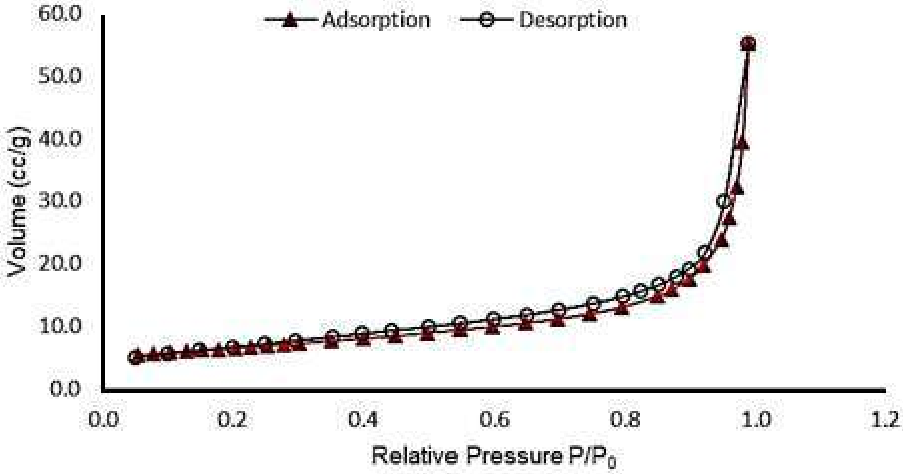

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

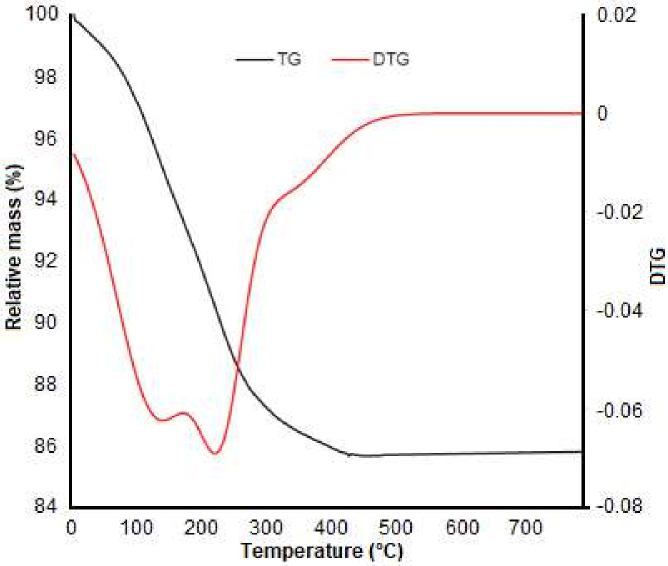

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56x10^{-2} cm^3/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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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Title: Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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

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

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Abstract

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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 $5x10^{-4}$ M, $1x10^{-3}$ M and $1x10^{-2}$ M. The result showed all of product have strong absorbance at 950–1050 cm^{-1} and 620–690 cm^{-1}, 420–460 cm^{-1}, double ring at 520–570 cm^{-1}, pore opening at 300–370 cm^{-1}. Vibration of –OH as silanol group or water was indicated by broad absorbance at 3400–3450 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 $5x10^{-4}$ M, $1x10^{-3}$ M dan $1x10^{-2}$ M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 cm^{-1}, 620–690 cm^{-1}, 420–460 cm^{-1}. Vibrasi double ring pada 520–570 cm^{-1}, pore opening 300–370 cm^{-1}. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada 3400–3450 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 applicated 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 NaA [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 linier 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, $Al(OH)_3$ 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 Kα radiation (λ = 1.54184 Å) 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 = 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) Sodium silicate	Sodium aluminate	Surfactant CTAB (M)
A	1	1	0
A1	1	1	$5x10^{-4}$ (1/2 cmc)
A2	1	1	$1x10^{-3}$ (cmc)
A3	1	1	$1x10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5x10^{-4}$ (1/2 cmc)
B2	15	1	$1x10^{-3}$ (cmc)
B3	15	1	$1x10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5x10^{-4}$ (1/2 cmc)
C2	25	1	$1x10^{-3}$ (cmc)
C3	25	1	$1x10^{-2}$ (10 cmc)

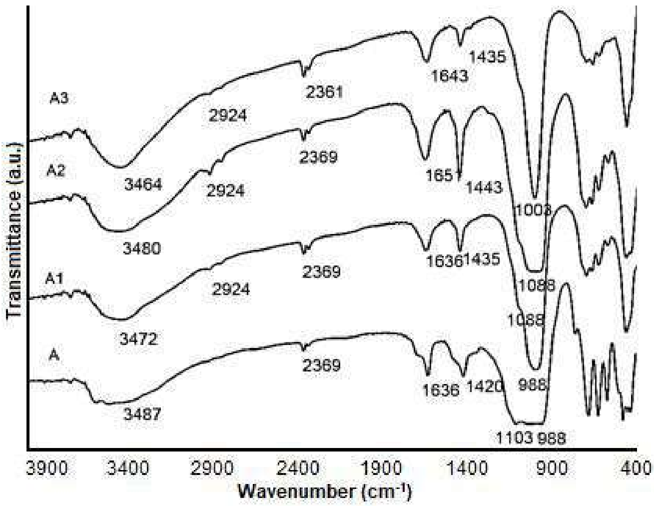

Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB $5x10^{-4}$ M (A2) with CTAB $1x10^{-3}$ M (A3) with CTAB $1x10^{-2}$ M

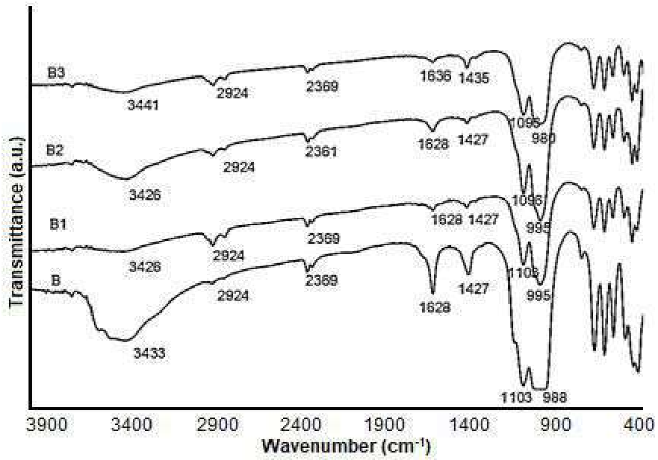

Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB $5x10^{-4}$ M (B2) with CTAB $1x10^{-3}$ M (B3) with CTAB $1x10^{-2}$ M

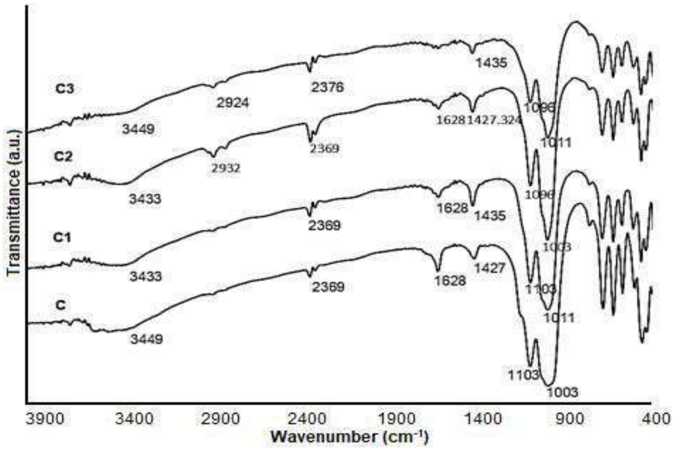

Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5x10^{-4}$ M (C2) with CTAB $1x10^{-3}$ M (C3) with CTAB $1x10^{-2}$ M

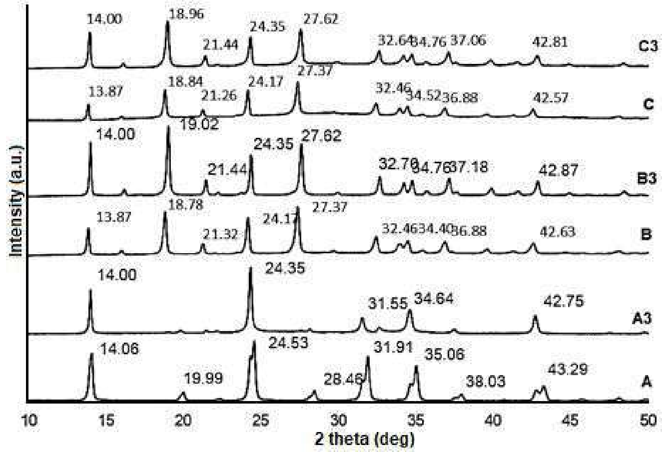

Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 500–650 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/$[SiO_4]^{4-}$ and aluminate/$[AlO_4]^{5-}$ 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 950–1250 and 3400–3500 cm^{-1}. The lower absorption intensity of wave number at 3400–3500 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 950–1250 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. Thus the shape and pore size of modified zeolite was expected homogenously.

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

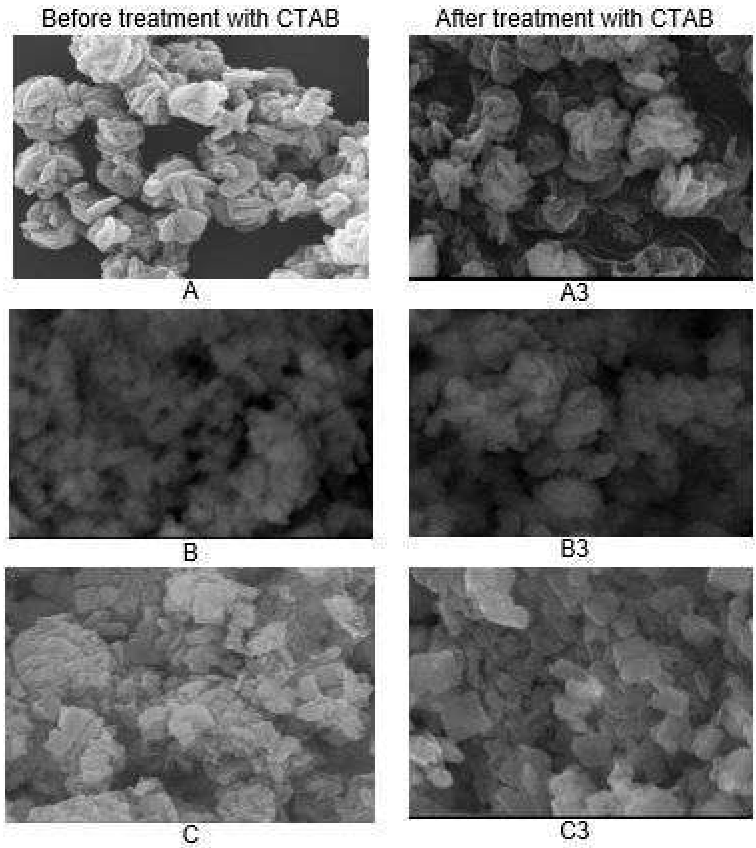

Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ M

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 $1x10^{-2}$ M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1x10^{-2}$ M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1x10^{-2}$ 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

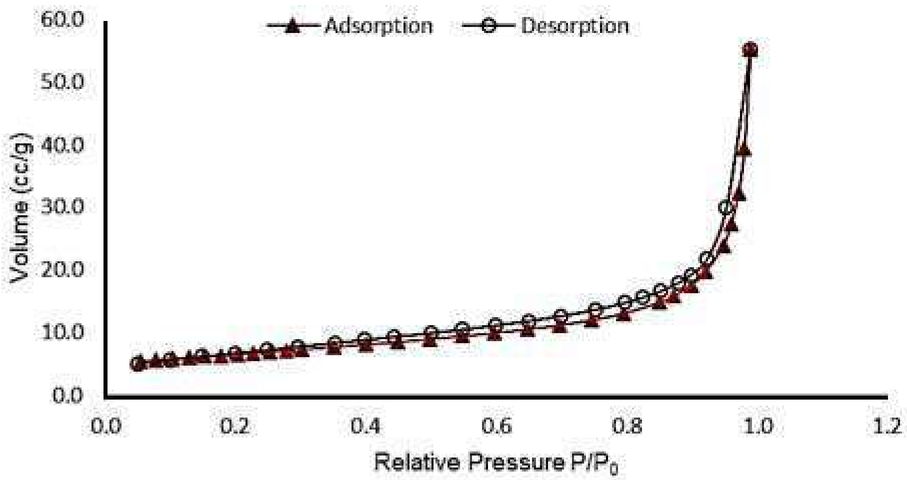

Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

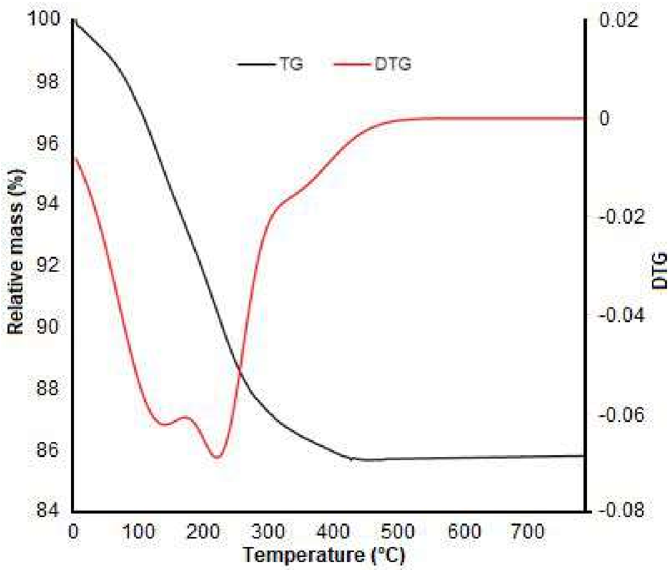

Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m^2/g. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56x10^{-2} cm^3/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 resides 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 SiO_4^{4-} and AlO_4^{5-} 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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Menerangkan bahwa artikel yang berjudul **Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent** yang terbit pada Jurnal Indonesian Journal of Chemistry (IJC) Vol. 18 No.1 benar-benar merupakan paper final yang dihasilkan dari review manuskrip yang disubmit dengan judul mula-mula **The Effect of Cetyltrimethylammonium Bromide Surfactant in Synthesis of Zeolite from Sugarcane Bagasse Ash**. Perubahan judul ini merupakan rekomendasi setelah proofreading akhir paper layout, sebelum terbit, untuk meningkatkan relevansi antara judul dan isi penelitian yang dilaporkan.

Demikian surat keterangan ini kami buat dengan sesungguhnya, agar dapat dipergunakan sebagaimana mestinya.

Yogyakarta, 17 Oktober 2020
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