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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

Jumlah Penulis : 3 orang

Status Pengusul : Penulis Utama

Identitas Jurnal Ilmiah :

- a. Nama Jurnal : Indonesian Journal of Chemistry
- b. Nomor ISSN : 1411-9420 (print); 2460-1578 (on-line)
- c. Vol, No., Bln Thn : Vol 18, No 1 (2018), Hal. 159 -165
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- e. DOI artikel (jika ada) : <https://doi.org/10.22146/ijc.22197>
- f. Alamat web jurnal : <https://jurnal.ugm.ac.id/ijc/>
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- url Turnitin: (5%)
<https://doc-pak.undip.ac.id/3033/1/Turnitin1.pdf>
- g. Terindex : Jurnal Internasional bereputasi (Q3) terindeks SCOPUS (SJR=0,215)
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☐ Jurnal Ilmiah Nasional Tidak Terakreditasi

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Komponen Yang Dinilai	Nilai Maksimal Jurnal Ilmiah			Nilai Akhir Yang Diperoleh
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a. Kelengkapan unsur isi jurnal (10%)	4,00			4
b. Ruang lingkup dan kedalaman pembahasan (30%)	12,00			12
c. Kecukupan dan kemutakhiran 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
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Isi artikel ini sudah memenuhi kelengkapan sebuah jurnal ilmiah, unsur-unsur jurnal juga sudah sesuai dan sinkron. Judul abstrak, pendahuluan sampai kesimpulan lengkap. Nilai 4

2. Ruang lingkup dan kedalaman pembahasan:

Pembahasan pada makalah ini sudah baik. Metode penelitian dan hasil disampaikan dengan baik dan didukung oleh referensi yang memadai. Nilai 12

3. Kecukupan dan kemutakhiran data/informasi dan metodologi:


Metode yang digunakan dalam artikel ini cukup mutakhir dan referensi yang digunakan sudah mencukupi dan mutakhir (< 10 th), data baik dan dibahas dengan baik. Nilai 12

4. Kelengkapan unsur dan kualitas terbitan:

Terlihat jurnal ini berkuwalitas dan unsur-unsurnya lengkap mencakup cover, dewan redaksi dan daftar isi semua memenuhi syarat jurnal ilmiah. Nilai 11,6

Semarang, 30 Maret 2020

Reviewer 1



Dr. Bambang Cahyono
 NIP. 196303161988101001

Unit Kerja : Departemen Kimia FSM UNDIP

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g. Kecukupan dan kemutakhiran data/informasi dan metodologi (30%)	12,00			12
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Penulis Utama: 0,6 x 39 = 23,4				

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Artikel yang dimuat, dibahas dan disusun secara lengkap. Nilai 4

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Data yang disajikan dibahas dengan detil dan lengkap, disertai referensi pendukung. Nilai 12

3. Kecukupan dan kemutakhiran data/informasi dan metodologi:

Kualitas dan kuantitas pustaka baik, sebagian besar tahun terbit kurang dari 10 th (90%).

Metodologi disajikan detil sehingga bisa diulang peneliti lain. Nilai 12

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

Unit Kerja : Departemen Kimia FSM UNDIP

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW
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b. Ruang lingkup dan kedalaman pembahasan (30%)	12	12	12
c. Kecukupan dan kemutakhiran 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): $0,6 \times 39,3 = 23,58$			

Semarang, 30 Maret 2020

Reviewer 2



Drs. Gunawan, M.Si, Ph.D
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Reviewer 1



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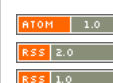
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Artikel 2 negara, abstrak terlampir

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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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(2) Afe Babalola University, Ado - Ekiti

(3) Afe Babalola University, Ado - Ekiti

(4) Afe Babalola University, Ado - Ekiti

(*) Corresponding Author

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:

References

- [1] Volesky, B., and Holan, Z.R., 1995, Biosorption of heavy metals, *Biotechnol. Prog.*, 11 (3), 235–250.
- [2] Pagana, A.E., Sklari, S.D., Kikkinides, E.S., and Zaspalis, V.T., 2008, Microporous ceramic membrane technology for the removal of arsenic and chromium ions from contaminated water, *Microporous Mesoporous Mater.*, 110 (1), 150–156.
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Abstrak artikel 2 negara ke 2

Improvement of Cycling Performance of Na_{2/3}Co_{2/3}Mn_{1/3}O₂ Cathode by PEDOT/PSS Surface Coating for Na Ion Batteries <https://doi.org/10.22146/ijc.24893>Yatim Lailun Ni'mah^(1*), Ju Hsiang Cheng⁽²⁾, Ming Yao Cheng⁽³⁾, Wei Nien Su⁽⁴⁾, Bing Joe Hwang⁽⁵⁾

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Abstract

The surface-modified Na_{2/3}Co_{2/3}Mn_{1/3}O₂ 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}O₂ 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}O₂ 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

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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>

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Abstract

The purpose of this research is to synthesize zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent. This research used cetyltrimethylammonium bromide surfactant to invent the high porosity, surface area, acidity and thermal stability of synthesized zeolite. The Silica was extracted by alkaline fusion using sodium hydroxide solution. The synthesis was conducted by hydrothermal process at 100 °C for 7 days, ageing process for 24 h and calcination at 500 °C for 5 h. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950\text{--}1050\text{ cm}^{-1}$ and $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$, double ring at $520\text{--}570\text{ cm}^{-1}$, pore opening at $300\text{--}370\text{ cm}^{-1}$. Vibration of -OH as silanol group or water was indicated by broad absorbance at $3400\text{--}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO₂ 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

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Synthesis of zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent (Article)

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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 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950\text{--}1050\text{ cm}^{-1}$ and $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$, double ring at $520\text{--}570\text{ cm}^{-1}$, pore opening at $300\text{--}370\text{ cm}^{-1}$. Vibration of --OH as silanol group or water was indicated by broad absorbance at $3400\text{--}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO_2 quartz and contain 12.23% and 12.19% of Si, 4.17% and 13.18% of Al, respectively. Observation on SEM revealed that the crystal produced using cetyltrimethylammonium bromide was more homogenous and regular in shape. © 2018, Gadjah Mada University. All rights reserved.

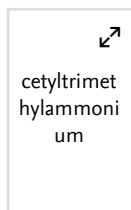
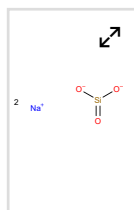
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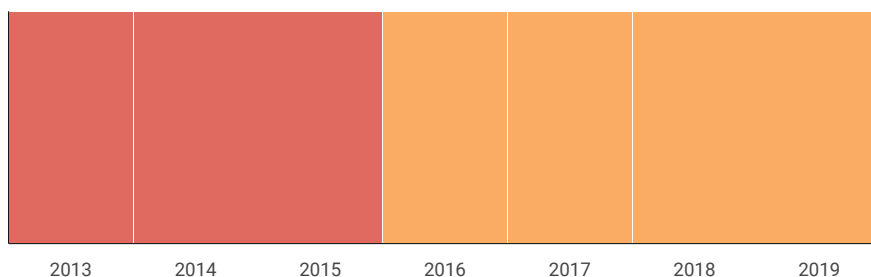
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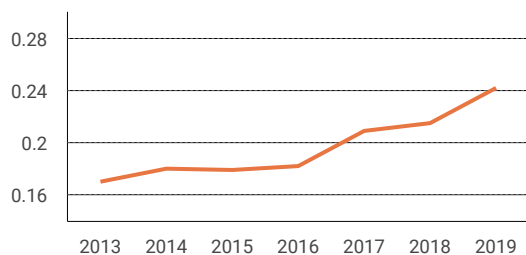
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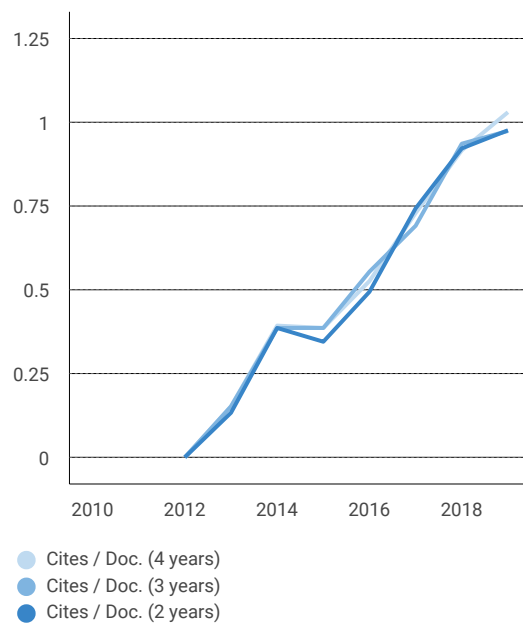
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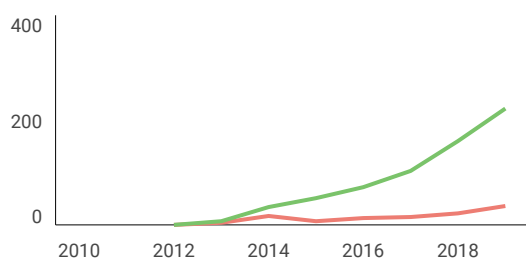
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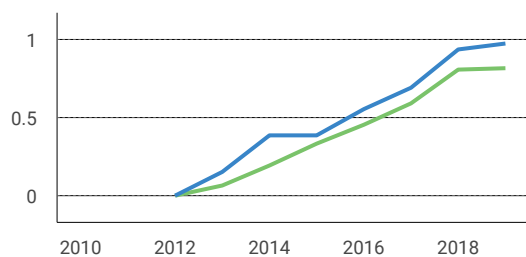
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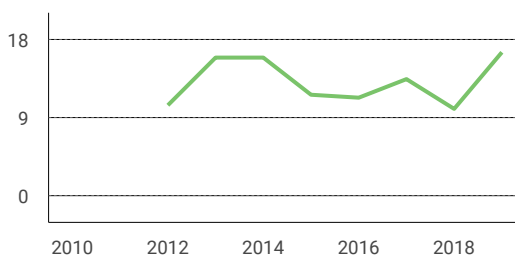
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Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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Received February 18, 2017; Accepted October 24, 2017

ABSTRACT

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

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

ABSTRAK

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

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

INTRODUCTION

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

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

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

There are many methods 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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

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

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

Procedure

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

RESULT AND DISCUSSION

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

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

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

Table 1. The composition of precursor and surfactant

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

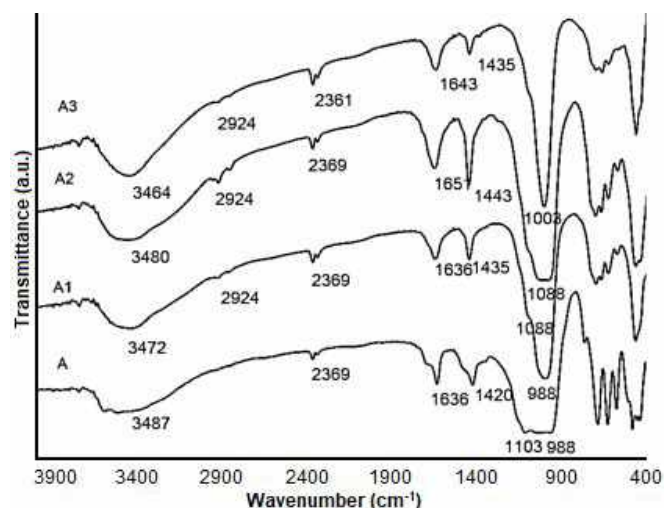


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

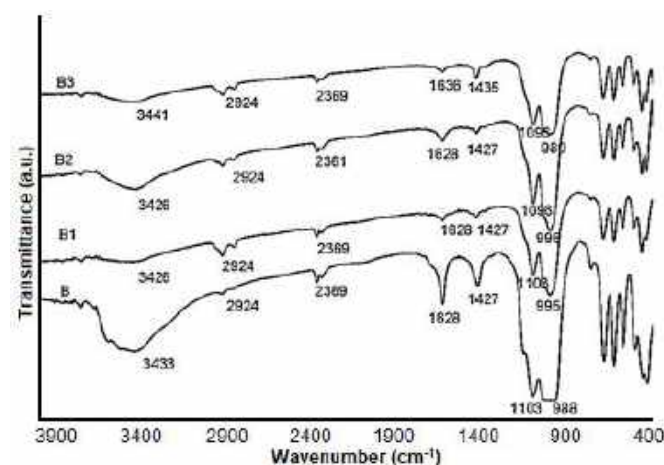


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

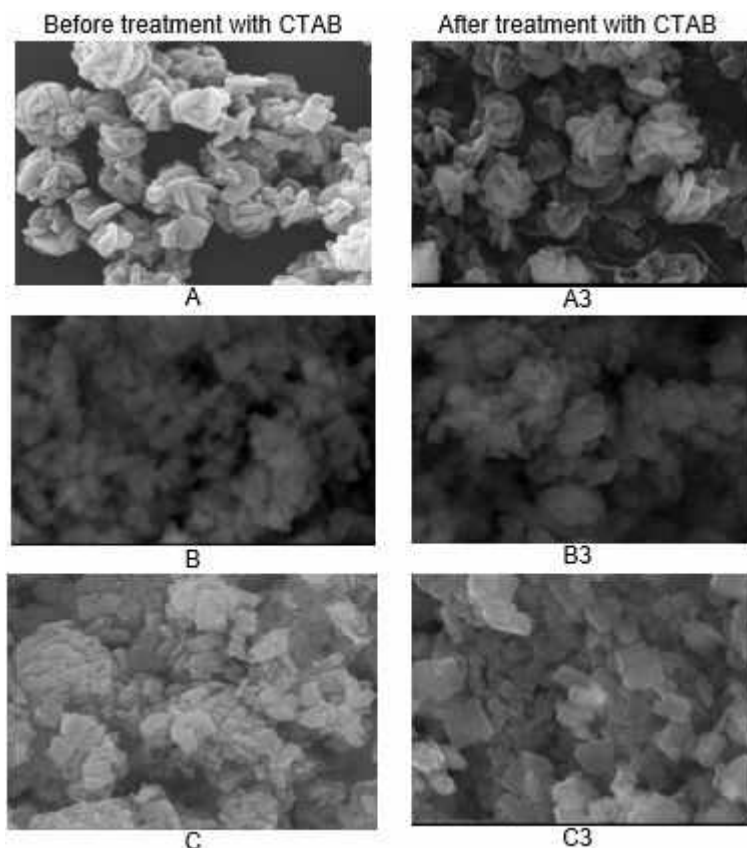


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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

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

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

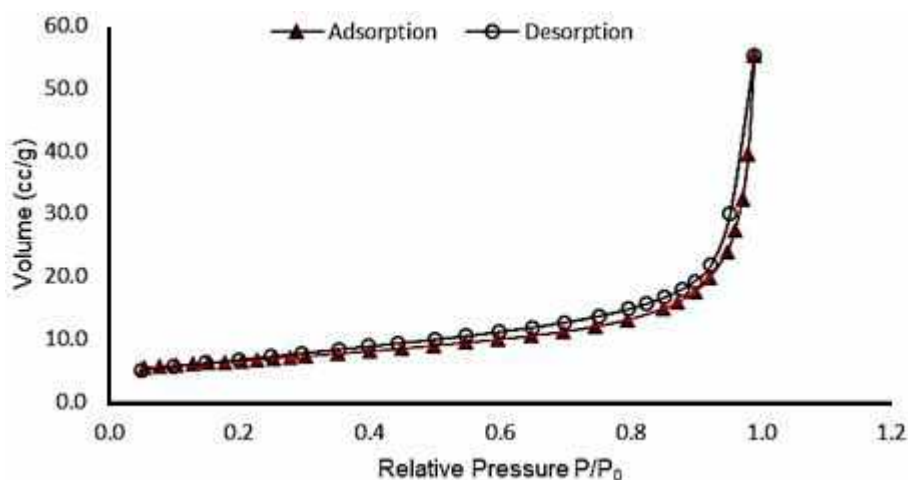


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

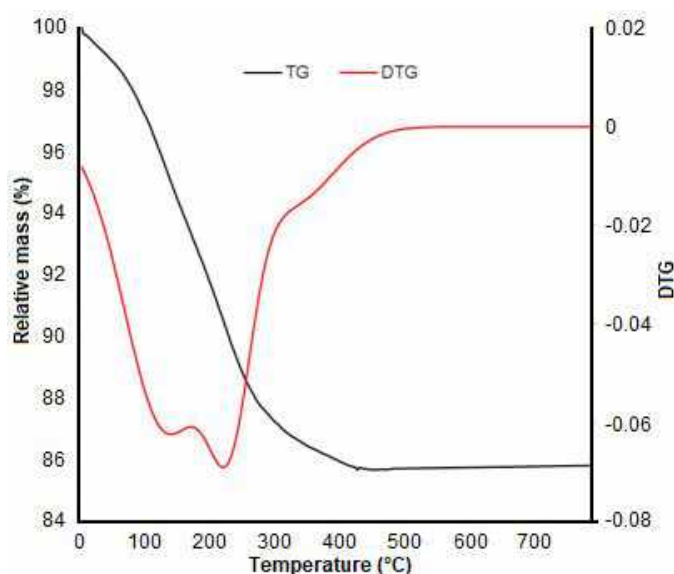


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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

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

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

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

CONCLUSION

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

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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

by Sriatun Sriatun

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File name: Cetyltrimethylammonium_Bromide_as_Structure_Directing_Agent.pdf (429.2K)

Word count: 4250

Character count: 21744

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Received February 18, 2017; Accepted October 24, 2017

ABSTRACT

The purpose of this research is to synthesize zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent. This research used cetyltrimethylammonium bromide surfactant to invent the high porosity, surface area, acidity and thermal stability of synthesized zeolite. The Silica was extracted by alkaline fusion using sodium hydroxide solution. The synthesis was conducted by hydrothermal process at 100 °C for 7 days, ageing process for 24 h and calcination at 500 °C for 5 h. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at 950–1050 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 SiO₂ quartz and contain 12.23% and 12.19% of Si, 4.17% and 13.18% of Al, respectively. Observation on SEM revealed that the crystal produced using cetyltrimethylammonium bromide was more homogenous and regular in shape.

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

ABSTRAK

Tujuan penelitian ini adalah membuat zeolit dari ampas tebu menggunakan setiltrimetilamonium bromida sebagai agen pengarah struktur. Dalam penelitian ini surfaktan setiltrimetilamonium bromida digunakan untuk meningkatkan porositas, luas permukaan, keasaman dan stabilitas termal. Silika diekstrak dari abu ampas tebu melalui penambahan alkali yaitu NaOH 6 M. Sintesis dilakukan secara hidrotermal pada 100 °C selama 7 hari, ageing selama 24 jam dan kalsinasi pada 500 °C selama 5 jam. Variasi rasio silikat/aluminat (v/v) adalah 1, 15 and 25, sedangkan variasi konsentrasi surfaktan 5×10^{-4} M, 1×10^{-3} M dan 1×10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 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 SiO₂ 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-biogas 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% P₂O₅ and 0.38% K₂O. 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^2, N^3 -tetramethylpropane-1,3-diamine) and HMPD ($N^1, N^1, N^2, N^2, 2, 2$ -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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

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

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

Procedure

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

RESULT AND DISCUSSION

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

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

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

Table 1. The composition of precursor and surfactant

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

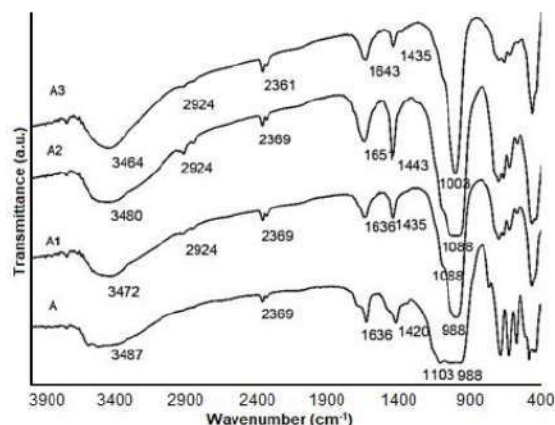


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

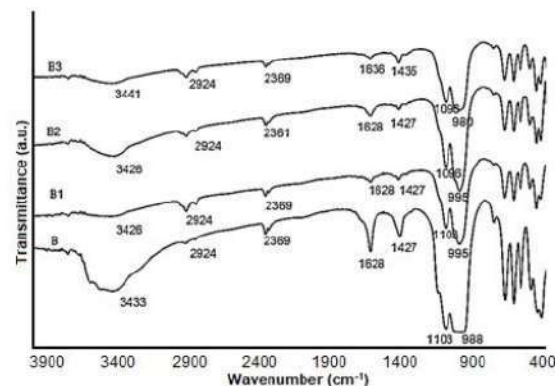


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

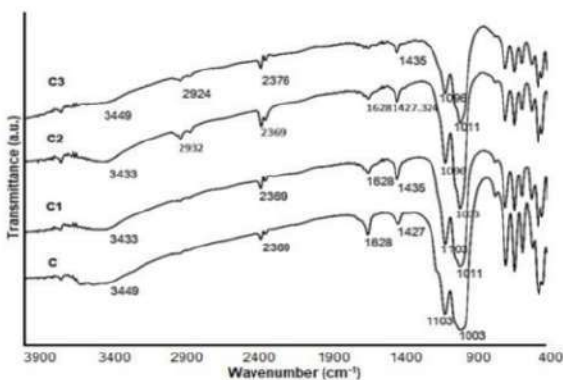


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5×10^{-4} M (C2) with CTAB 1×10^{-3} M (C3) with CTAB 1×10^{-2} M

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

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

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

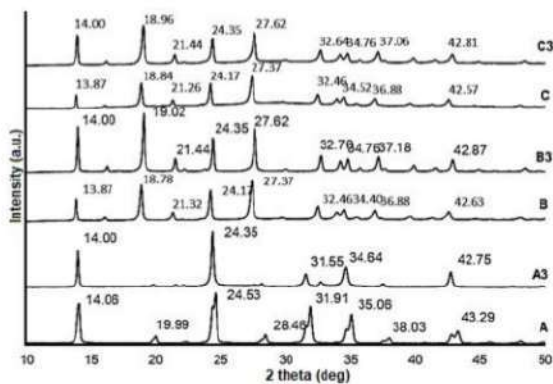


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Thus the shape and pore size of modified zeolite was expected 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

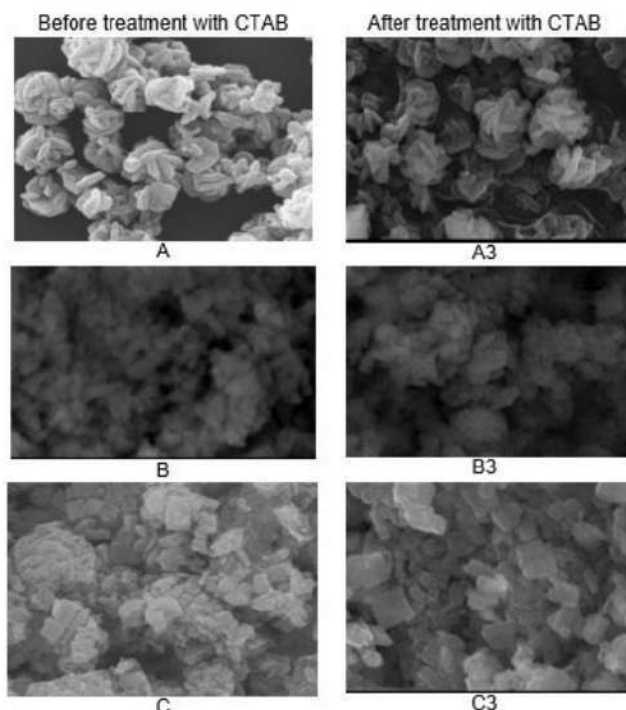


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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

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

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

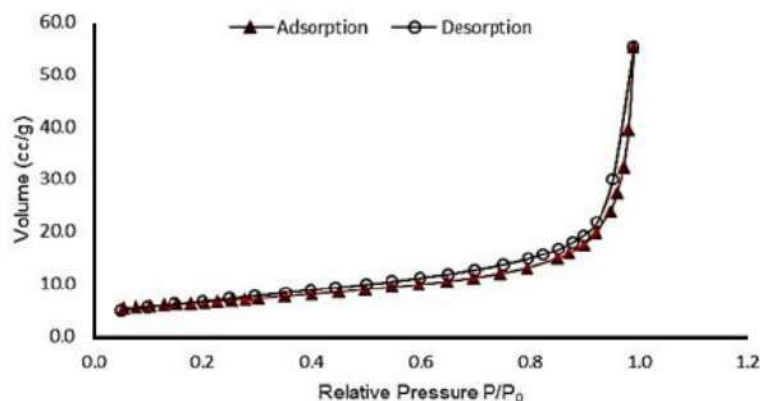


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

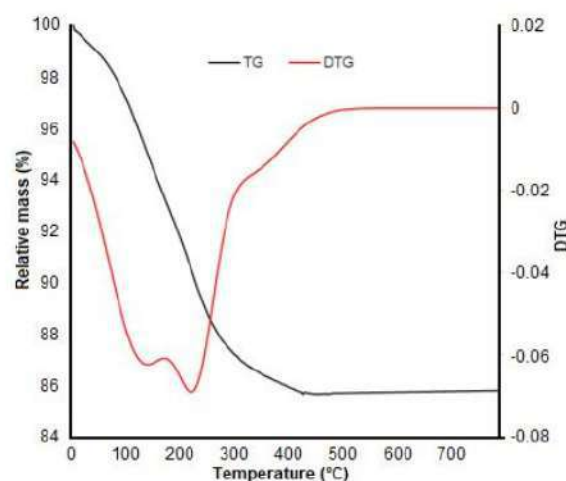


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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

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

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

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

CONCLUSION

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

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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

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INDONESIAN JOURNAL OF CHEMISTRY (IJC), 18 (1), 159-165
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No.	Tanggal	Aktivitas Korespondensi
1.	17 Februari 2017	Pembuatan akun pada Jurnal Indonesian Journal of Chemistry (IJC) (Lampiran 1)
2.	18 Februari 2017	Proses submission Tanggal submit artikel: 18 Februari 2017 Menerima email dari IJC ID: 22197- 41689 Judul pengajuan awal: The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash Penulis: Sriatun, Taslimah, Linda Suyati (Lampiran 2)
3.	Maret 2017	Status artikel: Awaiting Assignment (Maret 2017) (lampiran 3)
4.	28 Juni 2017	Proses review Menerima email dari editor IJC <ul style="list-style-type: none"> • permintaan perbaikan artikel (revisi) • Comment reviewer A dan B (Lampiran 4)
5.	28 Juni 2017	Komentar reviewer A dan B Reviewer A: komentar ada pada email Reviewer B: komentar pada email dan attachment (Lampiran 5)
6.	28 Juni 2017	Memperbaiki artikel <ul style="list-style-type: none"> • daftar perbaikan Round 1 (lampiran 6)
7.	19 Juli 2017	Mengirimkan artikel perbaikan dan daftar perbaikan ke editor. (Lampiran 7)
8.	25 Juli 2017	Menerima email dari editor IJC bahwa artikel memerlukan perbaikan Round 2 (Lampiran 8)
9.	25 Juli 2017	Memperbaiki artikel <ul style="list-style-type: none"> • Daftar perbaikan Round 2 (lampiran 9)
10.	30 Juli 2017	Menyampaikan artikel yang sudah direvisi Round 2 beserta daftar perbaikannya

		(Lampiran 10)
11.	5 Agustus 2017	Menerima email dari editor <ul style="list-style-type: none"> • Status: Accepted (Lampiran 11)
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13.	15 September 2017	Memperbaiki copyediting
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15.	24 Oktober 2017	Menyampaikan copyediting revisi Mengganti data BET adsorpsi desorpsi (menggantikan data analisis BET adsorpsi saja)
16.	30 Oktober 2017	Menerima permintaan proof reading
17.	2 November 2017	Pengiriman hasil proof reading
18.	4 November 2017	Menerima email dari editor <ul style="list-style-type: none"> - Menyarankan untuk mengubah judul
19.	5 November 2017	Mengirimkan hasil revisi judul pada proof reading Judul setelah perubahan: <i>Synthesis of zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent</i> Penulis: Sriatun, Taslimah, Linda Suyati
20.	13 Desember 2017	Menerima email informasi mengenai galley format
21.	13 Februari 2018	Menerima email permintaan koreksi akhir
22.	15 Februari 2018	Pengiriman koreksi akhir terhadap artikel
23.	28 Februari 2018	Menerima email pemberitahuan penerbitan artikel
24.	28 Februari 2018	Published 28 Februari 2018 Indonesian Journal of Chemistry Vol 18 No. 1 Tahun 2018 Judul: <i>Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent</i> Penulis: Sriatun, Taslimah, Linda Suyati
26.	17 Oktober 2020	Surat Keterangan Adanya perubahan Judul dari Editor



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Submission

Authors	Sriatun Sriatun, Taslimah Taslimah, Linda Suyati
Title	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
Original file	22197-41689-1-SM.DOCX 2017-02-18
Supp. files	None
Submitter	Mrs Sriatun Sriatun
Date submitted	February 18, 2017 - 09:27 PM
Section	Articles
Editor	Nuryono Nuryono
Abstract Views	1547

Author Fees

Article Submission	0.00 USD	PAY NOW
Article Publication	Paid December 13, 2017 - 10:18 AM	

Status

Status	Published Vol 18, No 1 (2018)
Initiated	2018-02-28
Last modified	2018-02-28

Submission Metadata

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Principal contact for editorial correspondence.	
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Bio Statement	Chemistry Department
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Affiliation	Diponegoro University
Country	Indonesia
Bio Statement	Chemistry Department

Title and Abstract

Tanggal submit artikel:
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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 $5 \cdot 10^{-4}$ M, 10^{-3} M and 10^{-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 SiO₂ 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 $5 \cdot 10^{-4}$ 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 SiO₂ 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 1 dan 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 continuously. 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 hydrothermal 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 cetyltrimethylammoniumbromide 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°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, $\text{Al}(\text{OH})_3$ 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°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°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°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°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. Cetyltrimethylammonium 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 $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ 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 ($\text{T} = \text{Si}$ or Al) and appears at $950\text{-}1250\text{ cm}^{-1}$ wavenumber, whereas the symmetric stretching vibration at $650\text{-}720\text{ cm}^{-1}$ and 500 cm^{-1} . Furthermore, the wavenumber at $1050\text{-}1150\text{ cm}^{-1}$ was asymmetric stretching vibration, $750\text{-}820\text{ cm}^{-1}$ is symmetric 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 $300\text{-}420\text{ cm}^{-1}$ is pore opening and $500\text{-}650\text{ cm}^{-1}$ is double ring [22]. Double ring is specific character of zeolite type, because it was an external linkage 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°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 intensity 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 affect the type of 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 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 NaP1 contained 18.49 % Na, 17.25% Al 16.60% Si. The decreasing of silica in the product NaP1 was caused calcination treatment at 500°C. The remainder of silica was transformed to be SiO₂ quartz.

Conclusion

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

Acknowledgments

This research is financially supported by DRPM DIKTI

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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	$5 \cdot 10^{-4}$ (1/2 cmc)
A2	1	1	$1 \cdot 10^{-3}$ (cmc)
A3	1	1	$1 \cdot 10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5 \cdot 10^{-4}$ (1/2 cmc)
B2	15	1	$1 \cdot 10^{-3}$ (cmc)
B3	15	1	$1 \cdot 10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5 \cdot 10^{-4}$ (1/2 cmc)
C2	25	1	$1 \cdot 10^{-3}$ (cmc)
C3	25	1	$1 \cdot 10^{-2}$ (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

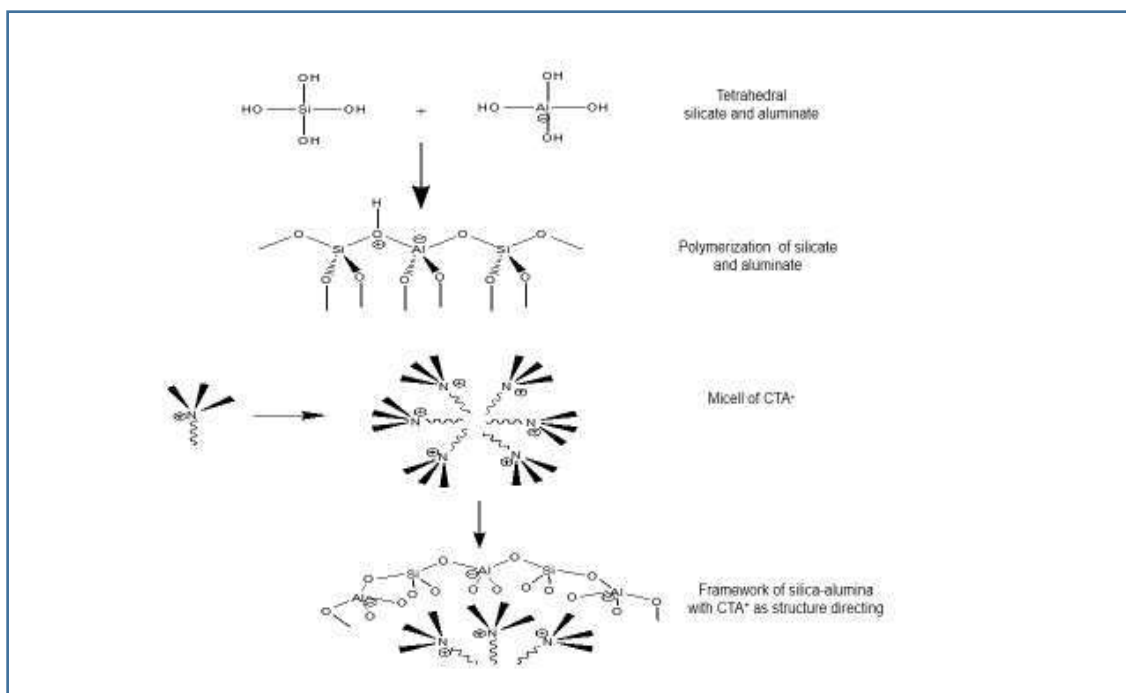


Figure 1. The mechanism of zeolite framework formation by CTA⁺ as structure directing

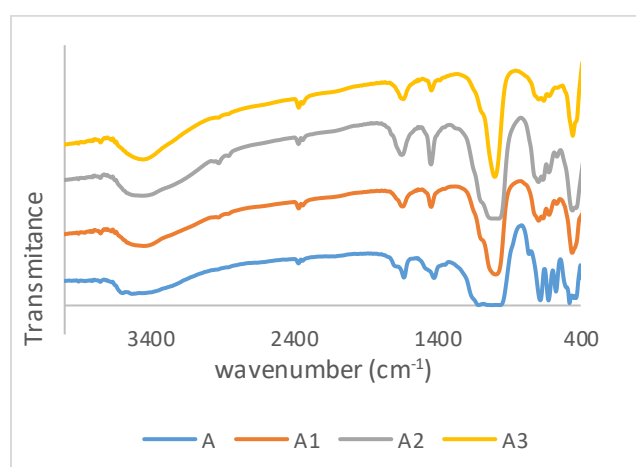


Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTA surfactant (A1) with CTA $5 \cdot 10^{-4} \text{ M}$ (A2) with CTA $1 \cdot 10^{-3} \text{ M}$ (A3) with CTA $1 \cdot 10^{-2} \text{ M}$

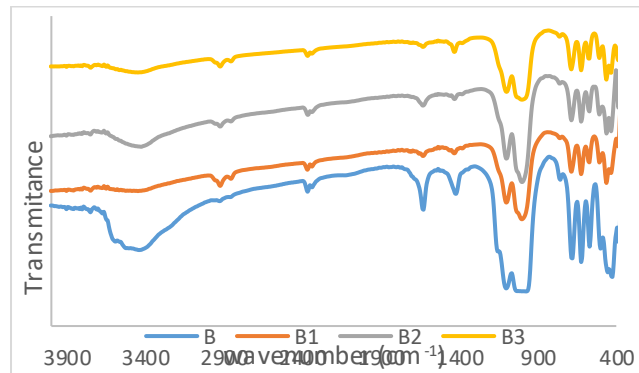


Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTA surfactant (B1) with CTA $5 \cdot 10^{-4}$ M (B2) with CTA $1 \cdot 10^{-3}$ M (B3) with CTA $1 \cdot 10^{-2}$ M

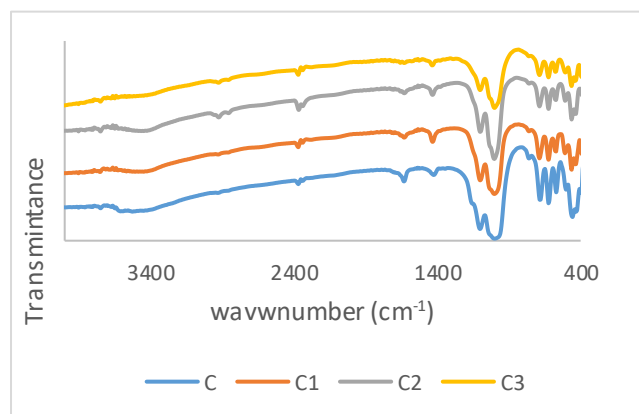


Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTA surfactant (C1) with CTA $5 \cdot 10^{-4}$ M (C2) with CTA $1 \cdot 10^{-3}$ M (C3) with CTA $1 \cdot 10^{-2}$ M

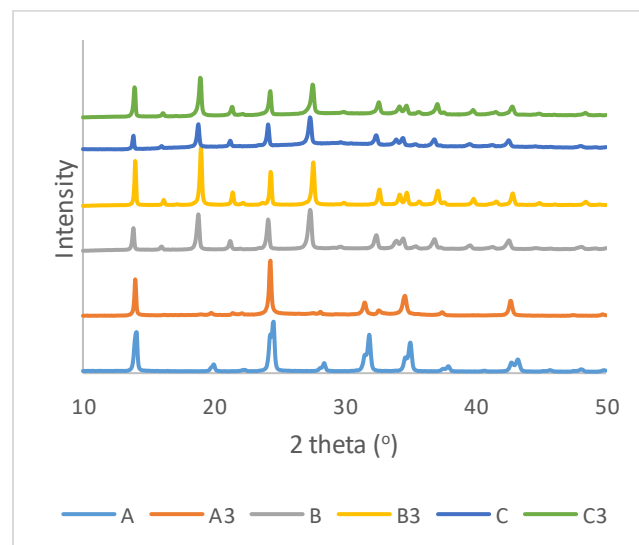
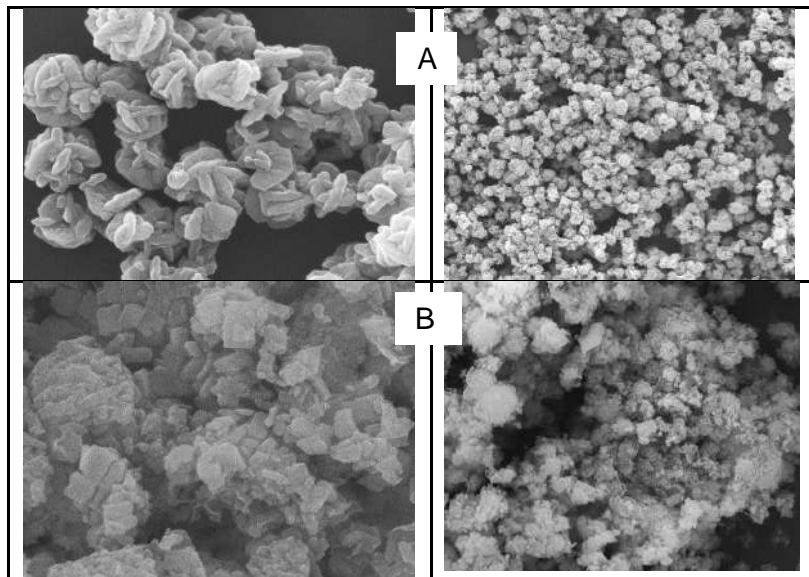


Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTA (A3) Ratio Si/Al 1 with CTA $1 \cdot 10^{-2}$ M (B) Ratio Si/Al 15 without CTA (B3) Ratio Si/Al 15 with CTA $1 \cdot 10^{-2}$ M (C) Ratio Si/Al 25 without CTA (C3) Ratio Si/Al 25 with CTA $1 \cdot 10^{-2}$ M



**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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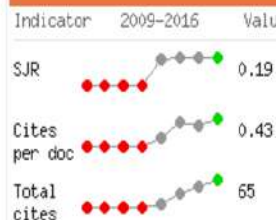
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Our decision is: Revisions Required

The comments of reviewers can be read below or/and in attachment

The Revised article has to be submitted within three weeks after receiving this email.

Best regards,

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1. The novelty of this research has not been described in detail, what advantage this study from previous similar studies, is there any importar breakthroughs resulting from this research?

2. Several additional data are necessarily required to support author's arguments within the manuscript (e.g. XRD and BET).

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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. Characterization data needs to be enriched by other characterization data fsuch as DTA and BET data.

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The present article reports the Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash. The results are great and worth to be published in this journal. However, 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?

2. Several additional data are necessarily required to support author's arguments within the manuscript (e.g. XRD and BET).

Reviewer B:

Additional Comment::

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. Characterization data needs to be enriched by other characterization data such as DTA and BET data.

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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 $5 \cdot 10^{-4}$ M, 10^{-3} M and 10^{-2} M. The result showed all of product have strong absorbance at $950\text{-}1050\text{ cm}^{-1}$ and $620\text{-}690\text{ cm}^{-1}$, $420\text{-}460\text{ cm}^{-1}$, double ring at $520\text{-}570\text{ cm}^{-1}$, pore opening at $300\text{-}370\text{ cm}^{-1}$. Vibration of –OH as silanol group or water was indicated by broad absorbance at $3400\text{-}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO₂ 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 $5 \cdot 10^{-4}$ M, 10^{-3} M dan 10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang $950\text{-}1050\text{ cm}^{-1}$, $620\text{-}690\text{ cm}^{-1}$, $420\text{-}460\text{ cm}^{-1}$. Vibrasi double ring pada $520\text{-}570\text{ cm}^{-1}$, pore opening $300\text{-}370\text{ cm}^{-1}$. Vibrasi gugus –OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada $3400\text{-}3450\text{ cm}^{-1}$. Data XRD menunjukkan bahwa produk sintesis mempunyai kristalinitas tinggi. Pada rasio Silikat/Aluminat 1 menghasilkan zeolite tipe sodalit sedangkan rasio 15 dan 25 menghasilkan NaP1 dan SiO₂ 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 1 dan 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 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 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 applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources 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 continuously. 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 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 linear 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°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, $\text{Al}(\text{OH})_3$ 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 specific duration until gel formed, the process was continued by hydrothermal treatment in teflon container at 100°C during 7 days, 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	$5 \cdot 10^{-4}$ (1/2 cmc)
A2	1	1	$1 \cdot 10^{-3}$ (cmc)
A3	1	1	$1 \cdot 10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5 \cdot 10^{-4}$ (1/2 cmc)
B2	15	1	$1 \cdot 10^{-3}$ (cmc)
B3	15	1	$1 \cdot 10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5 \cdot 10^{-4}$ (1/2 cmc)
C2	25	1	$1 \cdot 10^{-3}$ (cmc)
C3	25	1	$1 \cdot 10^{-2}$ (10 cmc)

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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. Cetyltrimethylammonium 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/ $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ 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.

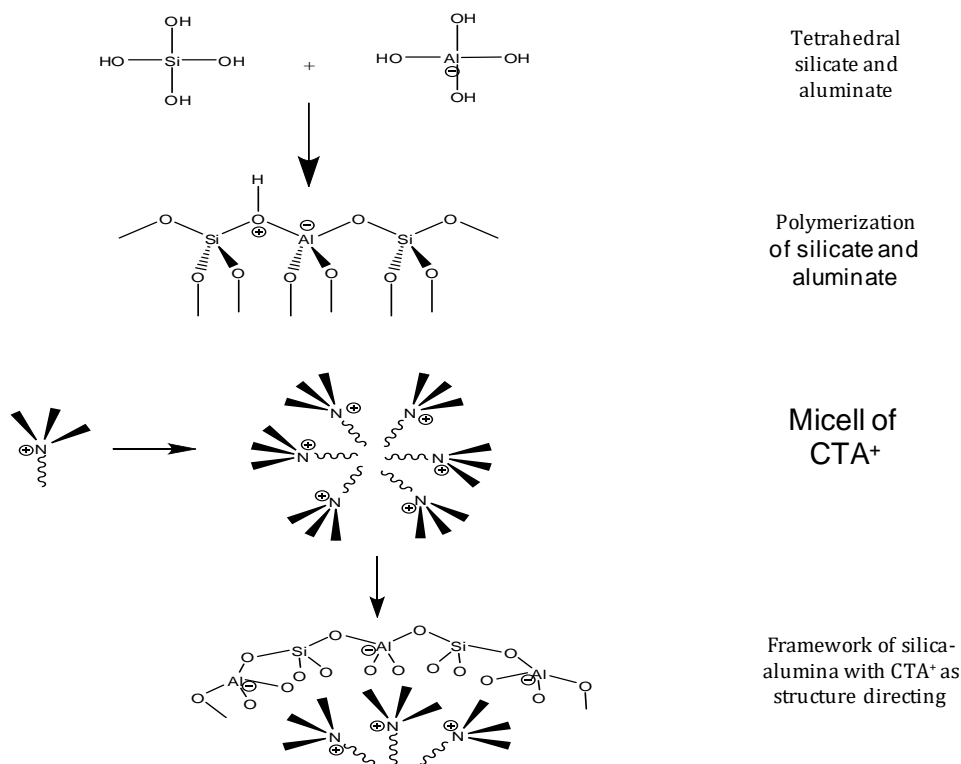


Figure 1. The mechanism of zeolite framework formation by CTA⁺ as structure directing (the letters in figure are too small, unreadable)

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 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.

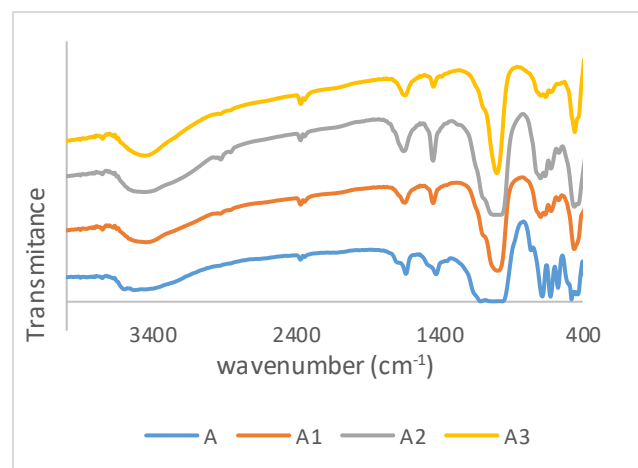


Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4} M (A2) with CTAB 1.10^{-3} M (A3) with CTAB 1.10^{-2} M
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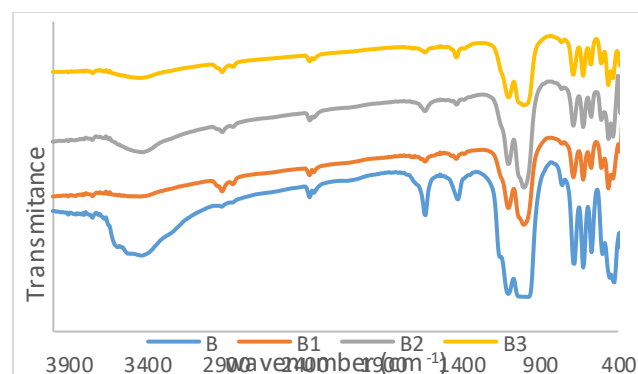


Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4} M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M

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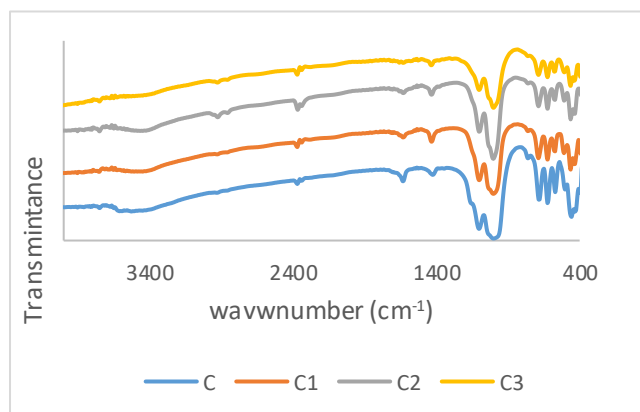


Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB $5 \cdot 10^{-4}$ M (C2) with CTAB $1 \cdot 10^{-3}$ M (C3) with CTAB $1 \cdot 10^{-2}$ M. Should the curves be color? The lines and scale are not clear/contract. a.u. should be used for transperence 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 ($\text{T} = \text{Si}$ or Al) and appears at $950\text{-}1250\text{ cm}^{-1}$ wavenumber, whereas the symetric stretching vibration at $650\text{-}720\text{ cm}^{-1}$ and 500 cm^{-1} . Furthermore, the wavenumber at $1050\text{-}1150\text{ cm}^{-1}$ was asymmetric stretching vibration, $750\text{-}820\text{ cm}^{-1}$ 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 $300\text{-}420\text{ cm}^{-1}$ is pore opening and $500\text{-}650\text{ cm}^{-1}$ is doublé ring [21]. Doublé ring is specific character of zeolite type, because it is an external linkage 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 $950\text{-}1250\text{ cm}^{-1}$ and $3400\text{-}3500\text{ cm}^{-1}$. The lower absorption intensity of wave number at $3400\text{-}3500\text{ cm}^{-1}$ in the CTAB modified zeolite showed the low levels of silanol groups in the sample, where this corresponds to the formation of framework of 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 $950\text{-}1250\text{ cm}^{-1}$ after the addition of CTAB.

In the FTIR spectra of zeolite modified by CTAB surfactant, no new absorption was observed. The spectra 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.

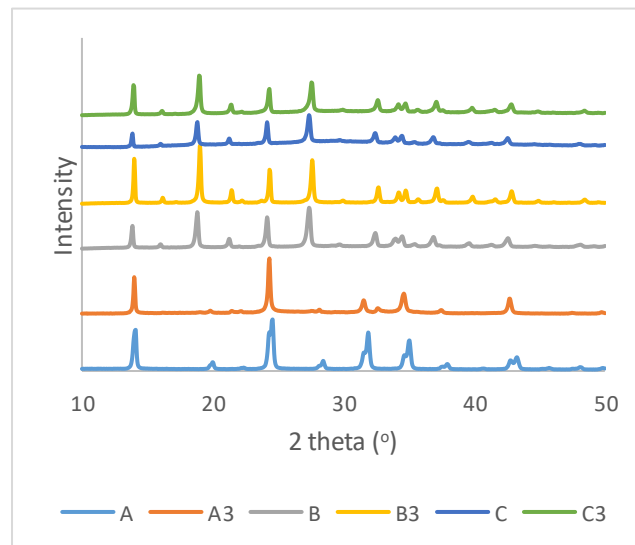


Figure 3. The pattern of diffractograms synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1.10^{-2} M

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

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. 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.

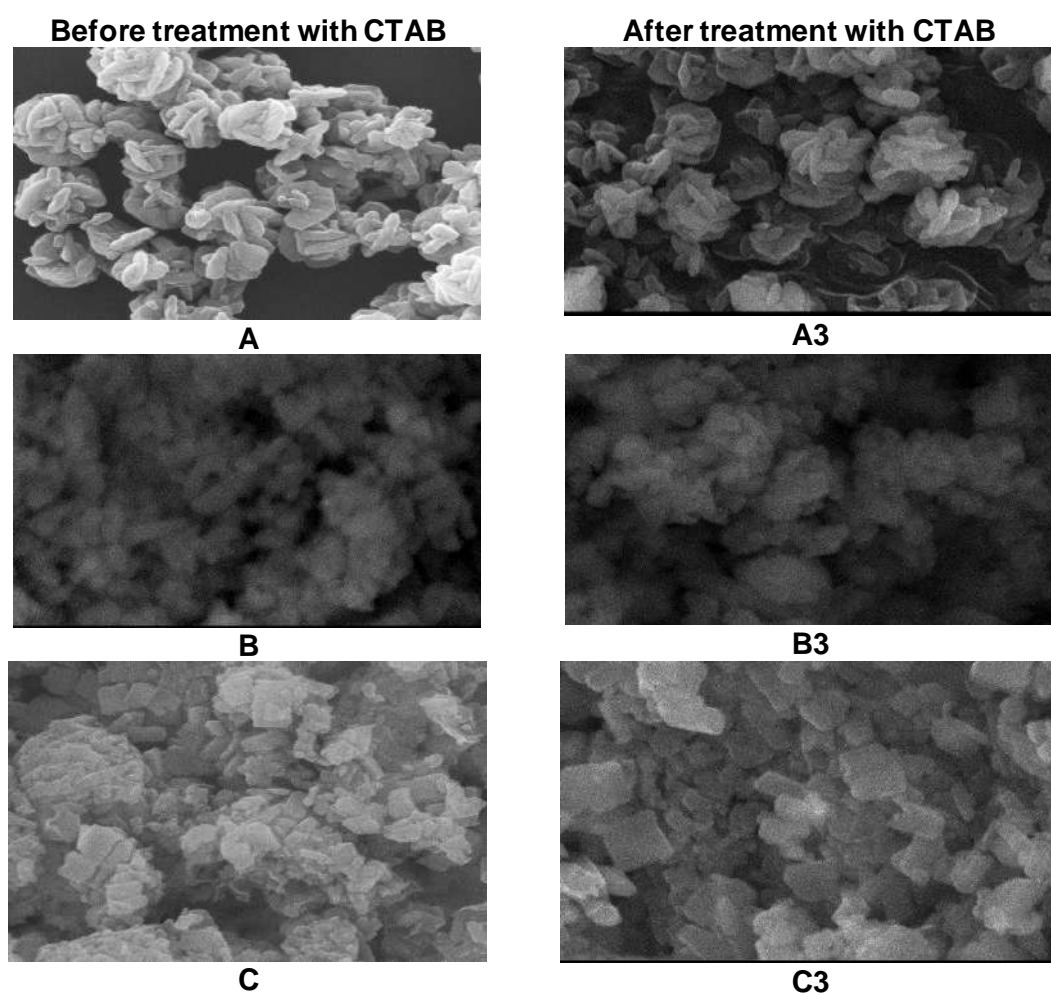


Figure 4. The SEM image of synthesized zeolite

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 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 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 m²/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 cm³/g and average pore radius was determined as 2.261 μ m. It means the NaP1 of this research have a micropore structure.

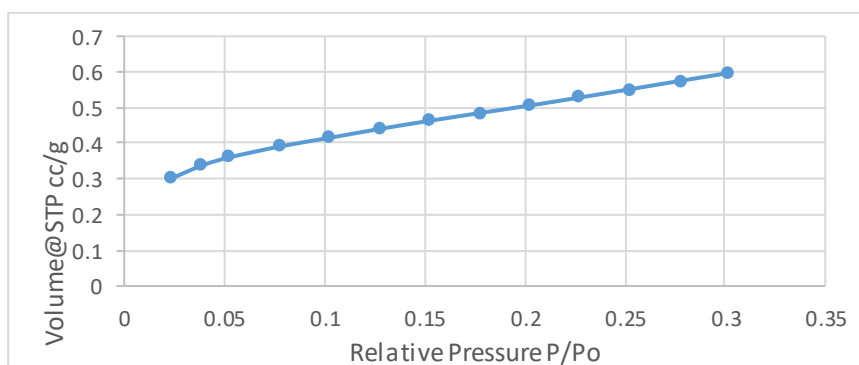


Figure 4. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

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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 which is due to the decomposition of the CTAB surfactant residues on the product zeolite surface.

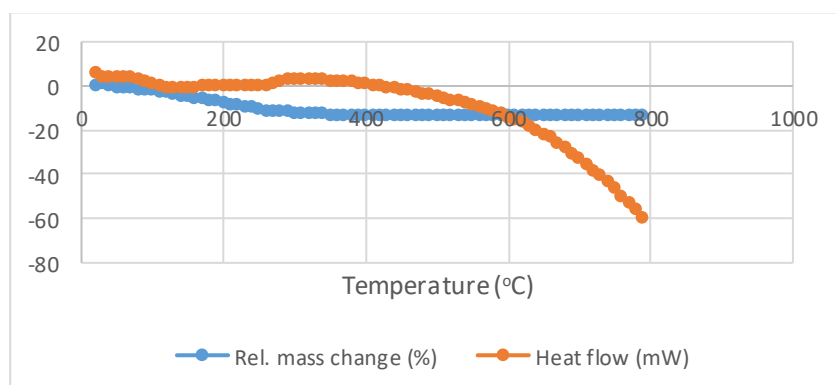


Figure 5. Thermogram of the synthesized product ratio Si/Al (v/v) 1

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 research 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

REFERENCES 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

IMPROVEMENT OF ARTICLE

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

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.

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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 l in Indonesian Journal of Chemistry (IJC). Once again than

Best regards

Sriatun

Laboratory of Inorganic Chemistry,
Dept of Chemistry FSM Undip

--

Terima Kasih

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Laboratorium Kimia Anorganik
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2 Attachments

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Nuryono Nuryono <nuryono_mipa@ugm.ac.id>
to me

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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comment reviewer

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 $5 \cdot 10^{-4}$ M, 10^{-3} M and 10^{-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 SiO₂ 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 $5 \cdot 10^{-4}$ 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 SiO₂ 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 1 dan 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 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 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 applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources 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 continuously. 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 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 linear 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°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, $\text{Al}(\text{OH})_3$ 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 specific duration until gel formed, the process was continued by hydrothermal treatment in teflon container at 100°C during 7 days, 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	$5 \cdot 10^{-4}$ (1/2 cmc)
A2	1	1	$1 \cdot 10^{-3}$ (cmc)
A3	1	1	$1 \cdot 10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5 \cdot 10^{-4}$ (1/2 cmc)
B2	15	1	$1 \cdot 10^{-3}$ (cmc)
B3	15	1	$1 \cdot 10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5 \cdot 10^{-4}$ (1/2 cmc)
C2	25	1	$1 \cdot 10^{-3}$ (cmc)
C3	25	1	$1 \cdot 10^{-2}$ (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. Cetyltrimethylammonium 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/ $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ 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.

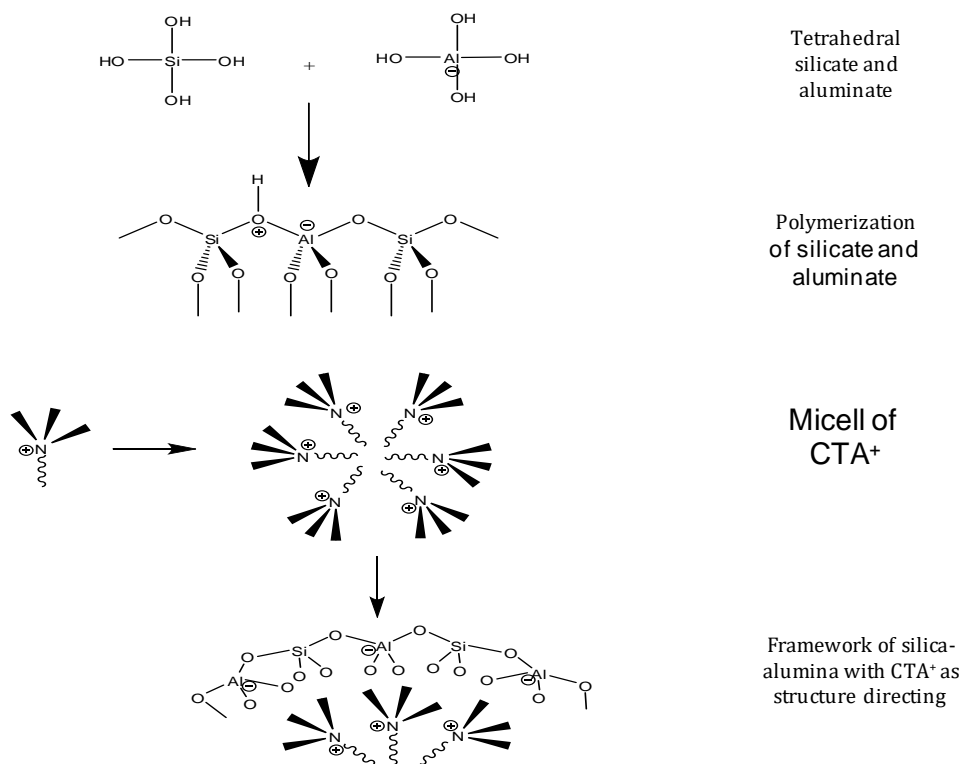


Figure 1. The mechanism of zeolite framework formation by CTA⁺ as structure directing (the letters in figure are too small, unreadable)

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 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.

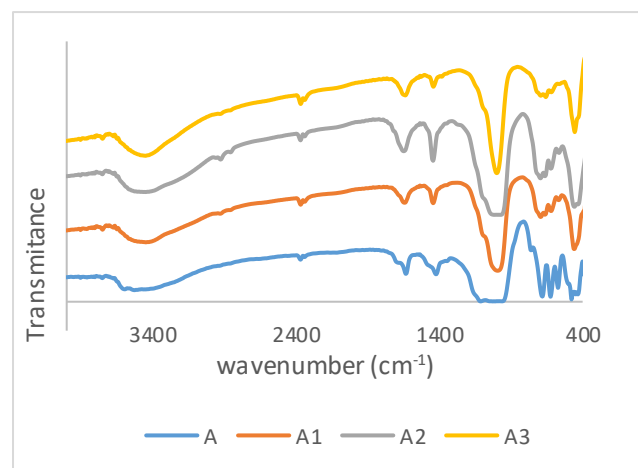


Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4} M (A2) with CTAB 1.10^{-3} M (A3) with CTAB 1.10^{-2} M
Should the curves be color? The lines and scale are not clear/contract

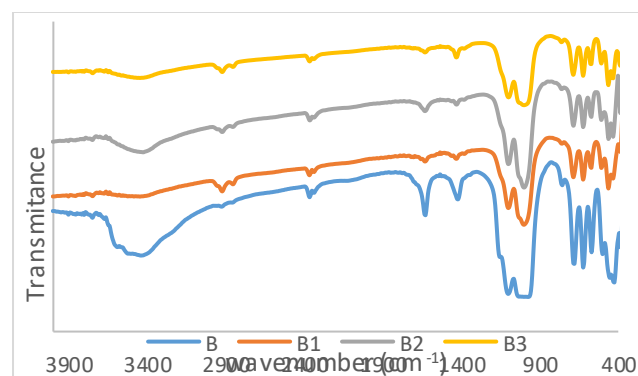


Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4} M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M

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

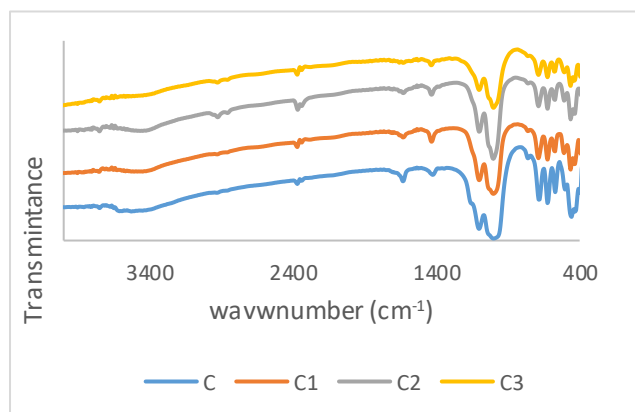


Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5.10^{-4} M (C2) with CTAB 1.10^{-3} M (C3) with CTAB 1.10^{-2} M. Should the curves be color? The lines and scale are not clear/contract. a.u. should be used for transperence unit.

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 ($\text{T} = \text{Si}$ or Al) and appears at $950\text{-}1250\text{ cm}^{-1}$ wavenumber, whereas the symetric stretching vibration at $650\text{-}720\text{ cm}^{-1}$ and 500 cm^{-1} . Furthermore, the wavenumber at $1050\text{-}1150\text{ cm}^{-1}$ was asymmetric stretching vibration, $750\text{-}820\text{ cm}^{-1}$ 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 $300\text{-}420\text{ cm}^{-1}$ is pore opening and $500\text{-}650\text{ cm}^{-1}$ is doublé ring [21]. Doublé ring is specific character of zeolite type, because it is an external linkage 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 $950\text{-}1250\text{ cm}^{-1}$ and $3400\text{-}3500\text{ cm}^{-1}$. The lower absorption intensity of wave number at $3400\text{-}3500\text{ cm}^{-1}$ in the CTAB modified zeolite showed the low levels of silanol groups in the sample, where this corresponds to the formation of framework of 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 $950\text{-}1250\text{ cm}^{-1}$ after the addition of CTAB.

In the FTIR spectra of zeolite modified by CTAB surfactant, no new absorption was observed. The spectra 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.

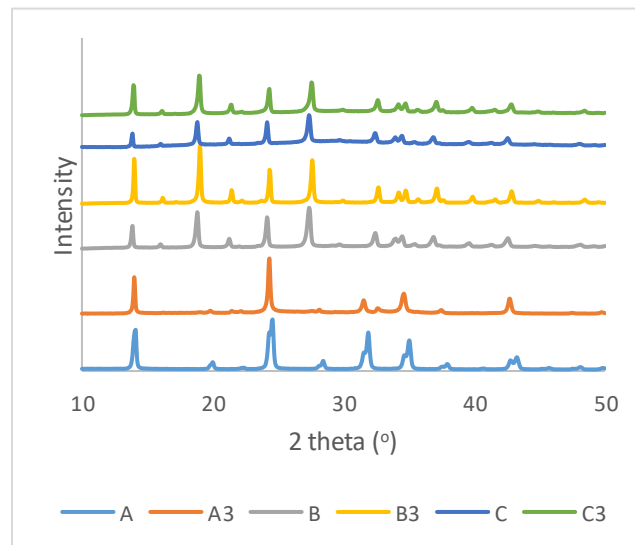


Figure 3. The pattern of diffractograms synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB $1.10^{-2}M$ (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB $1.10^{-2}M$ (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB $1.10^{-2}M$

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

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. 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.

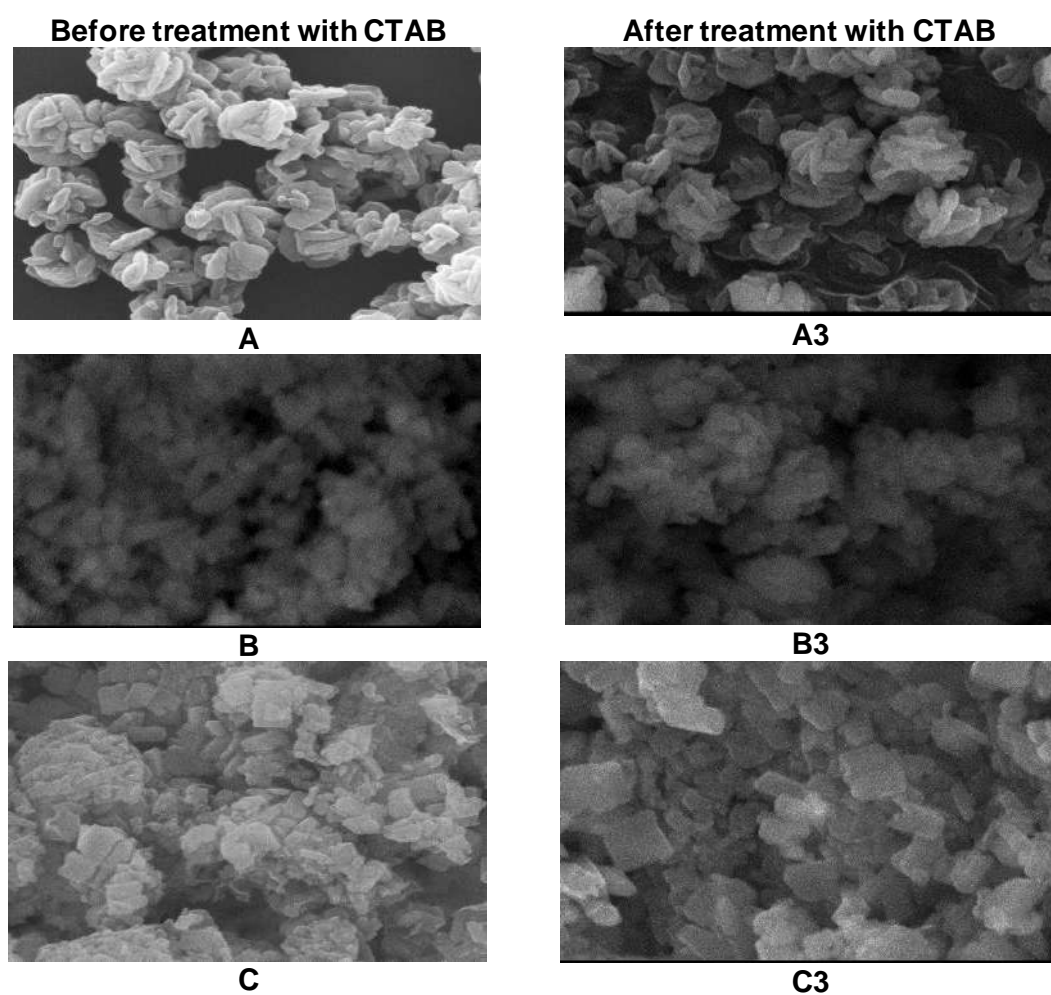


Figure 4. The SEM image of synthesized zeolite

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 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 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 m²/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 cm³/g and average pore radius was determined as 2.261 Å. It means the NaP1 of this research have a micropore structure.

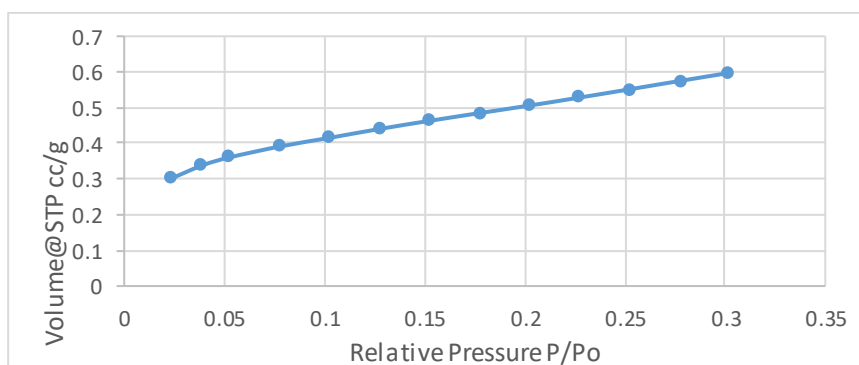


Figure 4. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

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 which is due to the decomposition of the CTAB surfactant residues on the product zeolite surface.

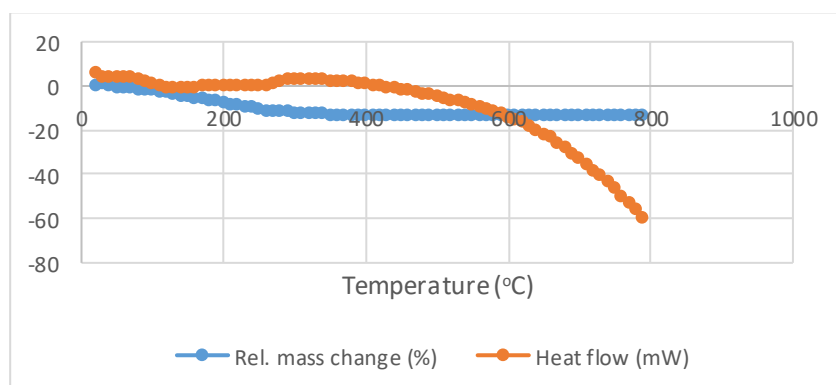


Figure 5. Thermogram of the synthesized product ratio Si/Al (v/v) 1

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 research 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

REFERENCES 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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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 K α radiation ($\lambda = 1.54184 \text{ \AA}$) at generator voltage 40 kV and current 40 mA. The simultaneous scanning electron microscope (SEM) and Energy-Dispersive X-ray (EDX) were performed using JEOL JSM 6510/LV/A/LA. The BET surface areas, pore volume and average pore size of samples were determined from N ₂ adsorption isotherms at liquid nitrogen temperature (-195,7 °C) using a Quantachrome NovaWin, Quantachrome Instrument version 10.01. The thermal gravimetric analysis (TGA) analysis of samples were determined using LINSEIS STA PlatinumSeries, Platinum evaluation 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)

	<div>Part: Procedure</div> <div>Table 1. The composition of precursor and surfactant</div> <table><tr><th rowspan="2">Sample Code</th><th colspan="2">Ratio of Precursor (v/v)</th><th rowspan="2">Surfactant CTAB (M)</th></tr><tr><th>Sodium silicate</th><th>Sodium aluminate</th></tr><tr><td>A</td><td>1</td><td>1</td><td>0</td></tr><tr><td>A1</td><td>1</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>A2</td><td>1</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>A3</td><td>1</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr><tr><td>B</td><td>15</td><td>1</td><td>0</td></tr><tr><td>B1</td><td>15</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>B2</td><td>15</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>B3</td><td>15</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr><tr><td>C</td><td>25</td><td>1</td><td>0</td></tr><tr><td>C1</td><td>25</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>C2</td><td>25</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>C3</td><td>25</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr></table> <div>See in article vol .17 how tables are formatted</div>	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)	<div>The Table 1 has been corrected according to the format of the article Vol. 17 of IJC</div> <div>Table 1. The composition of precursor and surfactant</div> <table><tr><th rowspan="2">Sample Code</th><th colspan="2">Ratio of Precursor (v/v)</th><th rowspan="2">Surfactant CTAB (M)</th></tr><tr><th>Sodium silicate</th><th>Sodium aluminate</th></tr><tr><td>A</td><td>1</td><td>1</td><td>0</td></tr><tr><td>A1</td><td>1</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>A2</td><td>1</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>A3</td><td>1</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr><tr><td>B</td><td>15</td><td>1</td><td>0</td></tr><tr><td>B1</td><td>15</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>B2</td><td>15</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>B3</td><td>15</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr><tr><td>C</td><td>25</td><td>1</td><td>0</td></tr><tr><td>C1</td><td>25</td><td>1</td><td>5.10⁻⁴ (1/2 cmc)</td></tr><tr><td>C2</td><td>25</td><td>1</td><td>1.10⁻³ (cmc)</td></tr><tr><td>C3</td><td>25</td><td>1</td><td>1.10⁻² (10 cmc)</td></tr></table>	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)
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	<div>Part: Result and discussion</div> <div>has been</div>	<div>The word has been omitted and replaced with : was</div>																																																																																																												
	<div>Figure 1. The mechanism of zeolite framework formation by CTA⁺ as structure directing (the letters in figure are too small, unreadable)</div>	<div>The letters in Figure 1 has been enlarged</div>																																																																																																												
	<div>Finally, the CTAB surfactant as structure directing was disappeared from the zeolite structure by calcination at 500°C</div>	<div>This section has been described according to the study results obtained</div>																																																																																																												

	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 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: REFERENCES 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, <i>International Journal for Service Learning in Engineering</i> , 5(2), 60-

		<p>66.</p> <p>- Unpublished documents have been omitted and replaced by journals.</p> <p>[4] Aida, N. and Dewanti, L., (2010), <i>Pembuatan Silika Gel Dari Abu Ampas Tebu Dengan Proses Ekstraksi Basa (NaOH) dan Sol Gel</i>, D3 Teknik Kimia Institut Teknologi Surabaya, Surabaya.</p> <p>[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, <i>Prosiding Seminar Nasional Kimia dan Pendidikan Kimia 2013</i></p> <p>[20] Yusri, S., (2012), <i>Synthesis and Characterization of Mesoporous ZSM-5 Zeolite Using Secondary template and Preliminary Study of Methane Oxidation Catalysis</i>, Skripsi, Kimia FMIPA UI.</p> <p>Substituted by:</p> <p>[4] Norsurayaa, S., Fazlenaa, H., Norhasyimi R., 2016, Sugarcane Bagasse as a Renewable Source of Silica to Synthesize Santa Barbara Amorphous-15 (SBA-15), <i>Procedia Engineering</i>, 148, 839 – 846</p> <p>[18] Hasan, F., Singh, R., Li, G., Zhao, D., Webley, P.A., 2012, Direct synthesis of hierarchical LTA zeolite <i>via</i> a low crystallization and growth rate technique in presence of cetyltrimethylammonium bromide, <i>Journal of Colloid and Interface Science</i>, 382,1–12</p> <p>[20] Wang, L., Yang, W., Xin, C., Ling, F., Sun, W., Fang, X., Yang, R., 2012, Synthesis of nano-zeolite IM-5 by</p>
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		hydrothermal method with the aid of PEG and CTAB, <i>Materials Letters</i> , 69,16–19

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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 $5 \cdot 10^{-4}$ M, 10^{-3} M and 10^{-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 SiO₂ 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 $5 \cdot 10^{-4}$ 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 SiO₂ 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 1 dan 25.

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 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 applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources 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 continuously. 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 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 linear 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°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, $\text{Al}(\text{OH})_3$ 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 $\text{K}\alpha$ radiaton ($\lambda = 1.54184 \text{ \AA}$) at generator voltage 40 kV and current 40 mA. The simultaneous scanning electron microscope (SEM) and Energy-Dispersive X-ray (EDX) were performed using JEOL JSM 6510/LV/A/LA. The BET surface areas, pore volume and average pore size of samples were determined from N_2 adsorption isotherms at liquid nitrogen temperature ($-195,7^\circ\text{C}$) 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	$5 \cdot 10^{-4}$ (1/2 cmc)
A2	1	1	$1 \cdot 10^{-3}$ (cmc)
A3	1	1	$1 \cdot 10^{-2}$ (10 cmc)
B	15	1	0
B1	15	1	$5 \cdot 10^{-4}$ (1/2 cmc)
B2	15	1	$1 \cdot 10^{-3}$ (cmc)
B3	15	1	$1 \cdot 10^{-2}$ (10 cmc)
C	25	1	0
C1	25	1	$5 \cdot 10^{-4}$ (1/2 cmc)
C2	25	1	$1 \cdot 10^{-3}$ (cmc)
C3	25	1	$1 \cdot 10^{-2}$ (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 SiO₂, Al₂O₃, MgO, CaO, Fe₂O₃ 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/[SiO₄]⁴⁻ and aluminate/[AlO₄]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.

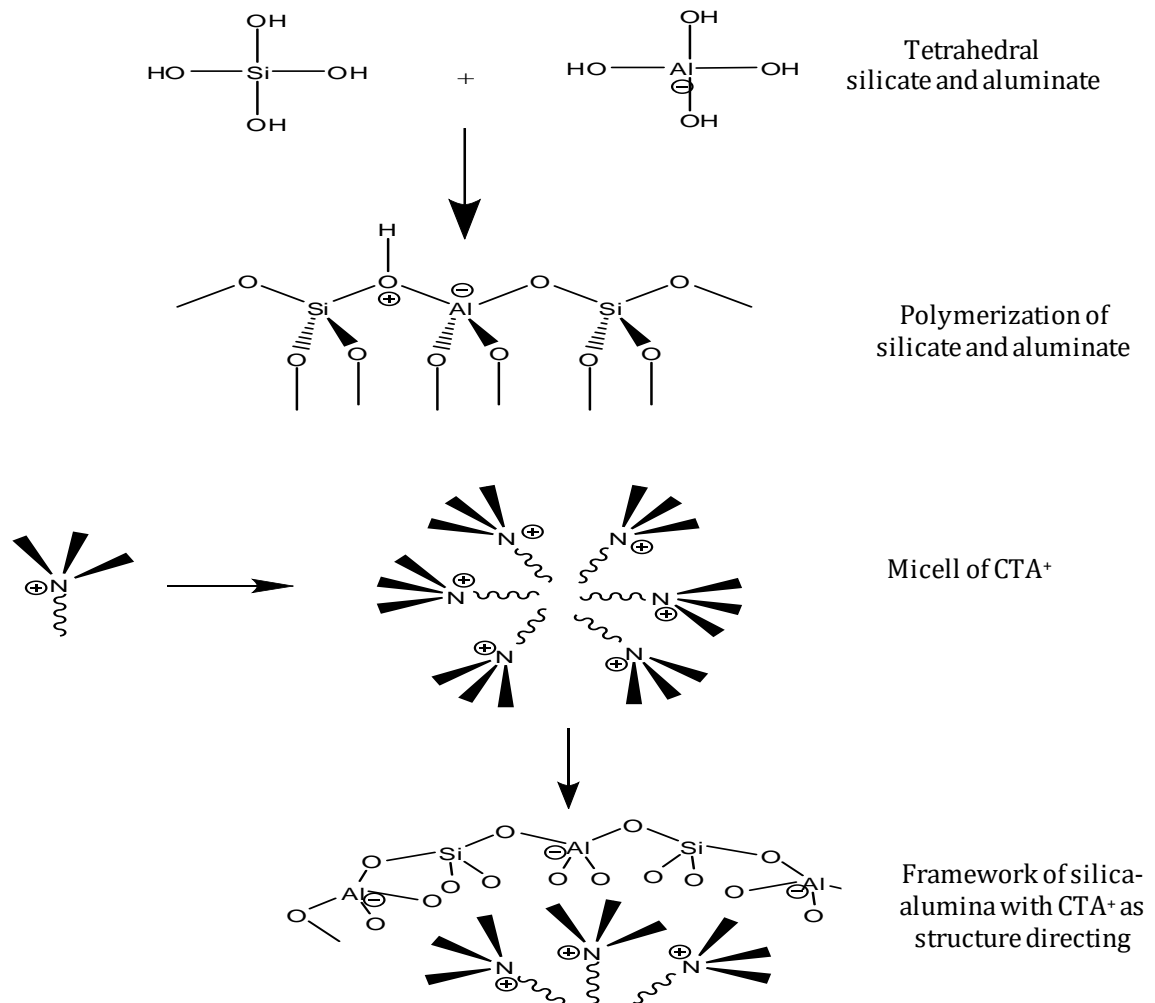


Figure 1. The mechanism of zeolite framework formation by CTA⁺ as structure directing

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.

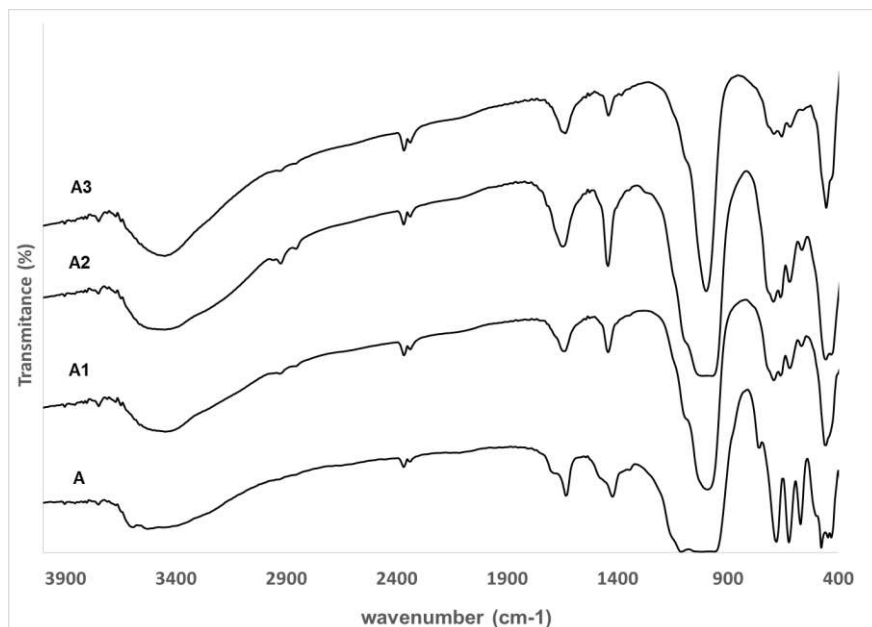


Figure 2a. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5.10^{-4}M (A2) with CTAB 1.10^{-3}M (A3) with CTAB 1.10^{-2}M (A4). (scale is too small)

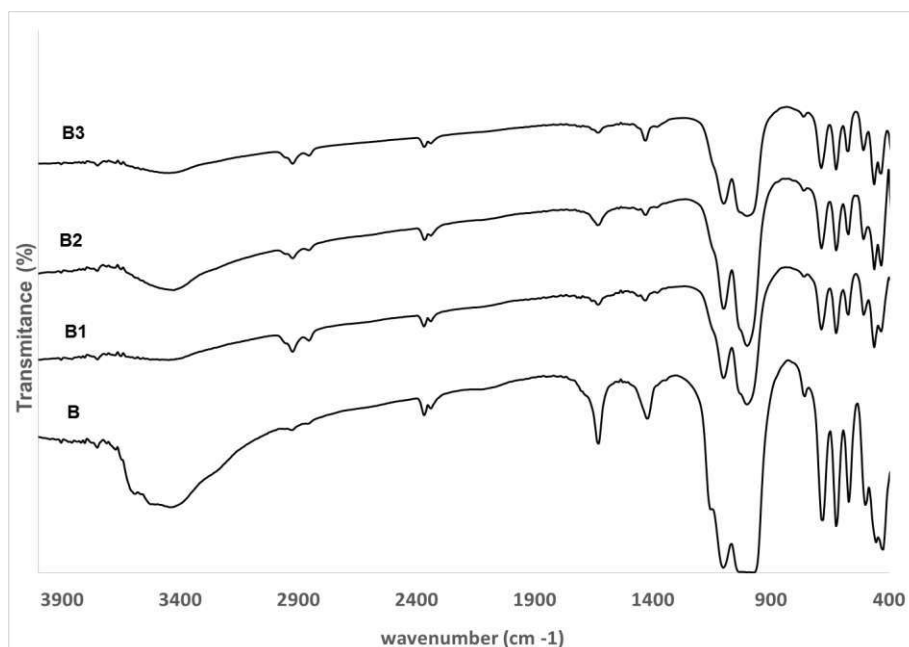


Figure 2b. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5.10^{-4} M (B2) with CTAB 1.10^{-3} M (B3) with CTAB 1.10^{-2} M (scale is too small)

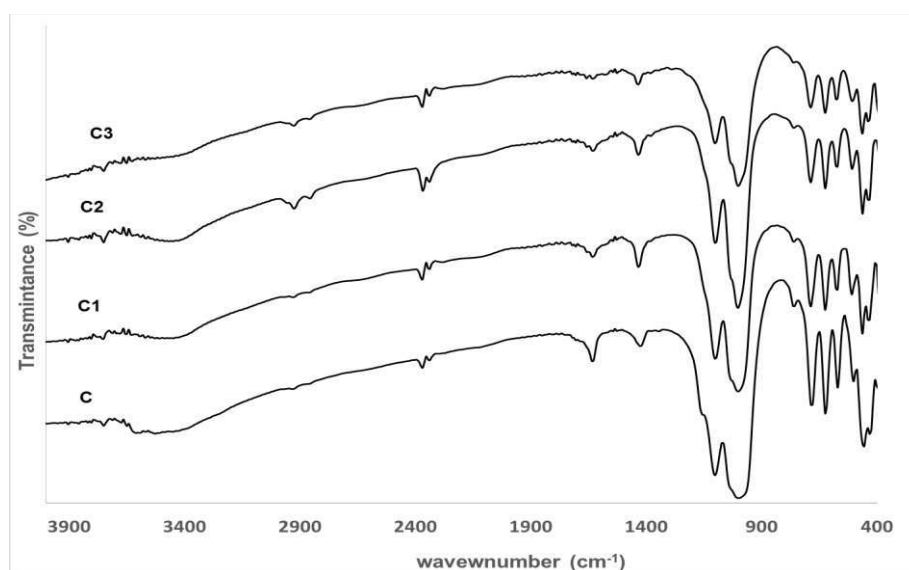


Figure 2c. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5.10^{-4} M (C2) with CTAB 1.10^{-3} M (C3) with CTAB 1.10^{-2} M (scale is too small)

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 ($\text{T} = \text{Si}$ or Al) and appears at $950\text{--}1250\text{ cm}^{-1}$ wavenumber, whereas the symmetric stretching vibration at $650\text{--}720\text{ cm}^{-1}$ and 500 cm^{-1} . Furthermore, the wavenumber at $1050\text{--}1150\text{ cm}^{-1}$ was asymmetric stretching vibration, $750\text{--}820\text{ cm}^{-1}$ 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 $300\text{--}420\text{ cm}^{-1}$ is pore opening and $500\text{--}650\text{ cm}^{-1}$ is double ring [21]. Double ring is specific character of zeolite type, because it is an external linkage 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 $950\text{--}1250\text{ cm}^{-1}$ and $3400\text{--}3500\text{ cm}^{-1}$. The lower absorption intensity of wave number at $3400\text{--}3500\text{ cm}^{-1}$ in the CTAB modified zeolite showed the low levels of silanol groups in the sample, where this corresponds to the formation of framework of 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.

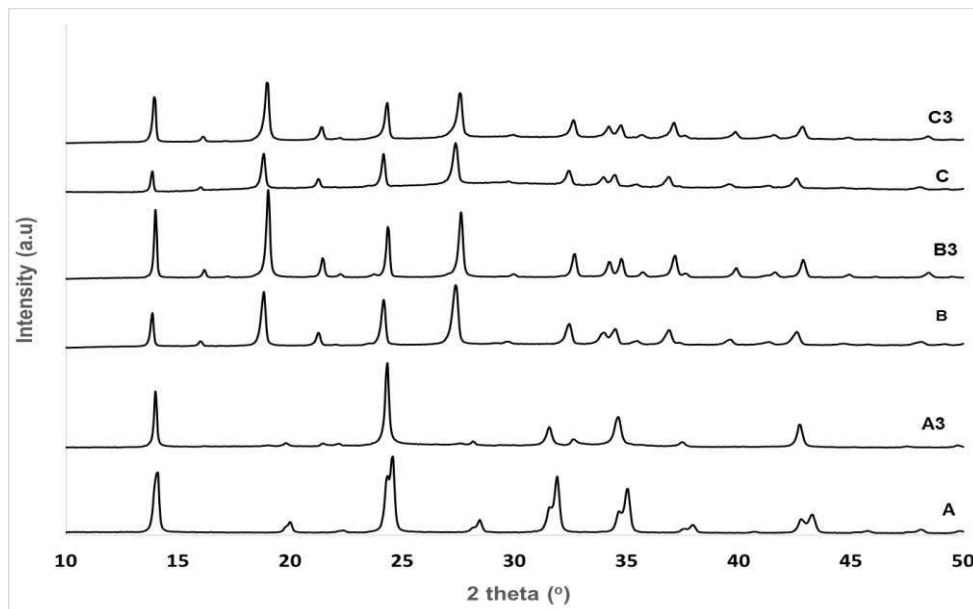


Figure 3. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2}M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2}M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1.10^{-2}M

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. 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.

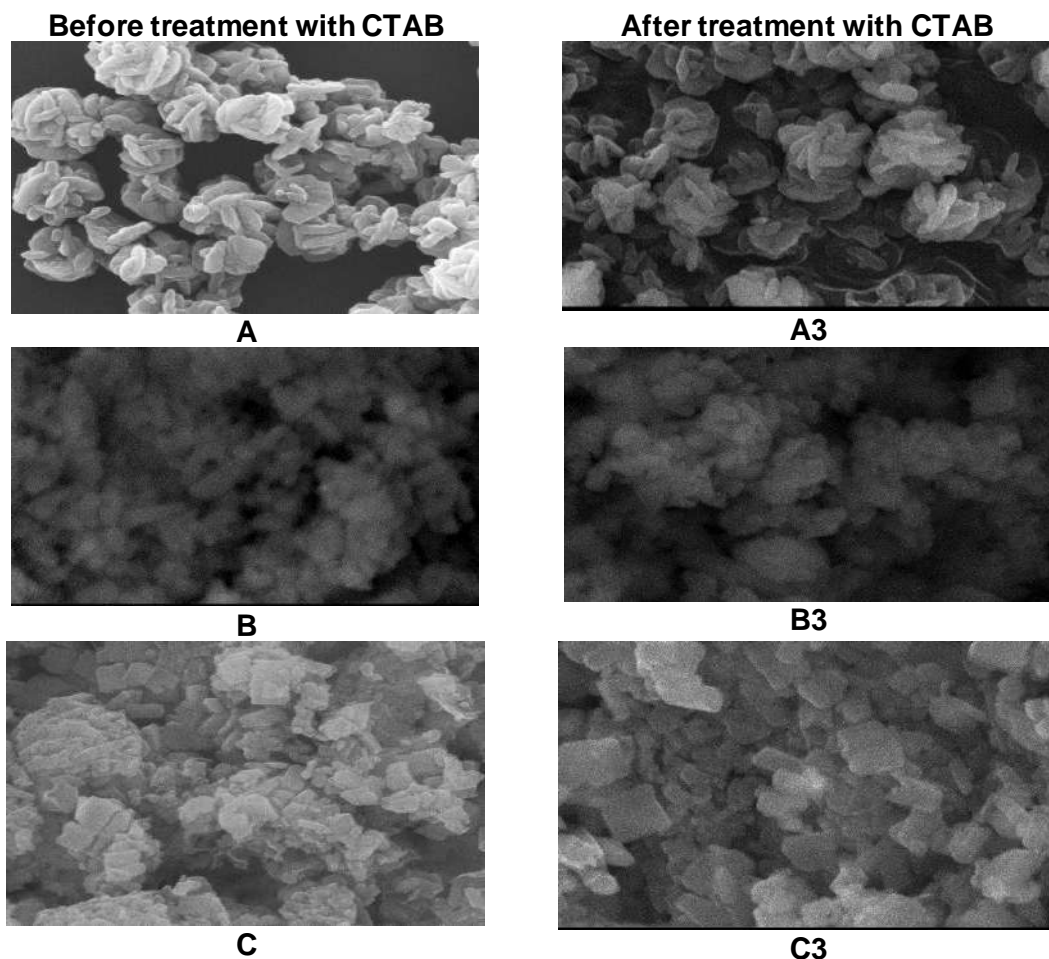


Figure 4. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1.10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1.10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1.10^{-2} M

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 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 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 CTAB $1.10^{-2}M$

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 $13.508 \text{ m}^2/\text{g}$. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be $5.28 \times 10^{-3} \text{ cm}^3/\text{g}$ and average pore radius was determined as 2.261°A . It means the NaP1 of this research have a micropore structure.

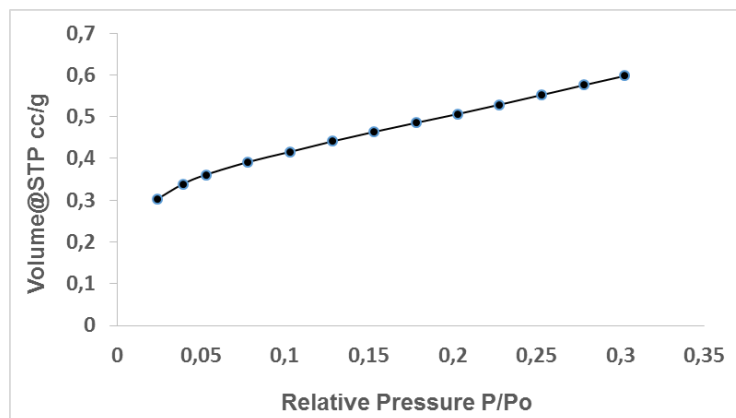


Figure 5. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1 (lines are too tin)

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°C , it means the loosely bond or free water molecules from product zeolite cage. Then the reducing weight also occurred at $270\text{--}400^\circ\text{C}$ which is due to the decomposition of the CTAB surfactant residues on the product zeolite surface.

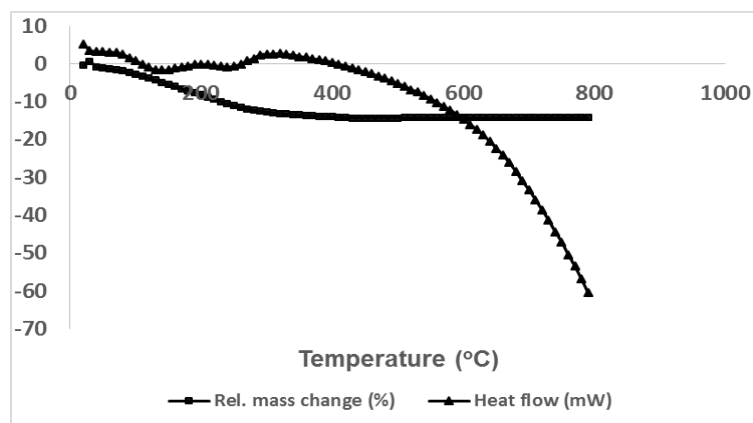


Figure 6. Thermogram of the synthesized product ratio Si/Al (v/v) 1 (there is not legend for x and y axis)

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

Sriatun, Taslimah, L. Suyati gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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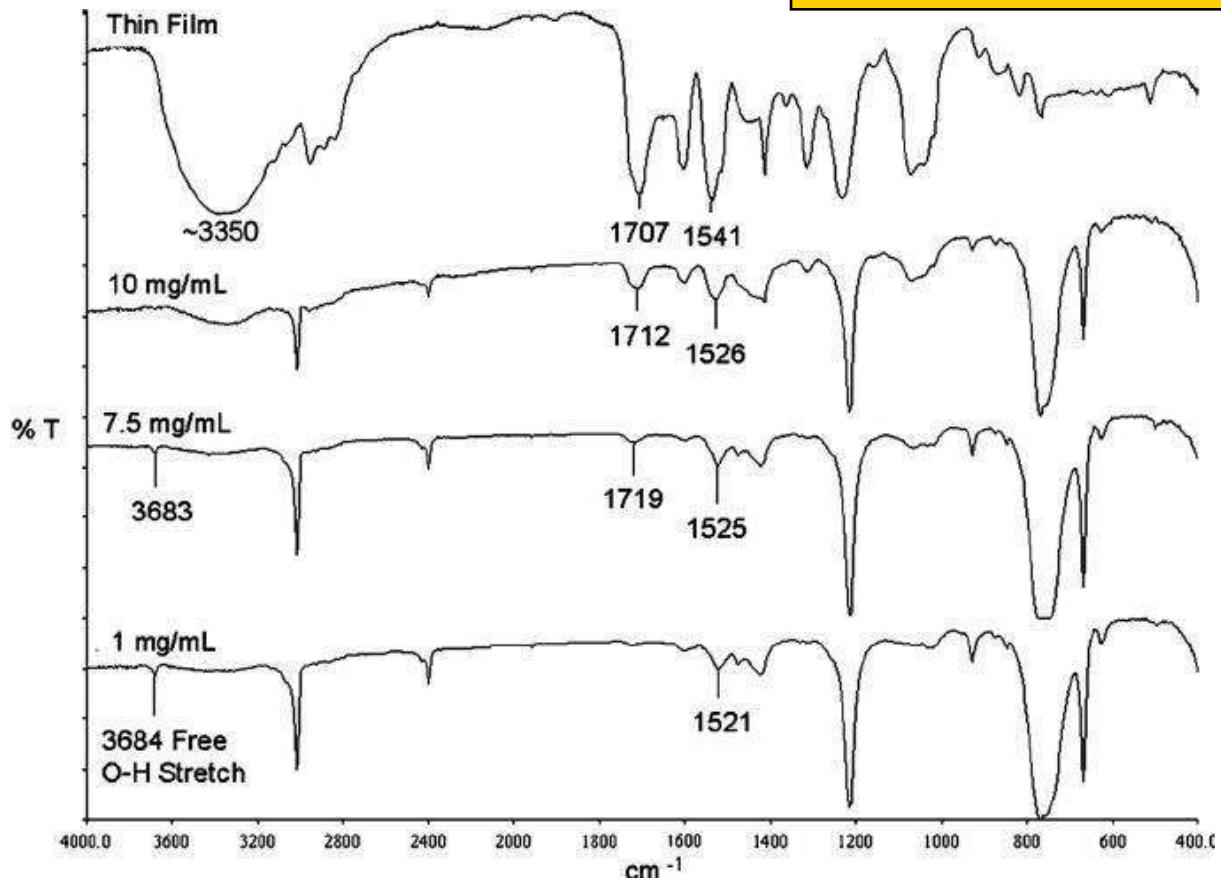
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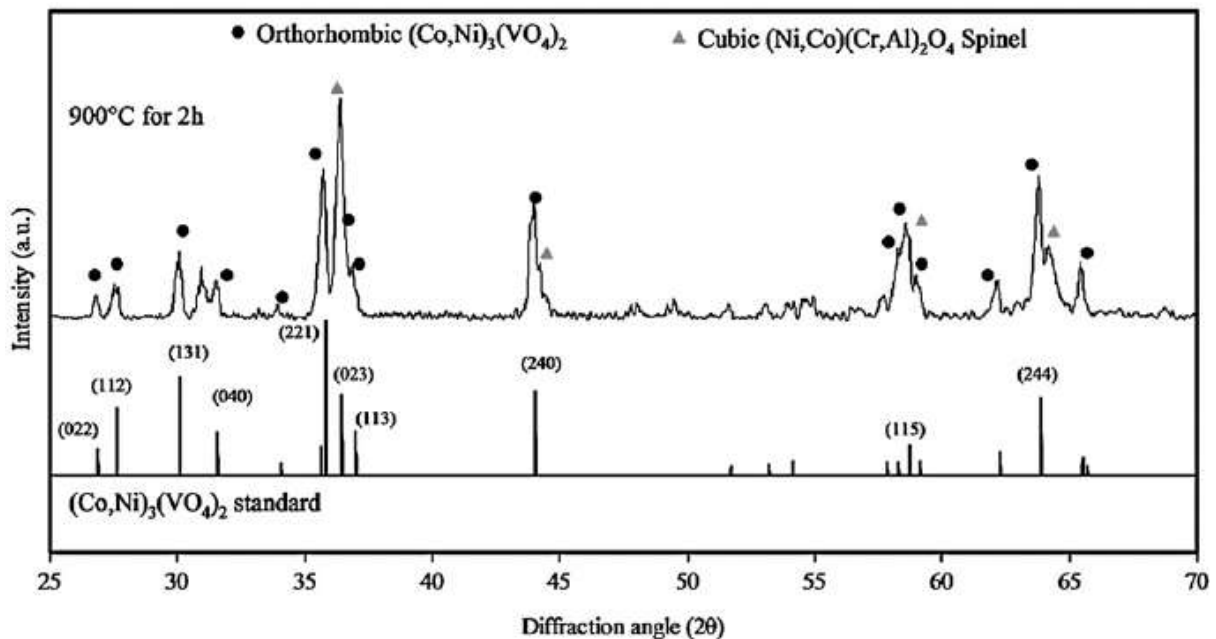
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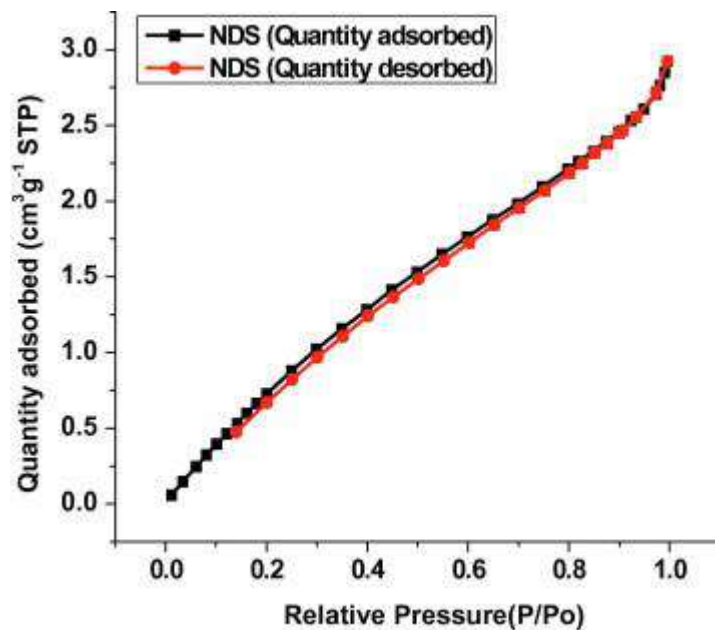
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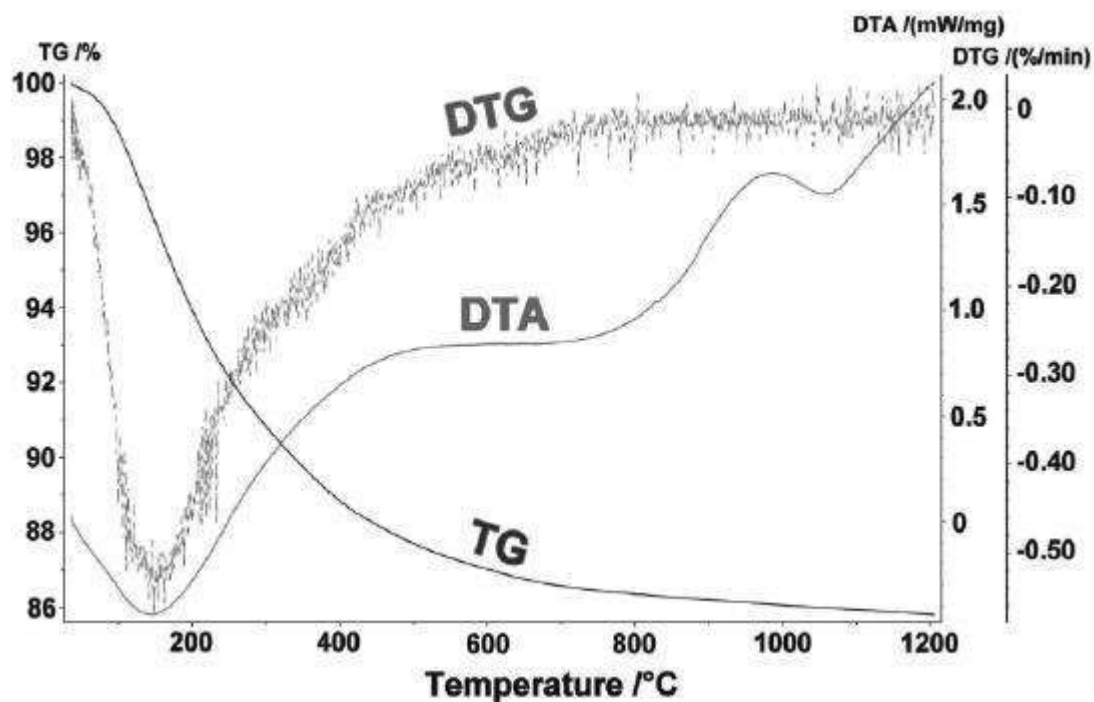
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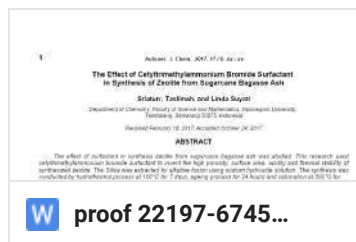
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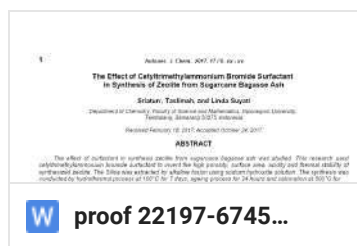
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Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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Department of Chemistry, Faculty of Science and Mathematics, Diponegoro University,
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Received February 18, 2017; Accepted October 24, 2017

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 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950\text{--}1050\text{ cm}^{-1}$ and $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$, double ring at $520\text{--}570\text{ cm}^{-1}$, pore opening at $300\text{--}370\text{ cm}^{-1}$. Vibration of --OH as silanol group or water was indicated by broad absorbance at $3400\text{--}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO_2 quartz and contain 12.23% and 12.19% of Si, 4.17% and 13.18% of Al, respectively. Observation on SEM revealed that the crystal produced using cetyltrimethylammonium bromide 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 5×10^{-4} M, 1×10^{-3} M dan 1×10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang $950\text{--}1050\text{ cm}^{-1}$, $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$. Vibrasi double ring pada $520\text{--}570\text{ cm}^{-1}$, pore opening $300\text{--}370\text{ cm}^{-1}$. Vibrasi gugus --OH yang menunjukkan keberadaan silanol dan air ditunjukkan oleh serapan pada $3400\text{--}3450\text{ cm}^{-1}$. Data XRD menunjukkan bahwa produk sintesis mempunyai kristalinitas tinggi. Pada rasio Silikat/Aluminat 1 menghasilkan zeolite tipe sodalit sedangkan rasio 15 dan 25 menghasilkan NaP1 dan SiO_2 kuarsa. Pada rasio Si/Al 1 dan 25 berturut-turut mempunyai Si sebesar 12,23% dan 12,19% serta Al sebesar 4,17% and 13,18%. Hasil SEM menunjukkan bahwa produk sintesis yang menggunakan setiltrimetilamonium bromida mempunyai bentuk yang lebih homogen dan teratur.

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

INTRODUCTION

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

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

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

There are many methods applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources 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 linear and branch molecule [17]. Directing agent also used to control the growth of crystal [18].

Based on previously studies information, there have never been conducted researches using cetyltrimethylammonium bromide (CTAB) surfactants as directing agent in synthesis of zeolite from sugarcane

bagasse ash. Therefore, in this study, we report the using of cetyltrimethylammonium bromide (CTAB) as structure directing agent in synthesis 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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The sample functional group is determined by FTIR spectrometer Shimadzu. The diffraction patterns of the samples were measured by X-ray diffractometer (XRD) Rigaku Multiplex with Cu $K\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$) at generator voltage 40 kV and current 40 mA. The simultaneous scanning electron microscope (SEM) and Energy-Dispersive X-ray (EDX) were performed using JEOL JSM 6510/LV/A/LA. The BET surface areas, pore volume and average pore size of samples were determined from N_2 adsorption isotherms at liquid nitrogen temperature (-195.7 °C) using a Quantachrome NovaWin2, Quantachrome Instrument version 2.2. The thermal gravimetric

analysis (TGA) analysis of samples was determined using LINSEIS STA Platinum Series, Platinum

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 ($\text{T} = \text{Si}$ or Al) and appears at 950–1250 cm^{-1} wavenumber, whereas the symmetric stretching vibration at 650–720 and 500 cm^{-1} . Furthermore, the wavenumber at 1050–1150 cm^{-1} was asymmetric stretching vibration, 750–820 cm^{-1} is symmetric stretching vibration in the external linkage. All 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

evaluation V1.0.138.

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	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)

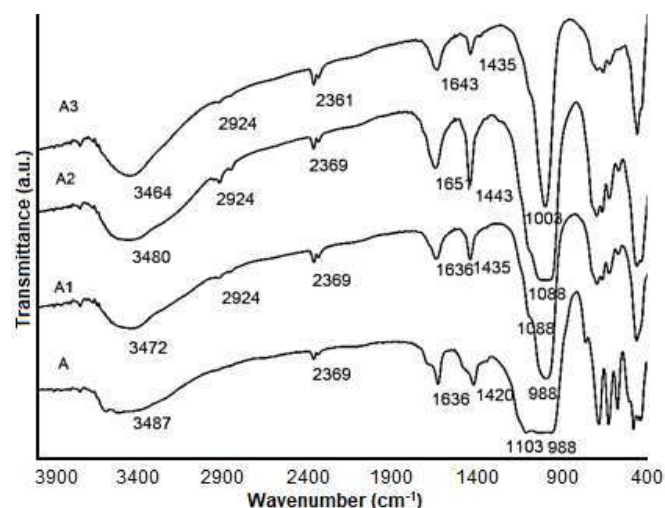


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

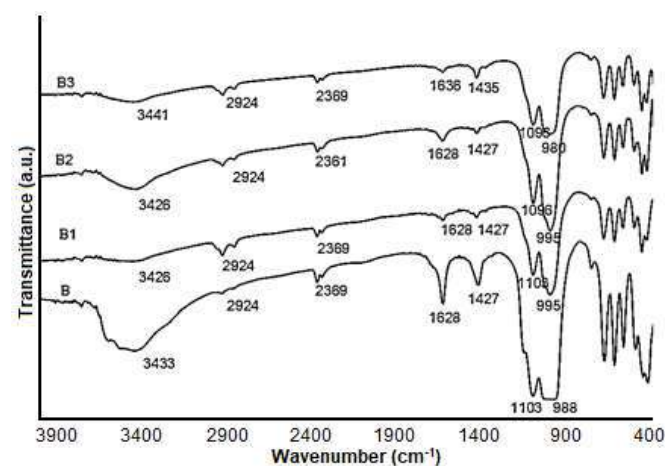


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

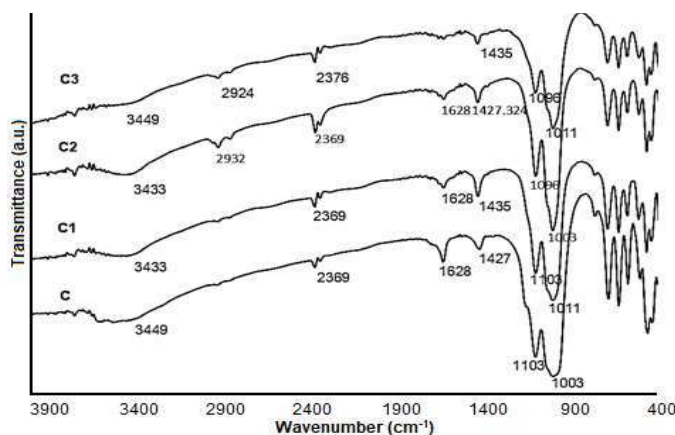


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB

When the cetyltrimethylammonium (CTAB) as cationic surfactant is used as a directing molecule in the synthesis process of zeolite, so it would be interacted electrostatically with the silicate/ $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ anions primary building units. This interaction depends on surfactant concentration. The effect of surfactant presence can be observed on spectra A1, A2 and A3 in Fig. 1, B1, B2 and B3 in Fig. 2, C1, C2 and C3 in Fig. 3. It could be clearly observed that the presence of cetyltrimethylammonium (CTAB) surfactants has significantly affected the intensity and patterns of absorption at the wavenumber 950–1250 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 homogeneously.

5x10⁻⁴ M (C2) with CTAB 1x10⁻³ M (C3) with CTAB 1x10⁻² M

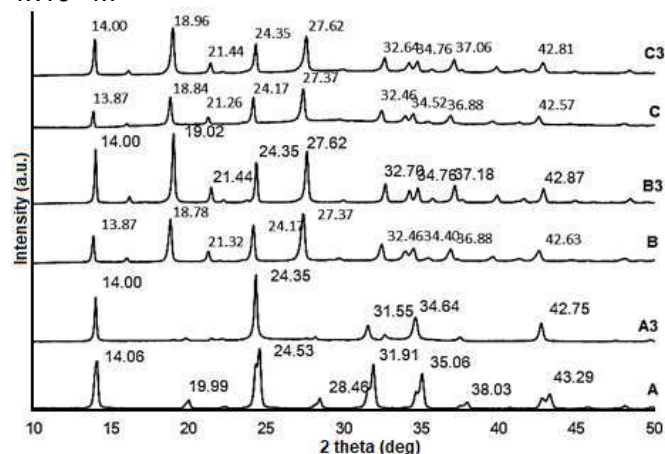


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

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

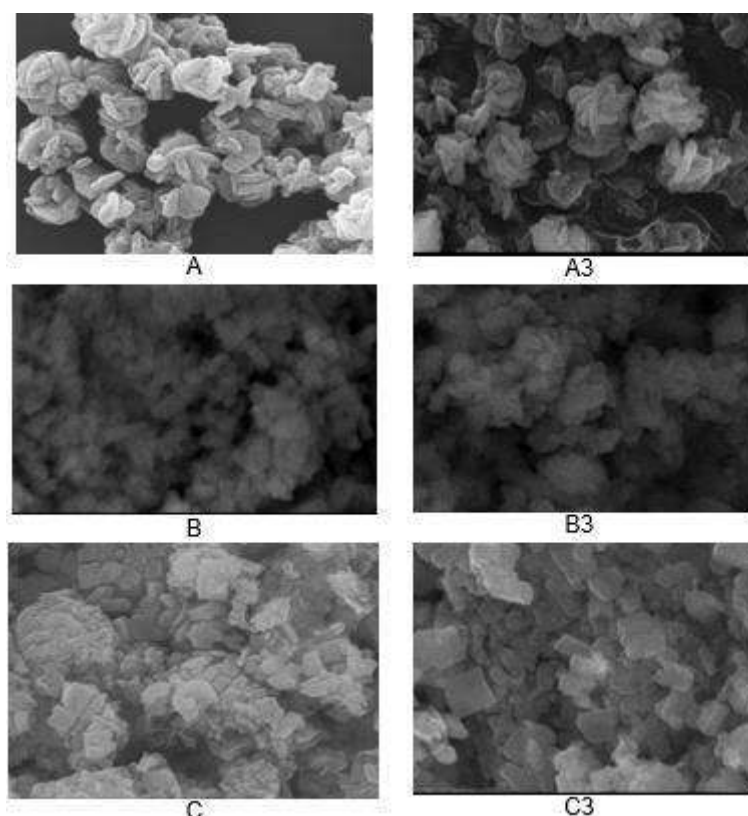


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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.

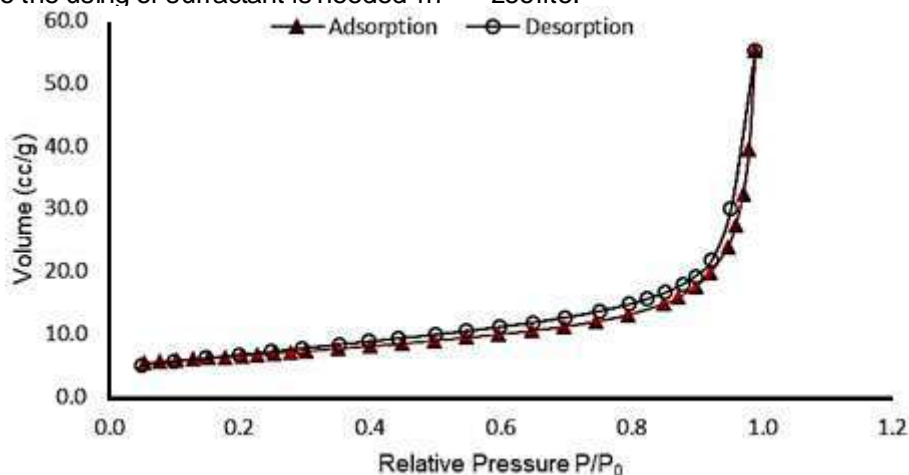


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

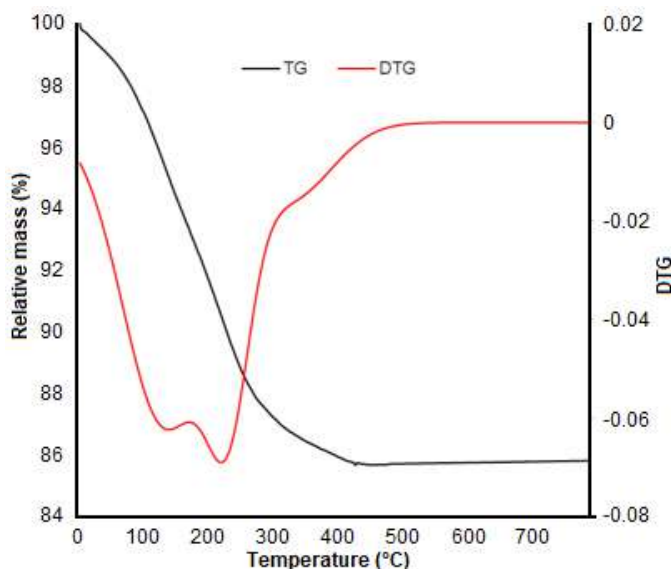


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m²/g. This reveals the existence

only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56×10^{-2} cm³/g and average pore radius was determined as 7.737 Å. It means the NaP1 of this research 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 residues on the product zeolite surface.

CONCLUSION

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

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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

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Received February 18, 2017; Accepted October 24, 2017

ABSTRACT

The purpose of this research is to synthesize zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent. This research used cetyltrimethylammonium bromide surfactant to invent the high porosity, surface area, acidity and thermal stability of synthesized zeolite. The Silica was extracted by alkaline fusion using sodium hydroxide solution. The synthesis was conducted by hydrothermal process at 100 °C for 7 days, ageing process for 24 h and calcination at 500 °C for 5 h. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at 950–1050 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 SiO₂ 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 5×10^{-4} M, 1×10^{-3} M dan 1×10^{-2} M. Hasil penelitian menunjukkan bahwa keseluruhan produk menghasilkan serapan yang kuat pada daerah bilangan gelombang 950–1050 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 SiO₂ 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% P₂O₅ and 0.38% K₂O. 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 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 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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

The sample functional group is determined by FTIR spectrometer Shimadzu. The diffraction patterns of the samples were measured by X-ray diffractometer (XRD) Rigaku Multiplex with Cu $K\alpha$ radiation ($\lambda = 1.54184 \text{ \AA}$) at generator voltage 40 kV and current 40 mA. The simultaneous scanning electron microscope (SEM) and Energy-Dispersive X-ray (EDX) were performed using JEOL JSM 6510/LV/A/LA. The BET surface areas, pore volume and average pore size of samples were determined from N_2 adsorption isotherms at liquid nitrogen temperature (-195.7 °C) using a Quantachrome NovaWin2, Quantachrome Instrument version 2.2. The thermal gravimetric

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 (TO₄). In all products, there can be observed the internal linkage, there is asymmetric stretching vibration of TO₄ (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

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)

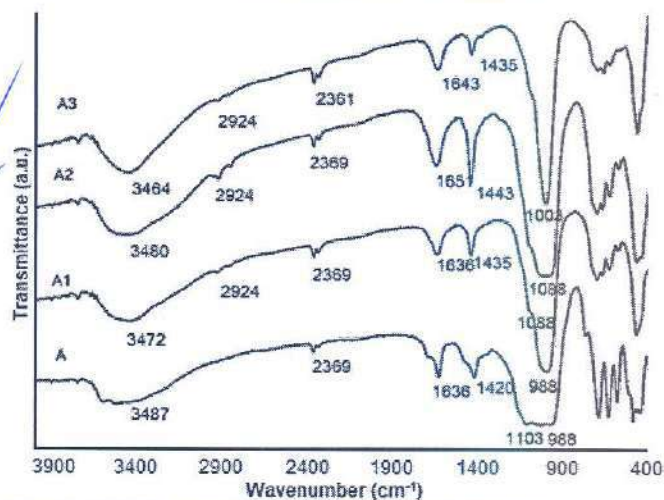


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5x10⁻⁴ M (A2) with CTAB 1x10⁻³ M (A3) with CTAB 1x10⁻² M

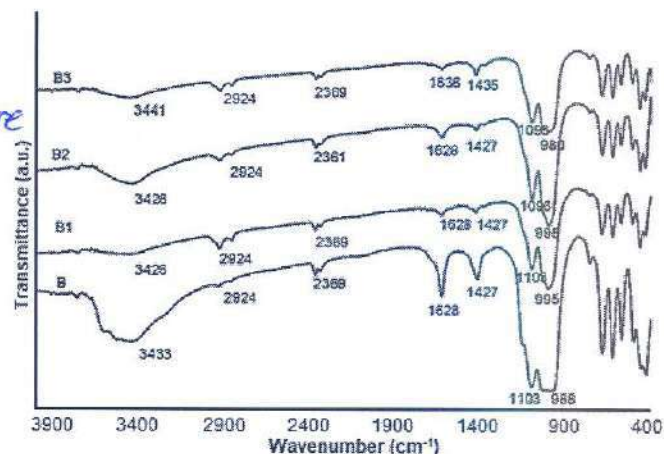


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5x10⁻⁴ M (B2) with CTAB 1x10⁻³ M (B3) with CTAB 1x10⁻² M

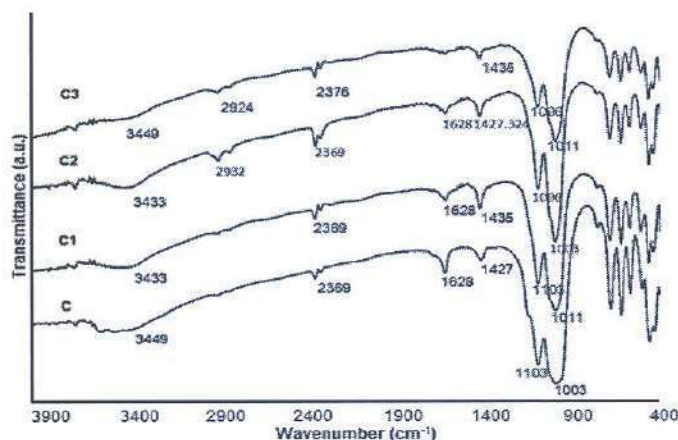


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5×10^{-4} M (C2) with CTAB 1×10^{-3} M (C3) with CTAB 1×10^{-2} M

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/ $[\text{SiO}_4]^{4-}$ and aluminate/ $[\text{AlO}_4]^{5-}$ anions primary building units. This interaction depends on surfactant concentration. The effect of surfactant presence can be observed on spectra A1, A2 and A3 in Fig. 1, B1, B2 and B3 in Fig. 2, C1, C2 and C3 in Fig. 3. It could be clearly observed that the presence of cetyltrimethylammonium (CTAB) surfactants has significantly affected the intensity and patterns of absorption at the wavenumber 950–1250 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.

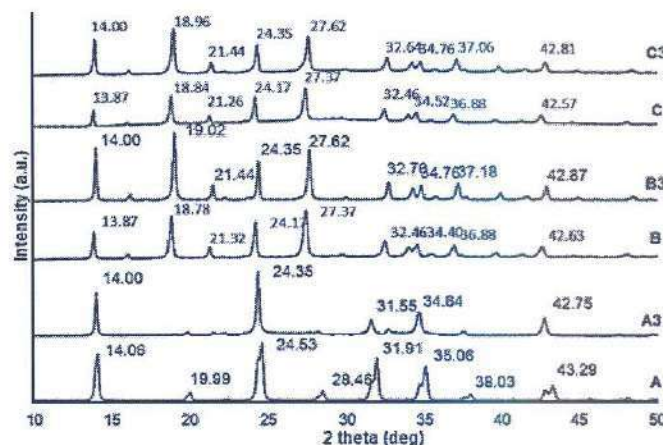


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

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

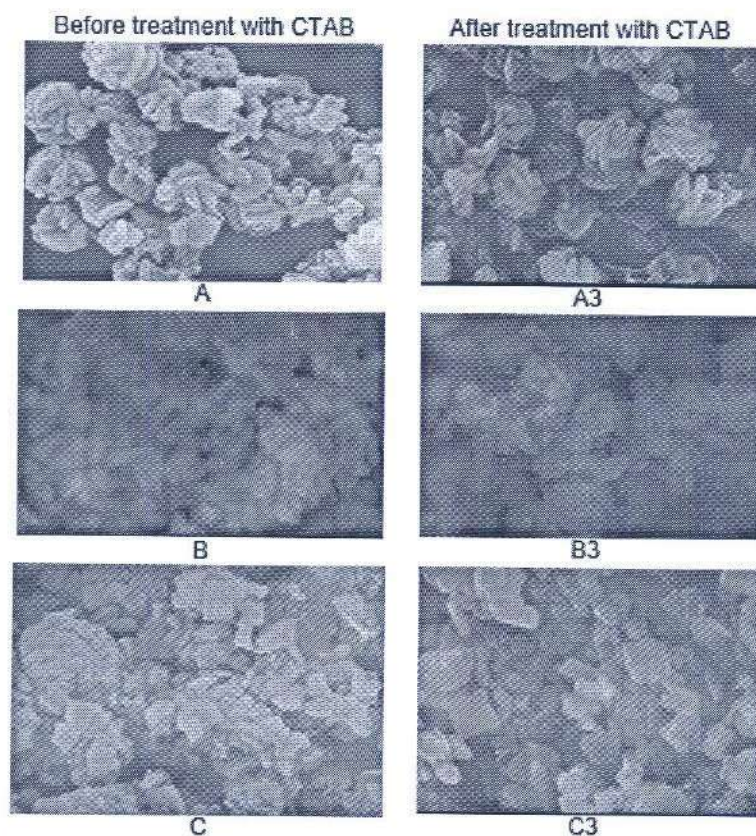


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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

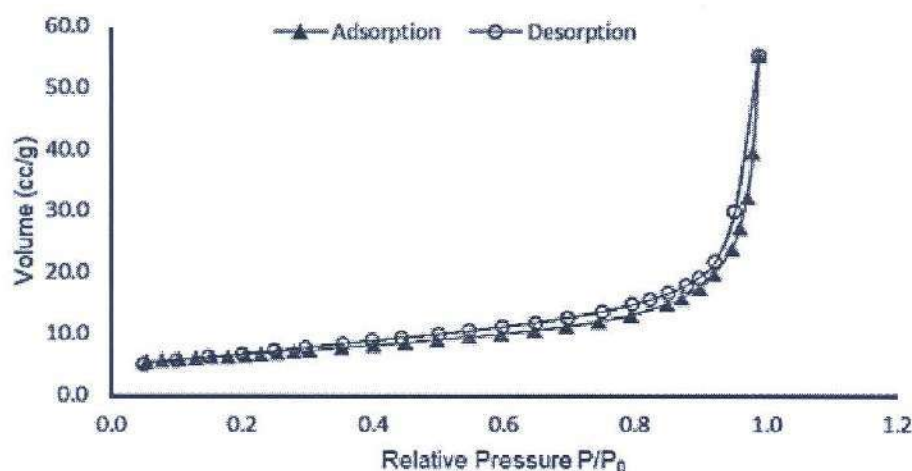


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

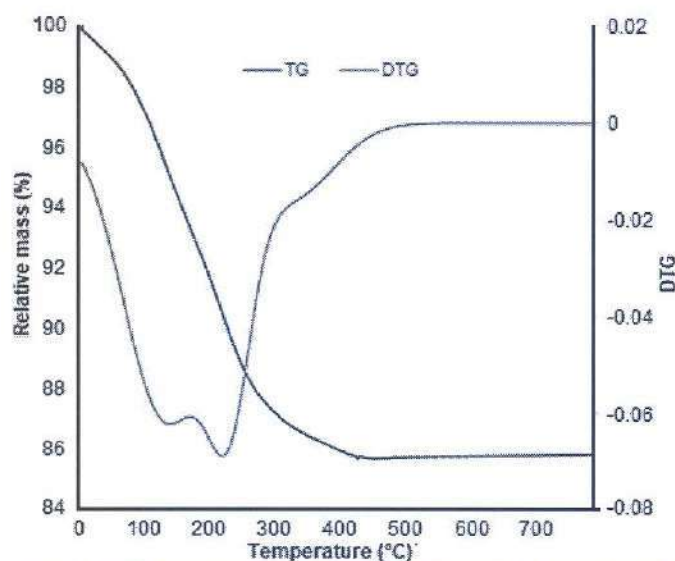


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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 m²/g. This reveals the existence

only a small number of active sorption sites. The total pore volume, V_p was found to be 8.56×10^{-2} cm³/g and average pore radius was determined as 7.737 Å. It means the NaP1 of this research 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 residues on the product zeolite surface.

CONCLUSION

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

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of

Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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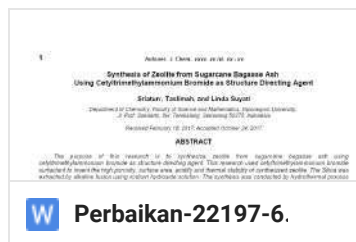
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Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

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Received February 18, 2017; Accepted October 24, 2017

ABSTRACT

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

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

ABSTRAK

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

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

INTRODUCTION

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

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

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

There are many methods applied to increase the economic values of sugarcane bagasse ash. One of these methods was extraction by alkaline fusion. This extraction produced silica as sodium silicate and a small amount of aluminate. If the sodium silicate solution was mixed with sodium aluminate solution would be occurred condensation polymerization under hydrothermal condition [5]. Most researchers have investigated some materials as low cost silicon and aluminum sources for synthesis of various zeolites. Synthesis of NaA zeolite from sugarcane bagasse ash [6], from kaolin waste [7], NaP1 zeolite from high silica fly ash [8-10], from illite-smectite [11], from aluminum solid waste [12]. NaX and 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^6, N^6 -tetramethylpropane-1,3-diamine) and HMPD ($N^1, N^1, N^6, N^6, 2, 2$ -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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

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

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

Procedure

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

RESULT AND DISCUSSION

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

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

It has been known that every type of zeolite has two kind of specific infrared vibration, they are internal and external linkage of tetrahedral (TO_4). In all products, there can be observed the internal linkage, there is asymmetric stretching vibration of TO_4 ($\text{T} = \text{Si}$ or Al) and appears at $950\text{--}1250\text{ cm}^{-1}$ wavenumber, whereas the symmetric stretching vibration at $650\text{--}720$ and 500 cm^{-1} . Furthermore, the wavenumber at $1050\text{--}1150\text{ cm}^{-1}$ was asymmetric stretching vibration, $750\text{--}820\text{ cm}^{-1}$ 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)		Surfactant CTAB (M)
	Sodium silicate	Sodium aluminate	
A	1	1	0
A1	1	1	5×10^{-4} (1/2 cmc)
A2	1	1	1×10^{-3} (cmc)
A3	1	1	1×10^{-2} (10 cmc)
B	15	1	0
B1	15	1	5×10^{-4} (1/2 cmc)
B2	15	1	1×10^{-3} (cmc)
B3	15	1	1×10^{-2} (10 cmc)
C	25	1	0
C1	25	1	5×10^{-4} (1/2 cmc)
C2	25	1	1×10^{-3} (cmc)
C3	25	1	1×10^{-2} (10 cmc)

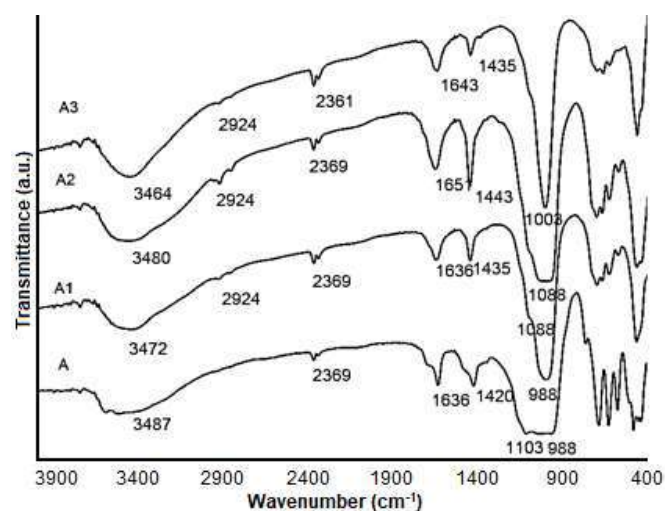


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

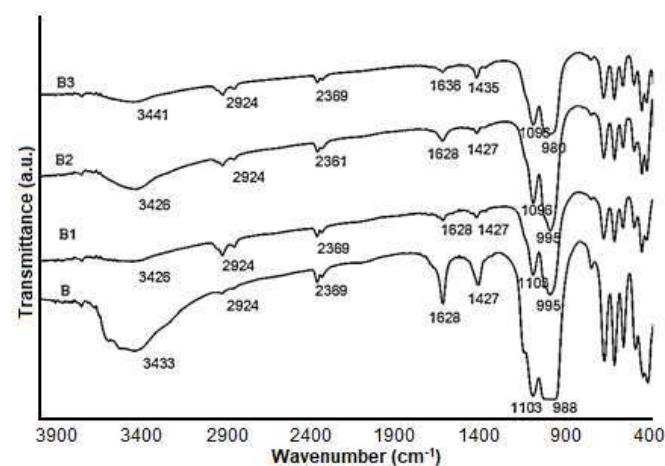


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

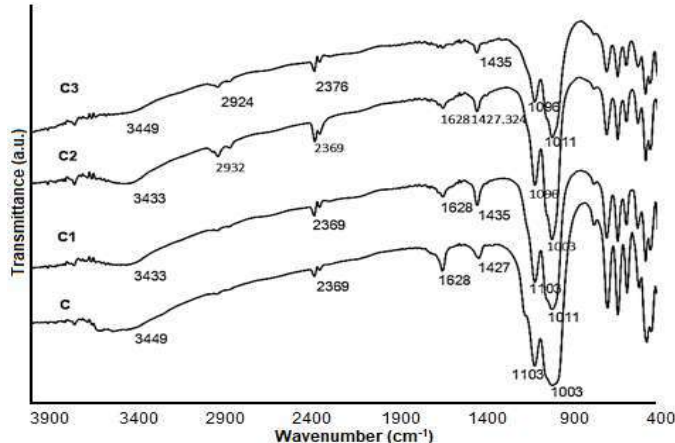


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5×10^{-4} M (C2) with CTAB 1×10^{-3} M (C3) with CTAB 1×10^{-2} M

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

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

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

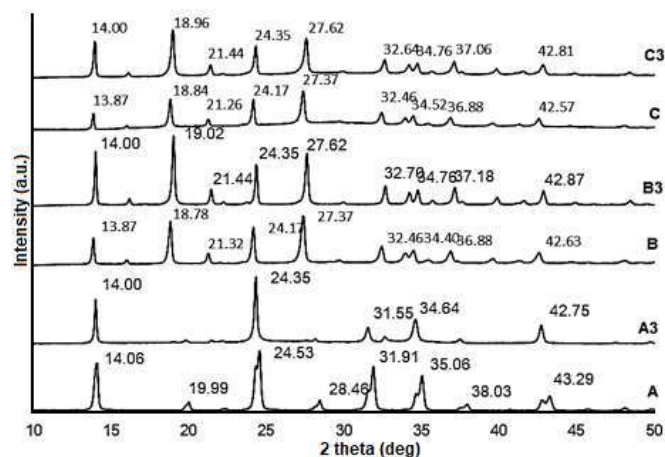


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

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

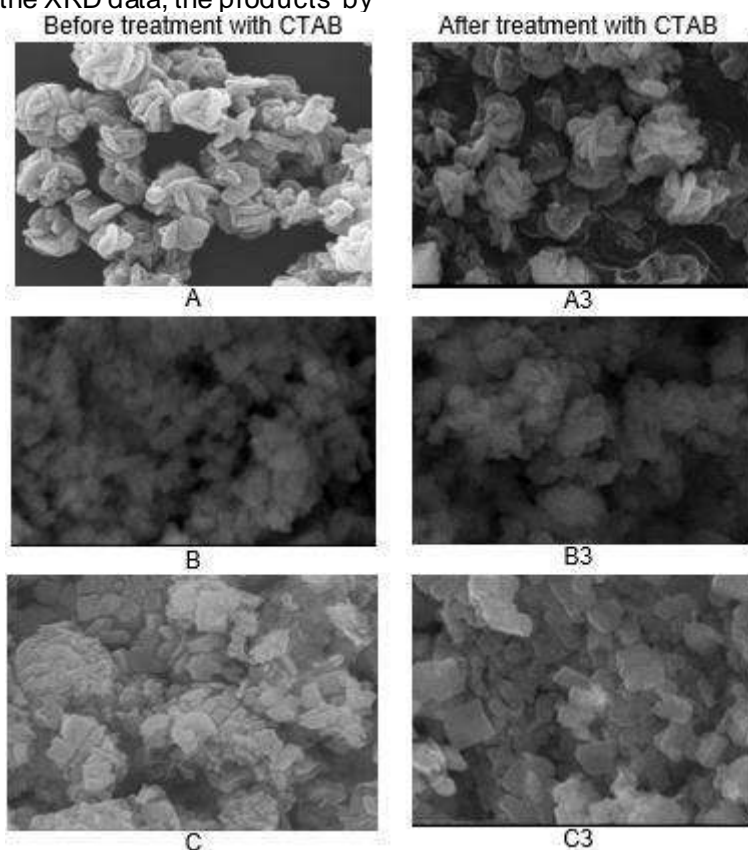


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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

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

Meanwhile the chemical composition of products synthesis in Table 2. Sample A1 and A3 are sodalite as the product of synthesis. For sample B1, B3, C1, and

C3 are NaP1. The entire product showed that ratio of Si/Al almost 1, these are consistent with the BET result

that this product is micropore material. The use of surfactant CTAB as a pore directing agent increases the

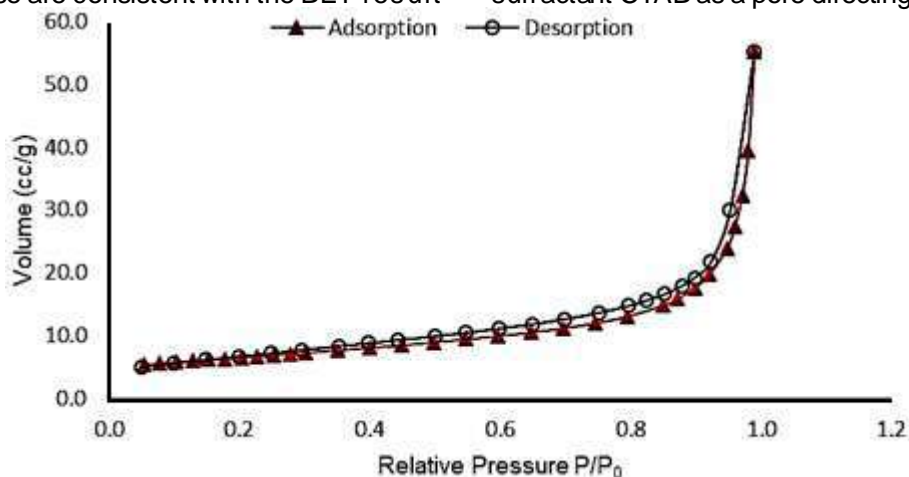


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

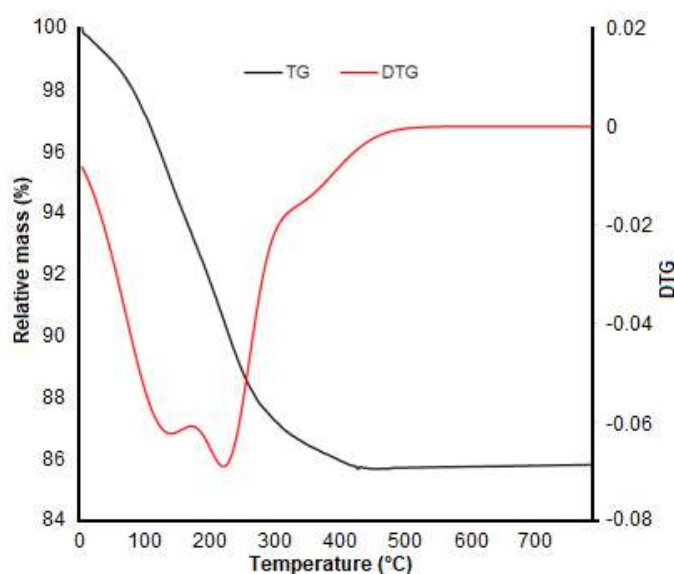


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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

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

The isotherm could be classified as type I of Brunauer's classification showing closed hysteresis loops. The calculated surface area of the prepared zeolite is relatively low $22.062 \text{ m}^2/\text{g}$. This reveals the existence only a small number of active sorption sites. The total pore volume, V_p was found to be $8.56 \times 10^{-2} \text{ cm}^3/\text{g}$ and average pore radius was determined as 7.737 \AA . 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°C which is due to the decomposition of the CTAB surfactant residues on the product zeolite surface.

CONCLUSION

In this study zeolite were successfully obtained from sugarcane bagasse ash by hydrothermal method at 100°C for 168 h. The type of zeolite was determined by the ratio of Si/Al in term the 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.

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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#22197 Summary

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Submission

Authors	Sriatun Sriatun, Taslimah Taslimah, Linda Suyati
Title	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
Original file	22197-41689-1-SM.DOCX 2017-02-18
Supp. files	None
Submitter	Mrs Sriatun Sriatun
Date submitted	February 18, 2017 - 09:27 PM
Section	Articles
Editor	Nuryono Nuryono
Abstract Views	1548

Author Fees

Article Submission	0.00 USD	PAY NOW
Article Publication	Paid December 13, 2017 - 10:18 AM	

Status

Status	Published Vol 18, No 1 (2018)
Initiated	2018-02-28
Last modified	2018-02-28

Artikel telah terbit pada tanggal 28 Februari 2018 Volume 18 No. 1

Submission Metadata

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Affiliation	Diponegoro University
Country	Indonesia
Bio Statement	Chemistry Department
Principal contact for editorial correspondence.	
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Keywords	surfactant; cetyltrimethylammonium bromide; synthesis; zeolite; sugarcane bagasse
Language	en

Supporting Agencies

Agencies	Sriatun, Diponegoro University; Faculty of Science and Mathematics, Department of Chemistry
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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>

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Abstract

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

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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Received February 18, 2017; Accepted October 24, 2017

ABSTRACT

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

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

ABSTRAK

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

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

INTRODUCTION

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

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

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

There are many methods 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, $\text{Al}(\text{OH})_3$ powder, cetyltrimethylammonium bromide powder, HF and HCl solution were purchase from Merck.

Instrumentation

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

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

Procedure

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

RESULT AND DISCUSSION

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

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

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

Table 1. The composition of precursor and surfactant

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

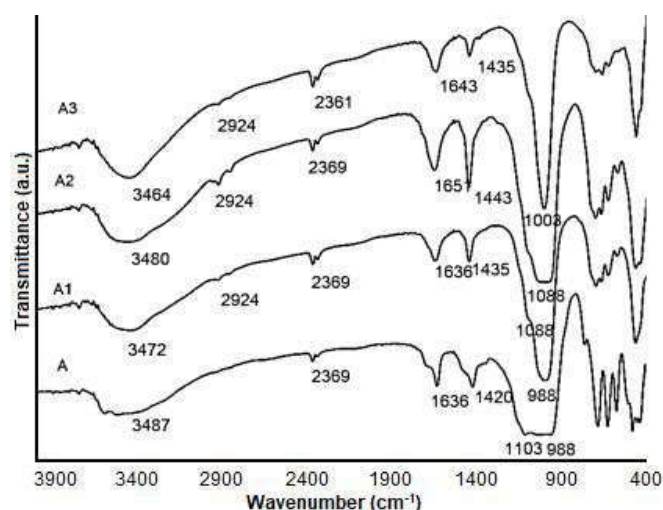


Fig 1. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 1 (A) without CTAB surfactant (A1) with CTAB 5×10^{-4} M (A2) with CTAB 1×10^{-3} M (A3) with CTAB 1×10^{-2} M

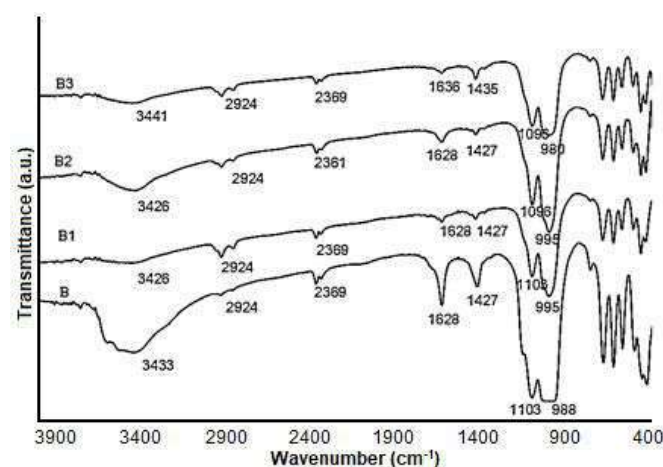


Fig 2. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 15 (B) without CTAB surfactant (B1) with CTAB 5×10^{-4} M (B2) with CTAB 1×10^{-3} M (B3) with CTAB 1×10^{-2} M

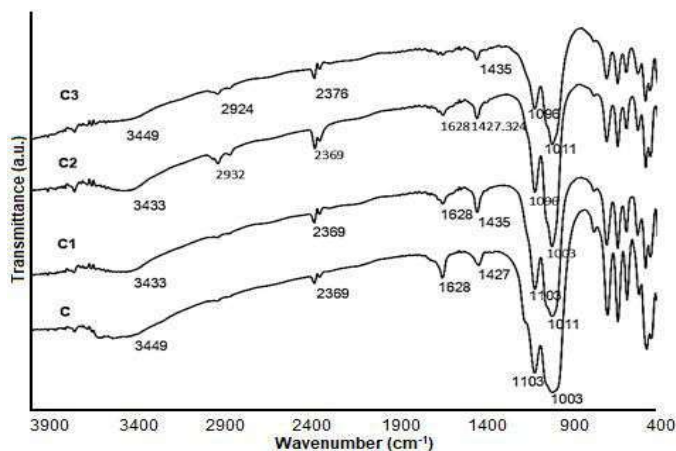


Fig 3. FTIR spectra of synthesized zeolite with ratio Si/Al (v/v) 25 (C) without CTAB surfactant (C1) with CTAB 5×10^{-4} M (C2) with CTAB 1×10^{-3} M (C3) with CTAB 1×10^{-2} M

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

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

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

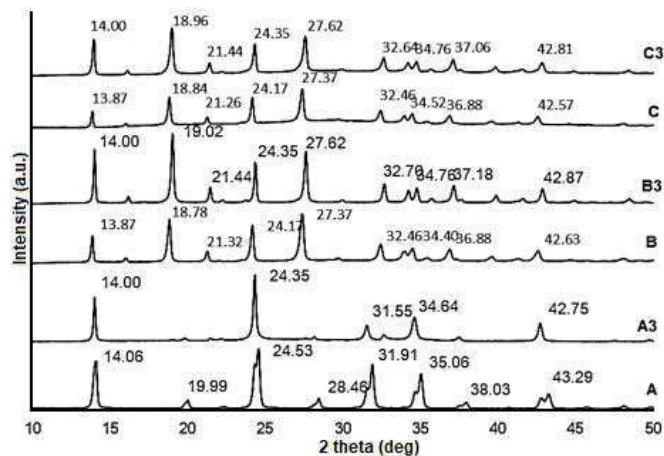


Fig 4. The pattern of diffractogram synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

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

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

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

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

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

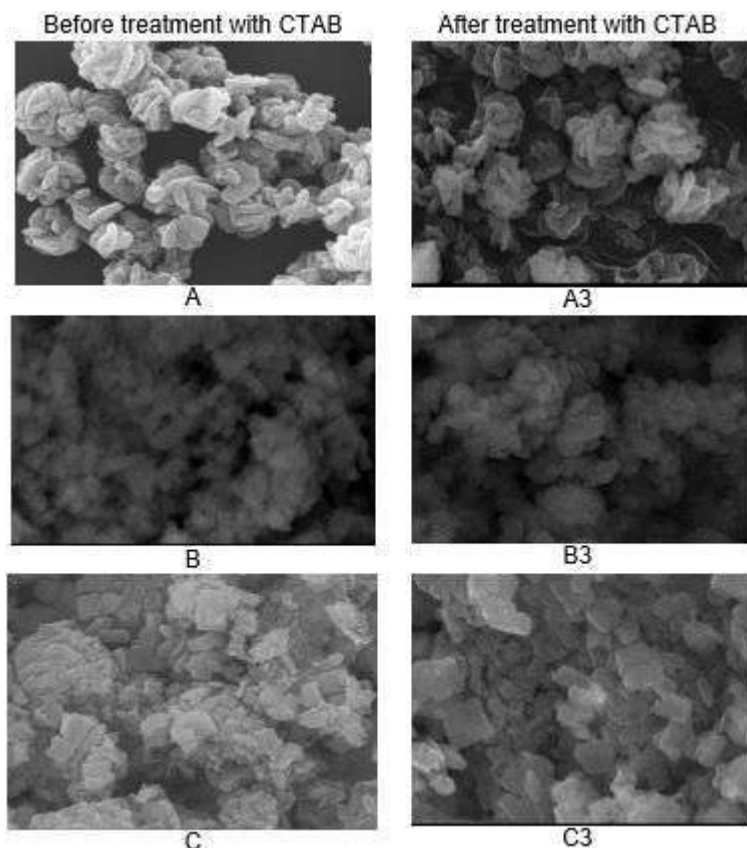


Fig 5. The SEM image of synthesized zeolite (A) Ratio Si/Al 1 without CTAB (A3) Ratio Si/Al 1 with CTAB 1×10^{-2} M (B) Ratio Si/Al 15 without CTAB (B3) Ratio Si/Al 15 with CTAB 1×10^{-2} M (C) Ratio Si/Al 25 without CTAB (C3) Ratio Si/Al 25 with CTAB 1×10^{-2} M

Table 2. The chemical composition of synthesized zeolite

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

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

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

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

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

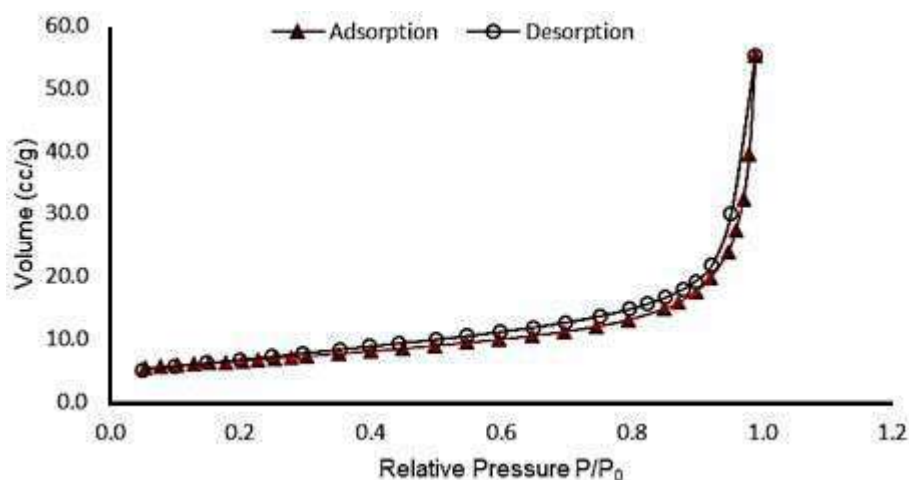


Fig 6. The isotherms adsorption of the synthesized product ratio Si/Al (v/v) 1

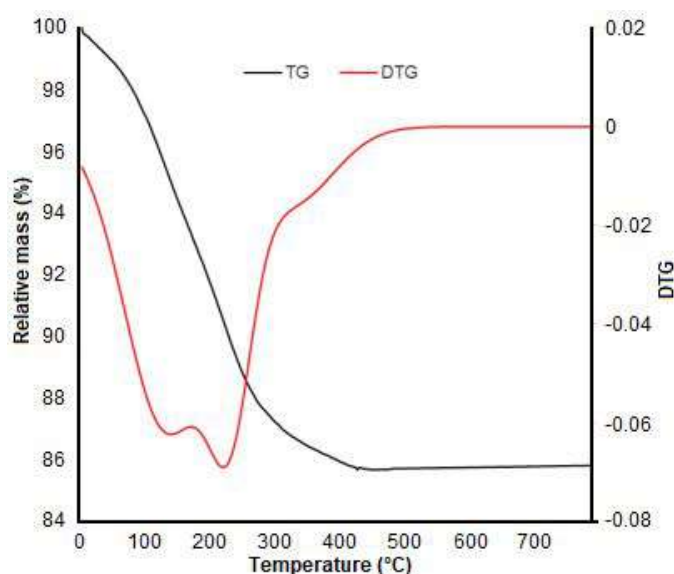


Fig 7. TGA of the synthesized product ratio Si/Al (v/v) 1

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

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

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

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

CONCLUSION

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

ACKNOWLEDGEMENT

Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.

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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

Yang menerangkan,



ISSN 1411-9420
Indones. J. Chem.
INDONESIAN JOURNAL OF CHEMISTRY

Prof. Dr. rer.nat. Nuryono, M.S
Editor in Chief Indonesian Journal of Chemistry

KLARIFIKASI ARTIKEL BUKAN BAGIAN DESERTASI

Judul : *“Synthesis of Zeolite from Sugarcane Bagasse Ash using Cetyltrimethylammonium Bromide as Structure Directing Agent”*

Penulis : Sriatun, Taslimah, Linda Suyati

Publikasi : Indonesian Journal of Chemistry (IJC) Vol. 18 No. 1, 159-165 Tahun 2018

- Artikel tersebut benar-benar bukan bagian dari desertasi studi S3 saya, dengan surat pernyataan terlampir (Lampiran 1)
- Artikel tersebut merupakan salah satu luaran penelitian skim hibah bersaing tahun 2013-2014 dengan judul *“Pengembangan Katalis Heterogen Berbasis Silika Alumina dari Ampas Tebu dan Aplikasinya pada Pembuatan Biodiesel dari Minyak Goreng Sisa Pakai”* dengan surat kontrak:
 1. Surat Penugasan Pelaksanaan Kegiatan Penelitian Multi Tahun Hibah bersaing tahun 2013 No. 154a-15/UN7.5/PG/2013 (Tahun pertama), Laporan akhir penelitian bisa di lihat di link berikut:
<https://doc-pak.undip.ac.id/3800/1/LAPORAN%20AKHIR%20HB%20TAHUN%20I%20SRIATUN.pdf>
 2. Surat Penugasan Pelaksanaan Kegiatan Penelitian Multi Tahun Hibah bersaing tahun 2014 No. 84-68/UN7.5.1/PG/2014 (Tahun kedua), Laporan akhir penelitian bisa dilihat pada link berikut:
<https://doc-pak.undip.ac.id/3800/2/LAPORAN%20AKHIR%20HB%20Tahun%20II%20Sriatun.pdf>
 3. Tim penelitian:
Ketua: Sriatun, S.Si, M.Si NIDN: 0015037102
Anggota 1: Dra. Taslimah, M.Si NIDN: 0009075602
Anggota 2: Dra. Linda Suyati, MSi NIDN: 0015016406

Luaran lainnya dari penelitian ini adalah:

1. Proceeding of 9th Joint Conference on Chemistry, 12-13 November 2014
Judul: *“Modification of Synthetic Zeolite from Bagasse Ash and Their Characterization”*
Penulis: Sriatun, Taslimah, Linda Suyati
Link artikel:
https://jcc.undip.ac.id/assets/attachments/JCC9%20-%20content/Proceeding%20content%20rev_Part21.pdf

Artikel mengkaji tentang modifikasi silika-alumina (zeolit) yang telah dibuat dengan agen ammonium klorida dan nikel klorida.

2. Publikasi pada jurnal nasional terakreditasi Jurnal Teknologi Industri Pertanian Vol. 25 No. 1 halaman 35-42 Tahun 2015

Judul: ***“Pemanfaatan Katalis Silika Alumina Dari Bagasse pada Pembuatan Biodiesel dari Minyak Goreng Sisa Pakai”***

Penulis: Sriatun, Taslimah, Linda Suyati

Link Artikel: <https://journal.ipb.ac.id/index.php/jurnaltin/article/view/9703>

Artikel ini mengkaji material silika alumina sebagai katalis pada reaksi transesterifikasi minyak goreng sisa pakai.

- Artikel tersebut sebelumnya pernah disubmit pada **International Journal of Renewable Energy Development (IJRED) pada tanggal 10 November 2016 (Lampiran 2)**

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

Penulis: Sriatun, Taslimah, Linda Suyati

Pada tanggal 13 November 2016 mendapatkan respon dari editor IJRED bahwa artikel tersebut **ditolak dengan alasan bidang (scope) tidak sesuai** (Lampiran 3).

- Artikel tersebut diperbaiki kembali setelah mengikuti FGD penyusunan manuskrip dan disubmit pada Indonesian Journal of Chemistry pada tanggal 18 Februari 2017 (Lampiran 4)

Judul: ***“The Effect of Cetyltrimethylammonium Bromide Surfactant in Synthesis of Zeolite from Sugarcane Bagasse Ash”***

Penulis: Sriatun, Taslimah, Linda Suyati

Judul tersebut sama dengan ketika disubmit pada IJRED, penulis adalah semua tim penelitian baik ketua maupun anggota.

Selanjutnya melalui proses review (**detail proses review sudah saya sertakan pada lembar korespondensi dan bisa diakses pada link: https://doc-pak.undip.ac.id/3073/2/C1_Kores.pdf**)

atas saran reviewer untuk menyesuaikan dengan hasil review diperlukan perubahan judul yang semula ***“The Effect of Cetyltrimethylammonium Bromide Surfactant in Synthesis of Zeolite from Sugarcane Bagasse Ash”*** menjadi ***“Synthesis of Zeolite from Sugarcane Bagasse Ash using***

Cetyltrimethylammonium Bromide as Structure Directing Agent” dan terpublikasi pada tanggal 28 Februari 2018 (Lampiran 5).

- Sebagai catatan saya mulai terdaftar sebagai mahasiswa S3 Program Doktor Teknik Kimia UNDIP pada September 2016 dan dengan promotor desertasi saya: (surat ijin belajar terlampir/atau bisa dilihat pada link: https://doc-pak.undip.ac.id/3801/1/Surat_Ijin_Belajar_Sriatun.pdf)

Promotor 1: Prof. Dr. rer. nat. Heru Susanto, S.T, M.T, M.M

Promotor 2: Prof. Dr. Widayat, S.T, M.T

Promotor 3: Adi Darmawan, S.Si, M.Si, Ph.D

Judul desertasi: Sintesis Katalis Bimetal/ZSM-5 untuk Produksi Bioavtur dengan Proses Hidrocracking

Publikasi: (**tidak digunakan pada pengajuan kenaikan pangkat ini**)

1. IOP Conf. Ser.: Mater. Sci. Eng. **509** 012138 Tahun 2019

Judul: Characteristic of ZSM-5 catalyst supported by nickel and molybdenum

Penulis: **Sriatun, Heru Susanto, Widayat, Adi Darmawan**

Link: <https://iopscience.iop.org/article/10.1088/1757-899X/509/1/012138/pdf>

2. Journal of Physics.: Conf. Ser. **1524** 011001 Tahun 2020

Judul: Synthesis of silica-rich zeolite using quaternary ammonium-based templates

Penulis: **S Sriatun, H Susanto, W Widayat, A Darmawan, S Sriyanti, R Kurniasari, R Kurniawati**

Link: <https://iopscience.iop.org/article/10.1088/1742-6596/1524/1/012087>

3. Indonesian Journal of Chemistry (**Accepted sampai tahap Proofreading**)

Judul: Hydrocracking of Coconut Oil on the NiO/Silica-Rich Zeolite Synthesized Using a Quaternary Ammonium Surfactant

Penulis: **Sriatun, Heru Susanto, Widayat, Adi Darmawan**

Catatan: Untuk detail klarifikasi dan data pendukung lainnya kami sertakan pada link berikut:

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SURAT PERNYATAAN DAN KLARIFIKASI ARTIKEL BUKAN BAGIAN DARI DESERTASI

Yang bertanda tangan dibawah ini saya:

Nama : Sriatun, S.Si, M.Si
 NIP : 197103151997022001
 Departemen : Kimia
 Fakultas : Sains dan Matematika
 Jenis : artikel penelitian
 Judul : Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent

Menyatakan dan mengklarifikasi bahwa artikel tersebut bukan merupakan bagian dari desertasi S3 saya. Adapun sebagai penguat hal tersebut adalah:

1. Artikel tersebut merupakan luaran penelitian yang berjudul **Pengembangan Katalis Heterogen Berbasis Silika Alumina dari Ampas tebu dan Aplikasinya pada Pembuatan Biodiesel dari Minyak Goreng Sisa Pakai** dari skim hibah bersaing tahun pelaksanaan 2014, dengan perjanjian pelaksanaan penelitian No. 84-68/UN7.5.1/PG/2014. Dalam Surat Pernyataan Validasi Karya Ilmiah, asal usul karya ilmiah dan sumber pendanaan telah disebutkan bahwa artikel tersebut berasal dari Program Hibah Bersaing tahun 2014 dengan nomer kontrak seperti tersebut di atas.
2. Pada bagian Acknowledgment dari artikel tersebut sudah tertuliskan sebagai berikut:
Acknowledgment
 Sriatun, Taslimah, and Suyati, L., gratefully acknowledge financial support from The Directorate of Research and Community Service, Ministry of Research, Technology and High Education via Grants Compete Award (Number: 84-68/UN7.5.1/PG/2014) and Chemistry Department of Diponegoro University for the facilities to carry out this research.
3. Artikel tersebut saya submit pada jurnal Indonesian Journal of Chemistry UGM pada tanggal **18 Februari 2017**.

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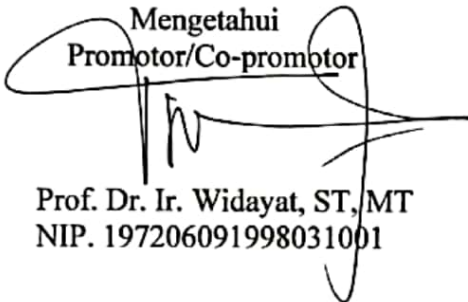
Authors	Sriatun Sriatun, Taslimah Taslimah, Linda Suyati
Title	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
Original file	22197-41689-1-SM.DOCX 2017-02-18
Supp. files	None
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Date submitted	February 18, 2017 - 09:27 PM
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Saya terdaftar sebagai mahasiswa S3 pada Departemen Teknik Kimia Fakultas Teknik UNDIP pada bulan September 2016, dimana pada masa submit artikel saya belum ada penentuan judul desertasi serta kegiatan laboratorium terkait studi S3 saya.

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Demikian surat klarifikasi ini saya buat dengan sebenar-benarnya.

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Prof. Dr. Ir. Widayat, ST, MT
NIP. 197206091998031001

Semarang, 16 Oktober 2020

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

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Sun, Nov 13, 2016 at 8:20 PM

To: Sriatun Sriatun <sriatun@live.undip.ac.id>

Dear Dr.Sriatun Sriatun:

Thank you for submitting your manuscript to the International Journal of Renewable Energy Development , "The Effect of cetyltrimethylammonium bromide surfactant in synthesis of zeolite from sugarcane bagasse ash". However, because we receive many more manuscripts than we can publish, we are limited to accepting about 1/3 of all submissions. Our policy is to favor articles of broad interest, high-novelty content, and good presentation. As a result of this policy, we must return a number of potentially interesting manuscripts.

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Authors	Sriatun Sriatun, Taslimah Taslimah, Linda Suyati
Title	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
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Title	Synthesis of Zeolite from Sugarcane Bagasse Ash Using Cetyltrimethylammonium Bromide as Structure Directing Agent
Abstract	<p>The purpose of this research is to synthesize zeolite from sugarcane bagasse ash using cetyltrimethylammonium bromide as structure directing agent. This research used cetyltrimethylammonium bromide surfactant to invent the high porosity, surface area, acidity and thermal stability of synthesized zeolite. The Silica was extracted by alkaline fusion using sodium hydroxide solution. The synthesis was conducted by hydrothermal process at 100 °C for 7 days, ageing process for 24 h and calcination at 500 °C for 5 h. The ratio of Si/Al (v/v) was 1, 15 and 25, meanwhile the concentration of cetyltrimethylammonium bromide was 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950\text{--}1050\text{ cm}^{-1}$ and $620\text{--}690\text{ cm}^{-1}$, $420\text{--}460\text{ cm}^{-1}$, double ring at $520\text{--}570\text{ cm}^{-1}$, pore opening at $300\text{--}370\text{ cm}^{-1}$. Vibration of -OH as silanol group or water was indicated by broad absorbance at $3400\text{--}3450\text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO₂ 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.</p>

Indexing

Keywords	surfactant; cetyltrimethylammonium bromide; synthesis; zeolite; sugarcane bagasse
Language	en

Supporting Agencies

Agencies	Sriatun, Diponegoro University; Faculty of Science and Mathematics, Department of Chemistry
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References	<p>[1] Srinivasan, R., and Sathiya, K., 2010, Experimental study on bagasse ash in concrete, <i>Int. J. Serv. Learn. Eng.</i>, 5 (2), 60–66.</p> <p>[2] <i>Indonesian Statistics</i>, 2012, http://www.bps.go.id.</p> <p>[3] Hanafi, A., and Nandang, A., 2010, Studi pengaruh bentuk silika dari ampas tebu terhadap kekuatan produk keramik, <i>Jurnal Kimia Indonesia</i>, 5 (1), 35–38.</p> <p>[4] Norsuraya, S., Fazlena, H., and Norhasyimi, R., 2016, Sugarcane bagasse as a renewable Source of silica to synthesize Santa Barbara Amorphous-15 (SBA-15), <i>Procedia Eng.</i>, 148, 839–846.</p> <p>[5] Cardoso, A.M., Paprocki, A., Ferret, L.S., Azevedo, C.M.N., and Pires, M., 2015, Synthesis of zeolite Na-P1 under mild conditions using Brazilian coal fly ash and its application in wastewater treatment, <i>Fuel</i>, 139, 59–67.</p> <p>[6] Moisés, M.P., da Silva, C.T.P., Meneguín, J.G., Giroto, E.M., and Radovanovic, E., 2013, Synthesis of zeolite NaA from sugarcane bagasse ash, <i>Mater. Lett.</i>, 108, 243–246.</p> <p>[7] Maia, A.Á.B., Neves, R.F., Angélica, R.S., and Pöllmann, H., 2015, Synthesis, optimisation and characterisation of the zeolite NaA using kaolin waste from the Amazon Region. Production of zeolites KA, MgA and CaA, <i>Appl. Clay Sci.</i>, 108, 55–60.</p> <p>[8] Kazemian, H., Naghdali, Z., Kashani, T.G., and Farhadi, F., 2010, Conversion of high silicon fly ash to Na-P1 zeolite: Alkaline fusion followed by hydrothermal crystallization, <i>Adv. Powder Technol.</i>, 21 (3), 279–283.</p> <p>[9] Babajide, O., Musyoka, N., Petrik, L., and Ameer, F., 2012, Novel zeolite Na-X synthesized from fly ash as a heterogeneous catalyst in biodiesel production, <i>Catal. Today</i>, 190 (1), 54–60.</p> <p>[10] Musyoka, N.M., Petrik, L.F., Fatoba, O.O., and Hums, E., 2013, Synthesis of zeolites from coal fly ash using mine waters, <i>Miner. Eng.</i>, 53, 9–15.</p> <p>[11] Baccouche, A., Srasra, E., and El Maaoui, M., 1998, Preparation of Na-P1 and sodalite octahydrate zeolites from interstratified illite-smectite, <i>Appl. Clay Sci.</i>, 13 (4), 255–273.</p> <p>[12] Sánchez-Hernández, R., López-Delgado, A., Padilla, I., Galindo, R., and López-Andrés, S., 2016, One-step synthesis of NaP1, SOD and ANA from a hazardous aluminum solid waste, <i>Microporous Mesoporous Mater.</i>, 226, 267–277.</p> <p>[13] Purnomo, C.W., Salim, C., and Hinode, H., 2012, Synthesis of pure Na-X and Na-A zeolite from bagasse fly ash, <i>Microporous Mesoporous Mater.</i>, 162, 6–13.</p> <p>[14] Wittayakun, J., Khemthong, P., and Prayoonpokarach, S., 2008, Synthesis and characterization of zeolite NaY from rice husk silica, <i>Korean J. Chem. Eng.</i>, 25 (4), 861–864.</p>
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LAMPIRAN 5

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⁾

(1) Diponegoro University

(2) Diponegoro University

(3) Diponegoro University

(*) 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 5×10^{-4} M, 1×10^{-3} M and 1×10^{-2} M. The result showed all of product have strong absorbance at $950-1050 \text{ cm}^{-1}$ and $620-690 \text{ cm}^{-1}$, $420-460 \text{ cm}^{-1}$, double ring at $520-570 \text{ cm}^{-1}$, pore opening at $300-370 \text{ cm}^{-1}$. Vibration of -OH as silanol group or water was indicated by broad absorbance at $3400-3450 \text{ cm}^{-1}$. The diffractograms XRD showed that the product had high crystallinity. The composition of product on ratio Si/Al 1 with concentration of cetyltrimethylammonium 10^{-2} M is sodalite, the ratio Si/Al 15 and 25 are NaP1 and SiO_2 quartz and contain 12.23% and 12.19% of Si, 4.17% and 13.18% of Al, respectively. Observation on SEM revealed that the crystal produced using cetyltrimethylammonium bromide were homogenous and regular in shape.

Keywords

surfactant; cetyltrimethylammonium bromide; synthesis; zeolite; sugarcane bagasse

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References

- [1] Srinivasan, R., and Sathiya, K., 2010, Experimental study on bagasse ash in concrete, *Int. J. Serv. Learn. Eng.*, 5 (2), 60–66.
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Brazilian coal fly ash and its application in wastewater treatment, *Fuel*, 139, 59–67.

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[10] Musyoka, N.M., Petrik, L.F., Fatoba, O.O., and Hums, E., 2013, Synthesis of zeolites from coal fly ash using mine waters, *Miner. Eng.*, 53, 9–15.

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 DOI: <https://doi.org/10.22146/ijc.22197>

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Indonesian Journal of Chemistry (ISSN 1411-9420 / 2460-1578) - Chemistry Department, Universitas Gadjah Mada, Indonesia.

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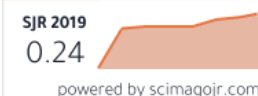
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**LAPORAN AKHIR
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**PENGEMBANGAN KATALIS HETEROGEN
BERBASIS SILIKA ALUMINA DARI AMPAS TEBU DAN APLIKASINYA PADA
PEMBUATAN BIODIESEL DARI MINYAK GORENG SISA PAKAI**

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tanggal 05 Desember 2012**

**UNIVERSITAS DIPONEGORO
NOVEMBER, 2013**

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RINGKASAN

Dalam rangka mendapatkan katalis heterogen berbasis silika alumina yang akan digunakan untuk mengkonversi minyak goreng sisa pakai menjadi biodiesel maka pada penelitian di tahun pertama ini dikembangkan material silika alumina dengan memanfaatkan ampas tebu sebagai sumber silika. Tujuan khusus penelitian tahun pertama adalah memperoleh material katalis heterogen berbasis silika alumina (zeolit) dengan ukuran pori dan luas permukaan yang besar serta stabilitas termal yang tinggi, sedangkan rencana di tahun kedua produk penelitian ini digunakan untuk memperoleh biodiesel dari minyak goreng sisa pakai.

Untuk mencapai tujuan pada tahun pertama tersebut, dilakukan serangkaian tahap kegiatan yaitu (1) pembuatan natrium silikat dari ampas tebu dan pembuatan natrium aluminat (2) sintesis material katalis. Parameter yang dikaji adalah rasio Si/Al, waktu kontak dan jenis surfaktan. Rasio Si/Al rendah yaitu 1, 2 dan 3, rasio sedang 5, 7,5 dan 15 serta rasio tinggi 25, 50 dan 100. Waktu kontak pada proses sintesis adalah 3 hari dan 7 hari. Sedangkan surfaktan yang digunakan untuk modifikasi adalah surfaktan cetiltrimetilamonium (CTAB) dan polietilenglikol (PEG).

Hasil penelitian menunjukkan bahwa ampas tebu mengandung Si 30,45 %, Al 3,1 %, sedangkan hasil ekstraksi dari ampas tersebut mengandung Si 70,82 % dan Al 1,04 %. Berdasarkan data FTIR menunjukkan bahwa semua produk sintesis tanpa surfaktan maupun yang disintesis menggunakan surfaktan CTAB maupun PEG sebagai pencetak pori memiliki serapan sangat kuat dengan pola khas pada daerah $960-1103\text{ cm}^{-1}$ yang merupakan vibrasi ulur asimetrik TO_4 ($\text{T} = \text{Si}$ atau Al), $650-850\text{ cm}^{-1}$ merupakan vibrasi ulur simetrik TO_4 , $500-650\text{ cm}^{-1}$ merupakan vibrasi eksternal *double ring* ciri khas dari material silika alumina tiga dimensi, $420-500\text{ cm}^{-1}$ merupakan vibrasi tekuk Si-O atau Al-O dan $300-420\text{ cm}^{-1}$ merupakan vibrasi *open pore* dari material ini serta daerah 3400 cm^{-1} yang merupakan vibrasi OH dari gugus silanolnya. Produk sintesis merupakan kristal silika alumina yang dibuktikan oleh adanya perubahan bentuk amorf dari silika ampas tebu menjadi kristal pada difraktogram XRD dari produk. Puncak utama produk rasio rendah pada $2\theta = 24,589$; $14,116$; $31,914$ yang merupakan senyawa sodalit dengan rumus kimia $\text{Na}_6\text{Al}_6\text{Si}_6\text{O}_{24}\text{Na}_2(\text{OH})_2(\text{H}_2\text{O})_2$ dan berstruktur ortorombik. Peningkatan rasio Si/Al menyebabkan pergeseran ke sudut 2θ lebih rendah serta munculnya puncak baru yaitu pada $2\theta = 13,874$; $27,374$ yang merupakan puncak silika alumina jenis NaA. Penggunaan surfaktan baik dengan CTAB maupun PEG pada rasio Si/Al 1 7 D menyebabkan pergeseran ke sudut 2θ lebih rendah namun intensitas puncaknya naik, sedangkan pada rasio 15 7D 10, 25 7D 10 dan 25 7D 05 pergeseran ke sudut yang lebih besar dan intensitasnya mengalami kenaikan, dengan demikian produk ini lebih kristalin. Dari data SEM EDX diketahui bahwa produk homogen dengan Si 17,04% dan Al 18,97 %.

PRAKATA

Alhamdulillah, puji syukur kami panjatkan kehadiran Allah SWT yang senantiasa memberikan hidayah dan petunjuk, sehingga peneliti dapat menyelesaikan penelitian yang berjudul “Pengembangan Katalis Heterogen Berbasis Silika Alumina dari Ampas Tebu dan Aplikasinya Pada Pembuatan Biodiesel dari Minyak Goreng Sisa Pakai,” ini dengan baik.

Pada kesempatan ini, peneliti mengucapkan banyak terima kasih kepada:

1. Pemberi dana penelitian dalam hal ini Ditlitabmas Dikti
2. Lembaga Penelitian Undip yang memberikan kesempatan kepada Peneliti untuk melaksanakan penelitian ini.
3. Dekan Fakultas MIPA, Ketua Jurusan Kimia dan Kepala laboratorium kimia anorganik yang telah memberikan fasilitas untuk terlaksananya penelitian ini.
4. Semua pihak yang telah membantu dari awal sampai terselesaikannya penelitian ini yang tidak bisa peneliti sebutkan satu persatu.

Peneliti menyadari sepenuhnya bahwa apa yang peneliti sampaikan masih jauh dari sempurna, untuk itu peneliti berharap ada pengembangan lebih lanjut dari penelitian ini. Besar harapan kami semoga informasi dari penelitian ini dapat berguna bagi para pembaca.

Semarang, November 2013

Peneliti

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**LAPORAN AKHIR
PENELITIAN HIBAH BERSAING
(BOPTN)**



**PENGEMBANGAN KATALIS HETEROGEN
BERBASIS SILIKA ALUMINA DARI AMPAS TEBU
DAN APLIKASINYA PADA PEMBUATAN BIODIESEL
DARI MINYAK GORENG SISA PAKAI**

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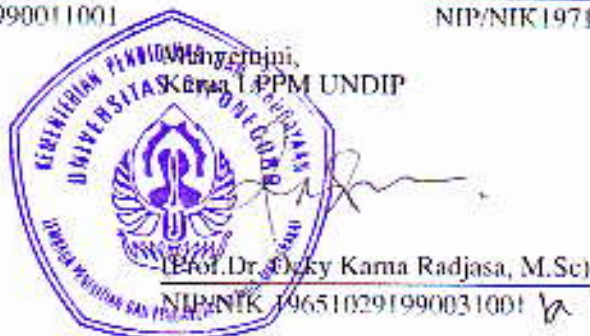
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RINGKASAN

Dalam rangka mendapatkan katalis heterogen berbasis silika alumina yang akan digunakan untuk mengkonversi minyak goreng sisa pakai menjadi biodiesel maka pada penelitian ini dikembangkan material silika alumina dengan memanfaatkan ampas tebu sebagai sumber silika. Tujuan khusus penelitian tahun pertama adalah memperoleh material katalis heterogen berbasis silika alumina (zeolit) dengan luas permukaan yang besar serta stabilitas termal yang tinggi, untuk memperoleh biodiesel dari minyak goreng sisa pakai.

Untuk mencapai tujuan pada tahun pertama tersebut, dilakukan serangkaian tahap kegiatan yaitu (1) pembuatan zeolit dari natrium silikat hasil ekstraksi abu ampas tebu yang direaksikan dengan natrium aluminat (2) Modifikasi terhadap zeolit yang disintesis dari abu ampas tebu menggunakan garam NH_4Cl dan NiCl_2 . (3) Karakterisasi produk sintesis menggunakan XRF, SEM, keasaman, BET dan DTA-TGA. (4) Uji katalitik katalis hasil sintesis untuk mengkonversi minyak goreng sisa pakai menjadi biodiesel pada variasi rasio minyak:metanol (6:1, 3: dan 1:6) waktu kontak (60 dan 120 menit). Karakter produk reaksi konversi ditentukan dengan GC, GC-MS, densitas, viskositas dan titik nyala.

Hasil penelitian menunjukkan bahwa pada data XRF, adanya modifikasi dengan garam NH_4Cl hanya mampu mengurangi sekitar 1% kadar Si, sedangkan adanya NiCl_2 menurunkan kadar Si sekitar 26 %. Kadar nikel yang tinggi juga didukung oleh morfologi permukaan pada pengamatan dengan SEM. Modifikasi dengan NH_4Cl dan NiCl_2 mampu meningkatkan keasaman total dan luas permukaan material zeolit. Analisis DTA menunjukkan bahwa adanya modifikasi dengan NH_4Cl menggeser daerah endotermik dari 104,3 -165,6°C menjadi 62,2-142,0°C sedangkan pada NiCl_2 menjadi 39,3-106,8°C. Termogram TGA menunjukkan keseluruhan produk sintesis maupun modifikasi memiliki kestabilan termal setelah temperatur 320°C. Pada uji katalitik rendemen produk reaksi konversi lebih tinggi pada rasio methanol:minyak 1:3 selama 60 menit, namun hanya sekitar 13,83 % yang berhasil dikonversi menjadi biodiesel tetapi ketika rasio minyak:methanol 1:6 rendemen menjadi 85,51 %. Densitas produk reaksi konversi berkisar 0,827-0,907, sedangkan titik nyalanya antara 106-110°C.

PRAKATA

Alhamdulillah, puji syukur kami panjatkan kehadirat Allah SWT yang senantiasa memberikan hidayah dan petunjuk, sehingga peneliti dapat menyelesaikan penelitian yang berjudul “Pengembangan Katalis Heterogen Berbasis Silika Alumina dari Ampas Tebu dan Aplikasinya Pada Pembuatan Biodiesel dari Minyak Goreng Sisa Pakai,” ini dengan baik.

Pada kesempatan ini, peneliti mengucapkan banyak terima kasih kepada:

1. Pemberi dana penelitian dalam hal ini Ditlitabmas Dikti
2. Lembaga Penelitian Undip yang memberikan kesempatan kepada Peneliti untuk melaksanakan penelitian ini.
3. Dekan Fakultas MIPA, Ketua Jurusan Kimia dan Kepala laboratorium kimia anorganik yang telah memberikan fasilitas untuk terlaksananya penelitian ini.
4. Semua pihak yang telah membantu dari awal sampai terselesaikannya penelitian ini yang tidak bisa peneliti sebutkan satu persatu.

Peneliti menyadari sepenuhnya bahwa apa yang peneliti sampaikan masih jauh dari sempurna, untuk itu peneliti berharap ada pengembangan lebih lanjut dari penelitian ini. Besar harapan kami semoga informasi dari penelitian ini dapat berguna bagi para pembaca.

Semarang, November 2014

Peneliti

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