

MJFAS MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCE

PRINT ISSN: 2289-5981 | ONLINE ISSN: 2289-599X

HOME ABOUT LOGIN REGISTER SEARCH CURRENT ARCHIVES
 ANNOUNCEMENTS SUBMISSION EDITORIAL TEAM REVIEWERS IBNU SINA
 INSTITUTE UNIVERSITI TEKNOLOGI MALAYSIA

Home > Vol 15, No 5 (2019)

Malaysian Journal of Fundamental and Applied Sciences



MJFAS promotes and supports I'M Research Consortium



MJFAS is the recipient of Current Research in Malaysia (CREAM 2018) Awards by the Ministry of Education Malaysia.



Journal abbreviation: **Mal. J. Fund. Appl. Sci.**
 ISSN: 2289-5981. e-ISSN: 2289-599X.

Citations	h-index	i10-index
1187	14	23

2019 *Google Scholar* Citation Reports



The Malaysian Journal of Fundamental and Applied Sciences (MJFAS) (formerly known as Journal of Fundamental Sciences (2005-2011), ISSN: 1823-626X) is a refereed research journal published by Penerbit UTM Press, Universiti Teknologi Malaysia. The aims and scope of the journal encompass research articles, original research reports, reviews, short communications and scientific commentaries from fundamental principles to practical applications in the broad field of mathematics, physics, chemistry, and biology. All manuscript submissions must be made through the journal's online manuscript system at [Online Submissions](#). This journal is indexed by [Clarivate Analytics](#) (formerly Thomson Reuters) and [Google Scholar](#).

Starting in the year 2019, MJFAS is published online with a frequency of six (6) issues per year (February, April, June, August, October, and December). Besides that, special issues of MJFAS will be published non-periodically from time to time.

The Malaysian Journal of Fundamental and Applied Sciences (MJFAS) is now entering its fourteen years and it is now the official journal of the [Indonesia-Malaysia Research Consortium](#) (I'M Research Consortium). The consortium's aims are to foster the development of the field through a cross-disciplinary approach and to reach consensus in areas of common interest in fundamental and applied sciences field. By making The Malaysian Journal of Fundamental and Applied Sciences the official journal of the I'M Research Consortium, we hope to provide a forum to bring together society members and to publish peer-reviewed consensus documents that emerge from the activities of the consortium.

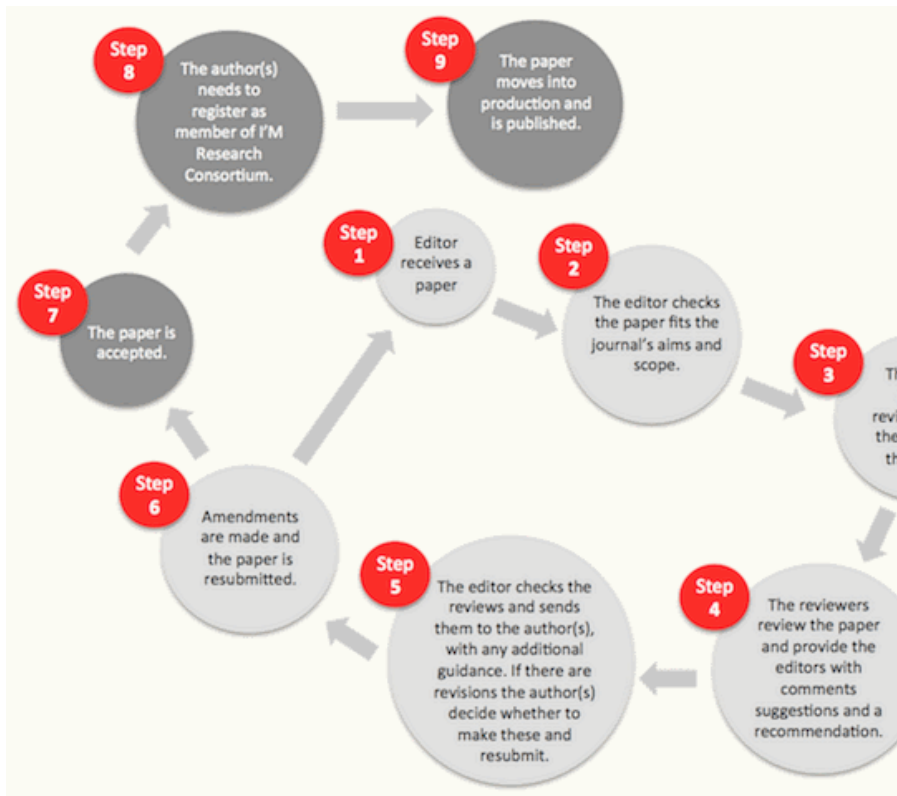


The Publishing Process of MJFAS and its Requirements

Below are the new requirements and other guidelines related to publication as well as communicating your results.

JOURNAL CONTENT
 Search

[Click image to enlarge]



Search Scope

All

Search

Browse

- [By Issue](#)
- [By Author](#)
- [By Title](#)

INFORMATION

- [For Readers](#)
- [For Authors](#)
- [For Librarians](#)

USER

Username

Password

Remember me

Login

FONT SIZE

Before the paper moves into production and is published (**Step 9**), if the author(s) is interested, the author(s) needs to register as a member of I'M Research Consortium (**Step 8**) through the following website:

<http://imresearchconsortium.ning.com/main/authorization/signUp>

The MJFAS is an Open Access journal. Publishing an article in the MJFAS requires article processing charges that will be billed to the submitting author following the acceptance of an article for publication. Apart from these article processing charges, there are no submission charges, page charges, or color charges. The fee to be paid following the acceptance of an article. The fee is **300 Malaysian Ringgit**. Article processing charges will be used for operating expenses of the journal.

For a detailed guideline for submission of an article please refer to [Online Submission](#). Please follow these guidelines to prepare and upload your article.

Editor's Message

Dear Colleagues,



As the Editor-in-Chief of the Malaysian Journal of Fundamental and Applied Sciences, I am writing to invite you to submit your most important research to the Journal. We envision the Journal as the best place to publish all of the levels of research in fundamental and applied sciences from all over the world, especially from researchers in Malaysia dan Indonesia.

Working with our knowledgeable and international Editorial Board members and I can assure you of a rapid, robust and fair peer-review process. As the Malaysian Journal of Fundamental and Applied Sciences is now indexed by [Clarivate Analytics](#) (formerly Thomson Reuters), we are especially aiming to reduce time to decision. We also have begun to work towards raising the Journal's impact factor.

For the coming submissions, the MJFAS will start implementing the "Your Paper, Your Way" initiative. In this way, authors can focus on the scientific quality of the paper. Journal-specific formatting such as reference style is no longer needed.

Thank you in advance for your valuable contributions to the Malaysian Journal of Fundamental and Applied Sciences.

Sincerely,

Prof. Dr. Hadi Nur ([short bio](#))
 Editor-in-chief
 e-mail: hadi@ibnusina.utm.my
 website: <http://hadinur.com>

Home > About the Journal > **Editorial Team**

Editorial Team

Editor-in-Chief

[Prof Dr Hadi Nur](#), Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia

Regional Managing Editor for North America and Europe

[Assoc Prof Dr Hendra Hermawan](#), Department of Mining, Metallurgical and Materials Engineering & CHU de Québec Research Center, Laval University, Canada

Regional Managing Editor for the Middle East and Africa

[Assoc Prof Dr Oki Muraza](#), Chemical Engineering Department, King Fahd University of Petroleum & Minerals, Dhahran, Saudi Arabia

Special Section Editor

[Dr Sheela Chandren](#), Universiti Teknologi Malaysia, Malaysia

Editors

[Prof Dr Bassim H Hameed](#), School of Chemical Engineering, Universiti Sains Malaysia
[Prof Dr Rafaqat Hussain](#), COMSATS Institute of Information Technology, Pakistan
[Prof Dr Taufiq Yap Yun Hin](#), Department of Chemistry, Putra University, Malaysia
[Prof Dr Tahir Ahmad](#), Universiti Teknologi Malaysia, Malaysia
[Prof Dr Takashi Suzuki](#), Department of Information and Computer Sciences Mathematical Science Course, Osaka University School of Engineering Science
[Assoc Prof Dr rer nat Rino R Mukti](#), Insitut Teknologi Bandung, Indonesia
[Assoc Prof Dr KK Viswanathan](#), Kuwait College of Science and Technology, Kuwait
[Assoc Prof Dr Sharif H Zein](#), University of Hull, UK
[Assoc Prof Dr Riadh Sahnoun](#), Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Norma Alias](#), Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Siew Ling Lee](#), Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Sib Krishna Ghoshal](#), Dept. of Physics, Fac. of Science, Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Suhairul Hashim](#), Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Yun Hau Ng](#), School of Energy and Environment, City University of Hong Kong
[Assoc Prof Dr Hong Heng See](#), Universiti Teknologi Malaysia, Malaysia
[Assoc Prof Dr Nik Ahmad Nizam Nik Malek](#), Universiti Teknologi Malaysia
[Dr Dedy Hermawan Baqos Wicaksono](#), Swiss German University, Indonesia
[Dr Hendrik Oktendy Lintang](#), Ma Chung University, Indonesia
[Dr Yung Szen Yap](#), Universiti Teknologi Malaysia, Malaysia
[Dr Wan Heng Fong](#), Universiti Teknologi Malaysia, Malaysia
[Dr Eng Ferry Iskandar](#), Physics of Electronic Materials Research Div. Department of Physics, Faculty of Mathematic and Natural Sciences, Institut Teknologi Bandung
[Dr Leny Yuliat](#), Ma Chung University, Indonesia

Editorial Office

[Ms Siti Nur Sakinah Ahmad](#)
[Ms Sabariah Ajis](#)



Copyright © 2005-2019 Penerbit UTM Press, Universiti Teknologi Malaysia. Disclaimer: This website has been updated to the best of our knowledge to be accurate. However, Universiti Teknologi Malaysia shall not be liable for any loss or damage caused by the usage of any information obtained from this website.



MJFAS is the recipient of Current Research in Malaysia (CREAM 2018) Awards by the Ministry of Education Malaysia.



JOURNAL CONTENT

Search

Search Scope

Browse

- [By Issue](#)
- [By Author](#)
- [By Title](#)

INFORMATION

- [For Readers](#)
- [For Authors](#)
- [For Librarians](#)

USER

Username

Password

Remember me

FONT SIZE



Home > Archives > Vol 14, No 4 (2018)

Vol 14, No 4 (2018)

October - December

Table of Contents

Research Article

Norm estimations, continuity, and compactness for Khatri-Rao products of Hilbert Space operators Arnon Ploymukda, Pattrawut Chansangiam	PDF 382-386
Mini review: Application of supercritical carbon dioxide in extraction of propolis extract Nor Faadila Mohd Idrus, Lee Nian Yan, Zuhaili Idham, Noor Aiyah Aris, Nicky Rahmana Putra, Ahmad Hazim Abdul Aziz, Mohd Azizi Che Yunus	PDF 387-396
West African kenaf (Hibiscus Cannabinus L.) natural fiber composite for application in automotive industry Tijjani Abdullahi, Zawati Harun, Mohd Hafiz Dzarfan Othman, Nasiru Aminu, Oguntunde Gabriel, Tijjani Aminu, Siti Aida Ibrahim, Noor Hasliza Kamarudin	PDF 397-402
Synthesis and characterization of two-stage curing reactive bio-based polymers Jing Jing Ang, Tuck Whye Wong, Zulhairun Abdul Karim	PDF 403-407
Characteristics of coconut frond as a potential feedstock for biochar via slow pyrolysis Nur Syaikh Mohamad Aziz, Adilah Shariff, Nurhayati Abdullah, Nurhidayah Mohamed Noor	PDF 408-413
Synthesis and characterization of zeolite NaX from Bangka Belitung Kaolin as alternative precursor Vita Nur Ifitahiyah, Didik Prasetyoko, Hadi Nur, Hasliza Bahruji, Hartati Hartati	PDF 414-418
Low cost palm oil-derivative based ceramic membranes for oily water separation Zhong Sheng Tai, Mohd Hafiz Dzarfan Othman, Siti Khadijah Hubadillah, Ahmad Fauzi Ismail, Mukhlis A Rahman, Juhana Jaafar, Khong Nee Koo, Mohd Haiqal Abd Aziz	PDF 419-424
Mechanical and flammability properties of poly(lactic acid)/poly(butylene adipate-co-terephthalate) blends and nanocomposites: Effects of compatibilizer and graphene Nilesh Kumar Shrivastava, Ooi Shu Wooi, Azman Hassan, Ibrahim Mohammed Inuwa	PDF 425-431
Evaluation of Swietenia mahagoni Jacq seed extracts in promoting wound healing properties Hartati Hartati, Hasmida Mohd-Nasir, Liza Md Salleh, Irma Suryani Idris, Azila Abd Aziz	PDF 432-436
Tensile behaviour for mercerization of single kenaf fiber Mohamad Ikhwan Ibrahim, Mohamad Zaki Hassan, Rozzeta Dolah, Mohd Zuhri Mohamed Yusoff, Mohd Sapuan Salit	PDF 437-439
Improvement of mechanical properties and fatigue life by shot peening process on ASTM A516 Grade 70 steel Mohd Rashdan Isa, Omar Suliman Zaroog, Kalaikathir Murugan, Sharif Osman Kabashi Guma, Fareg Saied Ali	PDF 440-442
Increasing the ionic conductivity of solid state polymer electrolyte using fly ash as a filler Yatim Lailun Ni'mah, Muhammad Fajar Taufik, Atetegap Maezah, Fredy Kurniawan	PDF 443-447
Design of microstrip hairpin bandpass filter for 2.9 GHz – 3.1 GHz s-band radar with defected ground structure Nanang Ismail, Teddy Surya Gunawan, Santi Kartika S, Teguh Praludi, Eki A.Z. Hamidi	PDF 448-455
Evaluation of chilling injury and internal browning condition on quality attributes, phenolic content, and antioxidant capacity during sub-optimal cold storage of Malaysian cultivar pineapples Noer Hartini Dolhaji, Ida Idayu Muhamad, Harisun Ya'akub, Azila Abd Aziz	PDF 456-461
Comparison of charantin extract from Momordica Charantia using modified supercritical carbon dioxide and soxhlet extraction method Ahmad Syahmi Zaini, Noor Aiyah Aris, Nicky Rahmana Putra, Syafiza Abd Hashib, Mohd Johari Kamaruddin, Zuhaili Idham, Mohd Azizi Che Yunus	PDF 462-466
Metabolomic profiling of serum in aging mice supplemented with tocotrienol-rich fraction for identification of female reproductive aging biomarkers Norrahiatul Adawiyah Aziz, Fathimah Mohamad, Teh Lay Kek, Nuraliza Abdul Satar	PDF 467-470
Improving forecasting accuracy of crude oil prices using decomposition ensemble model with reconstruction of IMFs based on ARIMA model Muhammad Aamir, Ani Shabri, Muhammad Ishaq	PDF 471-483
Separation of xanthone and vitamin E from Calophyllum inophyllum leaf Hakun Wirawastita Aparamarta, Safrina Hapsari, Reinaldi Ismawan, Violita Angraeni, Arief Widjaja, Tri Widjaja, Yi-Hsu Ju, Setiyo Gunawan	PDF 484-489
Heavy metals in air: Analysis using instrument, air pollution and human health - A review Siti Noor Syuhada Muhammad Amin, Azman Azid, Muhamad Shirwan Abdullah Sani, Ku Mohd Kalkausar Ku Yusof, Mohd Saiful Samsudin, Nurul Latiffah Abd Rani, Saiful Iskandar Khalit	PDF 490-494
Osteoblast adhesion and proliferation on porous chitosan/polycaprolactone scaffolds for bone tissue engineering application Rashid Mad Jin, Naznin Sultana	PDF 495-499
Heat transfer and mapping of THz radiation absorption in biological tissue using Mathematica based Simulink transform Usman Malik, Krisman Krisman, Riad Syech, Muhammad Hamdi	PDF 500-508
The influence of layer thickness on the electrical property of metal-CNT (metal: Cu) composite Salim Mustofa, Patricius Purwanto, Wisnu Ari Adi	PDF 509-511
Increased mitochondrial distribution in early-cleaving embryos indicate successful pre-implantation development Nor Shahida Abdul Rahman, Mimi Sophia Sarbandi, Wan Hafizah Wan Jusof, Zolkapli Eshak, Salina Othman, Fathiah Abdullah, Yuhanita Shafinie Kamsani, Suzanna Daud, Norazilah Mat Jin, Nor Ashikin Mohamed Noor Khan	PDF 512-514
Greater effect of contrast polarities on visual acuity measurements using chart with shorter wavelength background Chen Ai Hong, Nurulain Muhamad	PDF 515-519



MJFAS is the recipient of Current Research in Malaysia (CREAM 2018) Awards by the Ministry of Education Malaysia.



JOURNAL CONTENT

Search

Search Scope

All

Browse

- [By Issue](#)
- [By Author](#)
- [By Title](#)

INFORMATION

- [For Readers](#)
- [For Authors](#)
- [For Librarians](#)

USER

Username

Password

Remember me

FONT SIZE



Home > Vol 15, No 4 (2019)

Malaysian Journal of Fundamental and Applied Sciences



MJFAS promotes and supports I'M Research Consortium



MJFAS is the recipient of Current Research in Malaysia (CREAM 2018) Awards by the Ministry of Education Malaysia.



Journal abbreviation: **Mal. J. Fund. Appl. Sci.**
 ISSN: 2289-5981. e-ISSN: 2289-599X.

Citations	h-index	i10-index
1164	14	23

2019 *Google Scholar* Citation Reports



The Malaysian Journal of Fundamental and Applied Sciences (MJFAS) (formerly known as Journal of Fundamental Sciences (2005-2011), ISSN: 1823-626X) is a refereed research journal published by Penerbit UTM Press, Universiti Teknologi Malaysia. The aims and scope of the journal encompass research articles, original research reports, reviews, short communications and scientific commentaries from fundamental principles to practical applications in the broad field of mathematics, physics, chemistry, and biology. All manuscript submissions must be made through the journal's online manuscript system at [Online Submissions](#). This journal is indexed by [Clarivate Analytics](#) (formerly Thomson Reuters) and [Google Scholar](#).

Starting in the year 2019, MJFAS is published online with a frequency of six (6) issues per year (February, April, June, August, October, and December). Besides that, special issues of MJFAS will be published non-periodically from time to time.

The Malaysian Journal of Fundamental and Applied Sciences (MJFAS) is now entering its fourteen years and it is now the official journal of the [Indonesia-Malaysia Research Consortium](#) (I'M Research Consortium). The consortium's aims are to foster the development of the field through a cross-disciplinary approach and to reach consensus in areas of common interest in fundamental and applied sciences field. By making The Malaysian Journal of Fundamental and Applied Sciences the official journal of the I'M Research Consortium, we hope to provide a forum to bring together society members and to publish peer-reviewed consensus documents that emerge from the activities of the consortium.



The Publishing Process of MJFAS and its Requirements

Below are the new requirements and other guidelines related to publication as well as communicating your results.

JOURNAL CONTENT

Search



About

General Information

Web of Science Coverage

Peer Review Information

[← Return to Search Results](#)

MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCES

ISSN / eISSN **2289-5981 / 2289-599X**

Publisher **PENERBIT UTM PRESS, PENERBIT UTM PRESS, SKUDAI, JOHOR, MALAYSIA, 81310**

About

Malaysian Journal of Fundamental and Applied Sciences is ready to receive papers on all aspects of fundamental science research in the field of mathematics, physics, chemistry and biology.

General Information

Frequency	Quarterly
Issues Per Year	4
Country / Region	MALAYSIA

Web of Science Coverage

[Scope Notes](#)

Web of Science Core Collection

Emerging Sources Citation Index

Categories: Multidisciplinary | Multidisciplinary Sciences

Log into [Web of Science](#) to discover research literature from this journal.

Our policy towards the use of cookies

All Clarivate Analytics websites use cookies to improve your online experience. They were placed on your computer when you launched this website. You can change your cookie settings through your browser.

[Ok to Continue](#)

[Cookie Policy](#)

Publish Report	No
Publons Reviews	21
Publons Endorsements	2
Publons Partner	No

register for a free [Publons](#) account for more peer review information out this journal.

Editorial Disclaimer: As an independent organization, Clarivate Analytics does not become involved in and is not responsible for the editorial management of any journal or the business practices of any publisher. Publishers are accountable for their journal performance and compliance with ethical publishing standards. The views and opinions expressed in any journal are those of the author(s) and do not necessarily reflect the views or opinions of Clarivate Analytics.

Criteria for selection of newly submitted titles and re-evaluation of existing titles in the Web of Science are determined by the Web of Science Editors in their sole discretion. If a publisher's editorial policy or business practices negatively impact the quality of a journal, or its role in the surrounding literature of the subject, the Web of Science Editors may decline to include the journal in any Clarivate Analytics' product or service. The Web of Science Editors, in their sole discretion, may remove titles from coverage at any point if the titles fail to maintain our standard of quality, do not comply with ethical standards, or otherwise do not meet the criteria determined by the Web of Science Editors. If a journal is deselected or removed from coverage, the journal will cease to be indexed in the Web of Science from a date determined by the Web of Science Editors in their sole discretion – articles published after that date will not be indexed. The Web of Science Editors' decision on all matters relating to journal coverage will be final.

Clarivate

Accelerating innovation

© 2019 Clarivate

[Copyright Notice](#)

[Terms of Use](#)

[Privacy Notice](#)

[Cookie Policy](#)

[Help Center](#)

Follow us:



Our policy towards the use of cookies

All Clarivate Analytics websites use cookies to improve your online experience. They were placed on your computer when you launched this website. You can change your cookie settings through your browser.

[Ok to Continue](#)

[Cookie Policy](#)

Synthesis and characterization of zeolite NaX from Bangka Belitung Kaolin as alternative precursor

Vita Nur Iftitahiyah^a, Didik Prasetyoko^{a, *}, Hadi Nur^b, Hasliza Bahruji^c, Hartati^d

^a Department of Chemistry, Faculty of science, Institut Teknologi Sepuluh Nopember (ITS), Kampus ITS Sukolilo, Surabaya 60111, Indonesia

^b Ibnu Sina Institute for Scientific and Industrial Research, Universiti Teknologi Malaysia, 81310 UTM Skudai, Johor, Malaysia

^c Cardiff Catalysis Institute, Cardiff University, CF10 3AT Cardiff United Kingdom

^d Department of Chemistry, Faculty of Science and Technology, Universitas Airlangga, Kampus UNAIR, Surabaya 60115, Indonesia

* Corresponding author: didikp@chem.its.ac.id

Article history

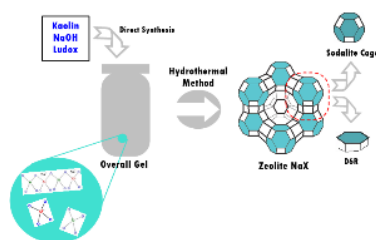
Submitted 5 January 2018

Revised 30 January 2018

Accepted 13 March 2018

Published Online 3 December 2018

Graphical abstract



Abstract

The potential use of kaolin as silica and alumina precursor for the synthesis of zeolite NaX was investigated in this study. The synthesis involved three steps of reactions; the preparation of seed gel, the formation of feedstock gel using kaolin and the combination of overall gel followed by hydrothermal treatment at 105°C for 12 hours. Analysis using X-ray Diffraction (XRD) method indicated the transformation of kaolin into pure phase zeolite NaX with a small amount of kaolin was still visible. Detail microscopic analysis showed the morphology of zeolite X consisted of octahedral particles with a crystallite diameter of 20-30 µm. Analysis of surface acidity using pyridine as probe molecule indicated the zeolite X has high Brønsted acidity with 0.181 mmol/g of acid sites, significantly higher than Lewis acidity ~0.053 mmol/g. The N₂ adsorption-desorption measurement indicated a type IV material with both microporous and mesoporous structures with an average pore size of 1.47 nm for micropore and 3.41 nm for mesoporous.

Keywords: Kaolin, zeolite NaX, porous materials, hydrothermal method

© 2018 Penerbit UTM Press. All rights reserved

INTRODUCTION

Zeolite consists of four connected AlO₄ and SiO₄ tetrahedrons connected via oxygen to form porous aluminosilicates framework. The intracrystalline channel in the zeolite framework is occupied with water molecule and cation to neutralise the negative charge of AlO₄. The mobility of the cation and the flexibility of the size, shape and pore structure of the zeolite allows modification to accommodate catalytic desire. Zeolite is commonly produced from hydrogels aluminate and sodium silicate, however, production of zeolite from alternative silica and alumina source such as bagasse fly ash [1] [2], rice hush ash [3] and kaolin [4] have received considerable attention since the past few decades. Kaolin is naturally occurring minerals that abundantly available in Indonesia particularly in south Sumatra, Bangka Belitung and Java island. Kaolin from Bangka Belitung has low iron and titanium content but rich with silica (54.9 wt.%) and alumina (36 wt.%). Studies were previously carried out for the synthesis of zeolite X using kaolin originated from Bulgaria [5] and also the synthesis of zeolite Y from natural kaolin [6], kaolin obtained from China [7] and Iran [8]. Kaolin is an ideal alternative candidate to replace commercial silica and alumina precursors due to a high level of silica and alumina with relatively low iron content. The use of kaolin as raw material for the synthesis of zeolite offers an alternative economical route by utilising naturally abundant resources rather than commercially available chemical.

The aim to synthesis zeolite NaX is due to its unique three-dimensional pore structure and surface acidity that can be utilised as a solid acid catalyst [9], ion exchanger [10], and adsorbent. Zeolite NaX belongs to faujasite family that composes of sodalite cage with 6-rings (D6R) to form hexagonal framework.

Studies that were carried out on the synthesis of zeolite X from kaolin required acid or base leaching treatment to reduce its resistance

towards chemical transformation to zeolite [11]. Kaolin also required thermal treatment at high temperature ~ 700–900°C to form amorphous metakaolin before it can be used for zeolite synthesize [12]. The additional pre-treatment process has an indirect effect on the production cost and also detrimental to the environment i.e., production of greenhouse gases.

Here we investigate the potential of kaolin as alternative silica and alumina precursors without the need for pre-treatment to produce zeolite NaX via hydrothermal method. The physical properties of zeolite X were analysed using XRD, SEM, FTIR and N₂-gas adsorption-desorption methods to obtain its crystal morphology, framework structure, pore structure and surface acidity.

EXPERIMENTAL

Materials

Kaolin from Bangka Belitung contained 36 wt.% Al₂O₃, 54.9 wt.% SiO₂, 3.34 wt.% Fe₂O₃ and 1.88 wt.% K₂O. The materials used in the synthesis were sodium hydroxide (99 wt.% NaOH Merck), sodium aluminate (53 wt.% NaAlO₂ Merck), silica colloidal (LUDOX) (30 wt.% SiO₂ and 70 wt.% H₂O Merck), ammonium acetate (Merck) for ion exchanged and acidity characterization. Demineralized water was used for cleaning and chemical preparation.

Synthesis of zeolite NaX

Bangka Belitung kaolin used in this study was obtained from Bangka Belitung (Sumatra, Indonesia). Chemical and mineralogical composition of the received kaolin is listed in Table 1. The as-received kaolin contains a low level of metal oxide impurities with Si to Al molar ratio of 1.53. Kaolin was used for the synthesis of zeolite X without prior pretreatment. The mixture kaolin and other precursors were

dissolved in NaOH solution to produce amorphous Si and Al gel mixture which was then used for the hydrothermal synthesis of zeolite X.

The synthesis of zeolite NaX was carried out following three steps; the preparation of seed gel, the production feedstock gel and the mixing of seed and feedstock gels to produce an overall gel. The seed gel (Al₂O₃.4SiO₂) was prepared by the addition of the NaAlO₂ dan silica colloidal mixture into NaOH solution. The mixture was stirred continuously to form a homogeneous mixture and was left to age for 24 h at room temperature. The feedstock gel (Al₂O₃.4.3SiO₂) was also prepared following similar procedures but sodium aluminate was replaced by kaolin. The resulting feedstock gel was then added to the seed gel with the ratio of 18:1 in order to get overall synthesis gel mixture. The gel mixture was left to age for 24 h at room temperature before transferred into Teflon line vessel and hydrothermally treated at 105°C for 12h. The simplified synthesis process was illustrated in the schematic diagram in Fig.1.

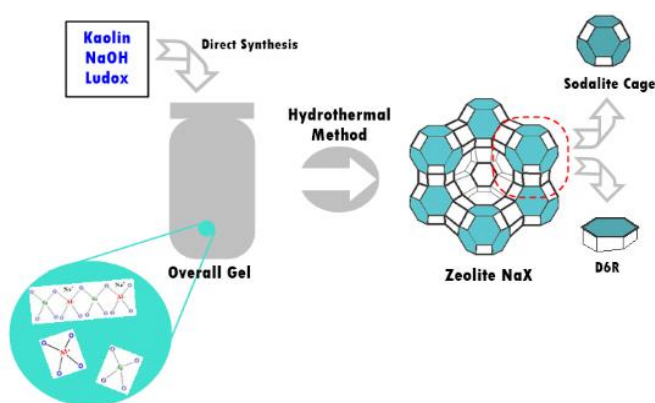


Fig. 1 The schematic diagram for the synthesis of zeolite NaX from kaolin.

The resulting powder was washed thoroughly with distilled water until the pH of the supernatant was neutral. The powder was then dried and calcined under an air flow at 500°C for 1h at 2°/min ramp rate. The synthesised zeolite NaX was ion-exchanged with NH₄⁺ by mixing the resulting powder with ammonium acetate to form NH₄X, as shown in the following equation (1)(2).



The NH₄X was then calcined at 550°C for 5 hours to form H-zeolite X framework.

Characterization of synthesized zeolite NaX

X-ray Fluorescence was used to obtain chemical composition of Kaolin. The synthesized NaX powder and kaolin were also characterised using X-ray Diffraction (XRD) Philip Expert with CuKα (λ = 1.5405 Å) radiation to determine the crystalline phase and crystallinity. Data were recorded in the range of 5–40° with a step size of 0.02°. The infrared spectra of NaX (SiO₂/Al₂O₃ ratio = 4) was recorded using Fourier Transform Infrared spectrophotometer (8400S Shimadzu) with KBr pellet method. Sample and KBr were mixed with the ratio of 1:99, crushed and molded into a pellet, then compressed using hydraulic pressure. The formed pellet was placed on the holder and recorded in 4000-400 cm⁻¹.

The morphology of the synthesis zeolite X was analysed using Scanning Electron Microscopy (SEM) ZEIS EVO MA 10 FT-IR. Prior to the analysis, the sample was placed on a carbon tape and coated using Pd / Au for 15 minutes at 6 x 10⁻² mBar pressure, then scanned to analyse the sample morphology.

Nitrogen adsorption-desorption isotherms were observed using the Quantachrome Corporation (Nova-1200) instrument. Prior to the analysis, 0.2 gram of sample was placed in the vacuum for 3 hours at

300 °C to evacuate adsorbed water, the sample was then exposed to nitrogen gas at 77 K. The specific surface area (S_{BET}) is calculated using BET (Brunauer-Emmet-Teller) equation. The total pore volume was obtained based on the amount of nitrogen adsorbed at P/P₀ 0-0.99, while the pore size distribution was analyzed using BJH (Barret-Joiner-Halenda). The micropore size distribution was analysed using SF (Saito-Foley) method.

Pyridine was used as a probe molecule for acidity studies. The sample was weight ~10 mg and pressed to form a thin and transparent film. The film was then placed in the sealed sample holder and annealed in a tubular furnace under vacuum at 300 °C for 3 hours. The film was cooled at 30 °C before exposed with pyridine. The temperature was increased to 150 °C to remove physisorbed pyridine on the surface. The chemical adsorption of pyridine on the zeolite X was measured using FTIR and the adsorbed pyridine peak was determined using the Gaussian method.

RESULTS AND DISCUSSION

Characterisation of zeolite NaX

Detail chemical composition of kaolin obtained from XRF analysis was summarised in Table 1. Kaolin predominantly consisted of SiO₂ and Al₂O₃ at ~ 91 wt. % with the impurities consisted of K₂O, Fe₂O₃, and traces of metal oxides. Considering the low level of metal oxide impurities, kaolin was used without prior pre-treatment for the synthesis of zeolite X. We provide EDX analysis of the as-synthesised zeolite X that indicated the NaX consisted of siliconium and oxygen as main elements for zeolite framework with sodium that presumably as stabilizer cation (Fig. 2). We imply that metal oxide impurities that were originally present in kaolin were successfully evacuated from the reaction mixture. The evacuation occurred during the dissolution of kaolin with sodium hydroxide followed by thorough washing with deionised water and air calcination at high temperature.

Table 1 Minerals analysis by XRF for Kaolin.

Minerals	Wt. %
Al ₂ O ₃	36
SiO ₂	54.9
P ₂ O ₅	0.88
K ₂ O	2.88
CaO	0.58
TiO ₂	0.551
V ₂ O ₅	0.03
Fe ₂ O ₃	3.37
CuO	0.097
Rb ₂ O	0.18
ZnO	0.02
NiO	0.585

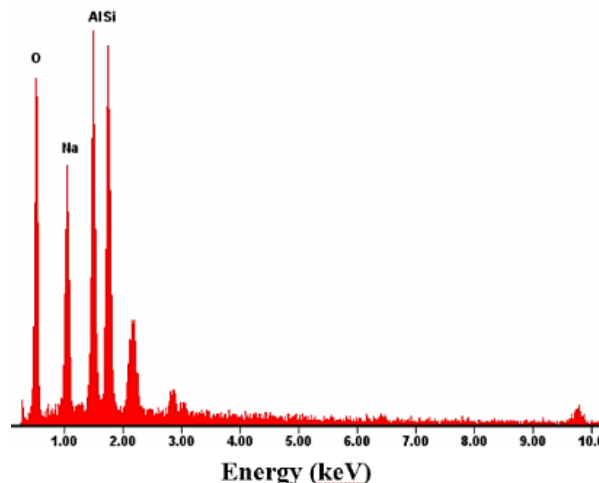


Fig. 2 EDX analysis on the elemental composition of synthesized NaX

In order to synthesis zeolite NaX with high crystallinity and purity, the synthesis condition should be carefully controlled including the purity of the precursors. The hydrothermal treatment requires optimisation of synthesis condition by controlling the temperature and the crystallisation time. Variation of the molar ratio of starting materials significantly determined morphology, surface area and acidity of the synthesized zeolite. Davis [13] reported that controlling the temperature of hydrothermal treatment affected the nucleation and crystal growth of zeolite. In general, hydrothermal synthesis at high temperature produced high thermal energy that consequently shortened the crystallization time. However, synthesis at high temperature often produced large aggregates of zeolite. We found the optimum crystallization temperature for the synthesis of zeolite NaX from kaolin was achieved at 105 °C. Detail analysis of the morphological structure and the purity of the synthesized zeolite was analysed using XRD and infrared spectroscopy.

X-ray diffractogram of kaolin, synthesised zeolite NaX and commercial zeolite NaX were shown in Fig 3. The peaks corresponded to kaolin at 20°, 20.5°, 22°, 35°, 36°, 39° and 40° were significantly reduced in the as-synthesised zeolite NaX. The diffractogram pattern of zeolite NaX synthesised using kaolin showed peaks at $2\theta = 6.15; 10.02; 23.28; 26.64; 31.95^\circ$ which were identical to the peaks appeared in standard zeolite X [13]. The XRD pattern of synthesized zeolite NaX was also in accordance with the peaks of NaX according to International Zeolite Association (IZA)[14]. However, two peaks corresponded to kaolin at 13° and 25° were still present but at significantly low intensity in zeolite NaX which suggested incomplete dissolution of kaolin with sodium hydroxide during the preparation of gel mixture.

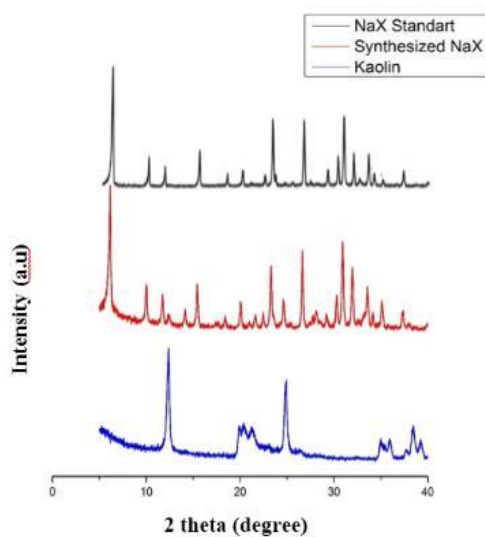


Fig. 3 The XRD pattern of NaX commercial standard, synthesize NaX and kaolin.

The synthesized zeolite NaX was also analysed using infrared spectroscopy to provide spectroscopic evidence on the structure and the functional group of zeolite X. The IR spectra of zeolite X was also compared to kaolin as shown in Fig.4. The presence of a band at 1107 cm^{-1} in the infrared analysis of kaolin indicated the stretching vibration of tetrahedral SiO_4 and AlO_4 . The peak appeared at 1029 cm^{-1} is corresponded to the vibration of Si-O-Si (Si-O-Si in-plane stretching). The vibration of Al-O-H appeared at 913 cm^{-1} which subsequently disappeared in NaX spectra. The characteristic bands of NaX which consisted of FAU type structure occurred between 1250-950 cm^{-1} [15] which showed external asymmetric stretching of TO_4 , where T is Si or Al. The band appeared at 790-650 cm^{-1} is external vibration associated with the sensitive tetrahedral structure of zeolite X (external symmetric vibration). This is a typical vibration that appears in most silica materials [16]. Asymmetric stretching and symmetric stretching of synthesized NaX can be seen occurred at 977.94 cm^{-1} and 744.55 cm^{-1} . The specific TO_4 (T = Si or Al) vibrational peak for zeolite was identified by band appeared at 451 cm^{-1} . The peak observed at 561 cm^{-1}

was identified as vibrational of D6R rings that distinguished zeolite X structure with another aluminosilicate zeolite. The spectroscopic evidence obtained from the infrared analysis further consolidate the formation of zeolite X crystal structure that was synthesised using that kaolin as silica and alumina precursor.

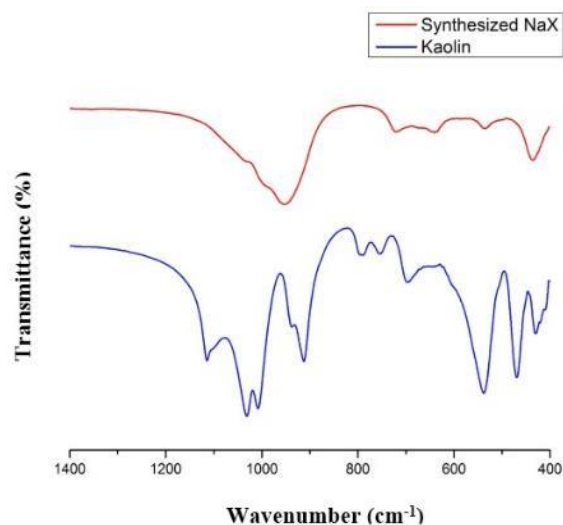


Fig. 4 The Spectra FTIR of synthesized NaX and kaolin.

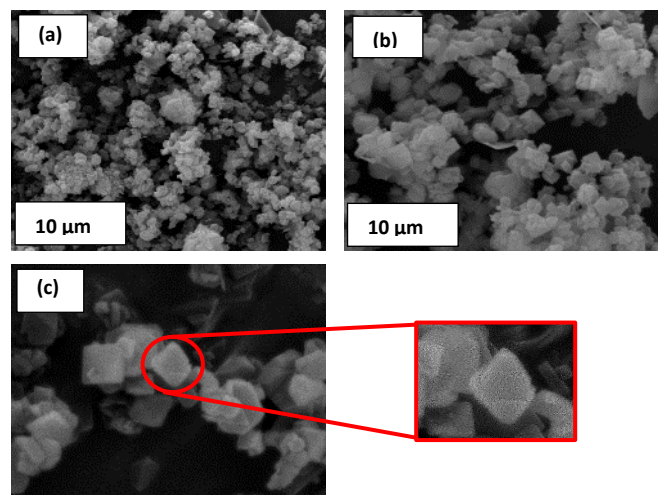


Fig. 5 The SEM micrograph of synthesized NaX.

The morphology and crystallite size of the synthesised material was analysed using Scanning Electron Microscopy (SEM). Fig. 5 shows the morphology of NaX which appears as a hexagonal structure with average crystallite size ~ 10-20 μm . This is a typical morphology for zeolite X which were observed previously by others [17] [18]. SEM images also show the NaX crystallite was in uniform aggregate size and some crystallite agglomerates to form large aggregates. The presence of remaining kaolin as a result of incomplete dissolution which was indicated by XRD analysis was observed as a thin layer occurs around the NaX crystallite. The incomplete dissolution of kaolin also affected the Si/Al ratios of the synthesised zeolite X. The EDX analysis shown Fig.2 indicated that the zeolite X was consisted of 23 wt % of Si and 18 % of Al to give the Si/Al ratio of 1.28. This is significantly lower than the experimental value which also suggested incomplete dissolution of kaolin during the synthesis.

The specific surface area of kaolin and zeolite X were analysed using BET method with the nitrogen adsorption-desorption profile provides information on the type of pore of zeolite (Fig. 6). Kaolin showed no nitrogen adsorption at relative pressure, P/P_0 within 0.1-0.3 which indicated the type II non-porous material. The non-porous kaolin also has a very low surface area of ~ 14 m^2/g . The transformation of

kaolin to zeolite X resulting in a high surface area (80 m²/g) material that is beneficial for research and industrial purposes. The as-synthesised zeolite HX showed an isotherm type IV which indicated the presence of mesoporous structure within the framework.

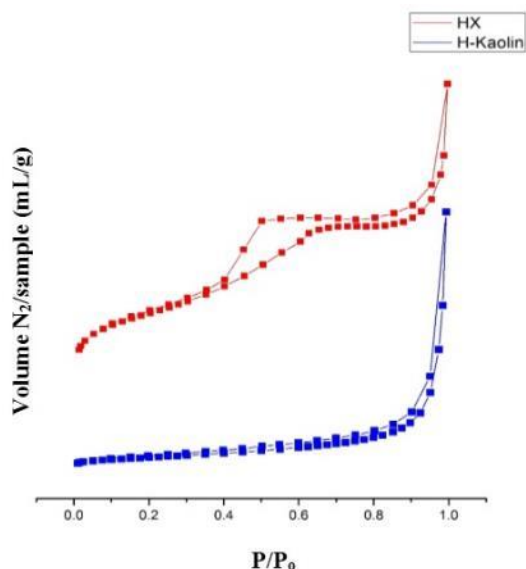


Fig. 6 The Isotherm N₂ Adsorption-Desorption of HX and H-kaolin.

The isotherm N₂ profile of HX exhibited an increasing nitrogen molecule adsorption at low relative pressure (0 to 0.3) corresponding to the adsorption occurred to fill the micropores of zeolite. At P/P₀ of 0.3, the surface of zeolite X was covered by a monolayer nitrogen molecule. Adsorption of nitrogen to the zeolite surface was continuously increased at P/P₀ ~ 0.4-0.7, which indicated the nitrogen adsorption on the uniform slit-shaped intracrystal mesopore. The surface of the pore only allowed a limited layer of adsorbate, its called condensation and causing the hysteresis loop. Hysteresis loop was observed at P/P₀ 0.4-1, occurred due to the desorption of nitrogen.

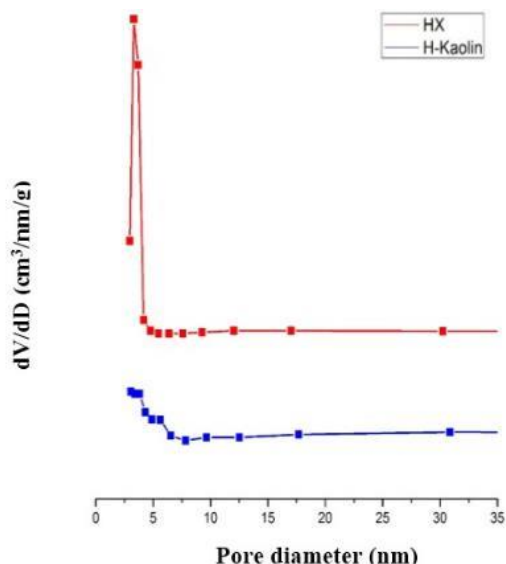


Fig. 7 The pore size distribution of kaolin and synthesized HX from BJH (Ballet, Joiner, Halenda) method.

Analysis of the pore size distribution using the BJH (Barret, Joiner, Halenda) method in Fig. 7 showed the presence of meso-sized pore type structure ~ 2-5 nm in diameter with a peak centered at 3.4 nm. It implies that the zeolite NaX consisted of the intra-crystal mesoporous structure with narrow and sharp pore distribution. We also compare the pore size distribution of zeolite NaX with the raw material kaolin that showed no significant pore structure can be observed in kaolin. This is in

agreement with the N₂ adsorption-desorption analysis that revealed type II isotherm corresponded to the non-porous structure of kaolin. The formation of mesopores within the zeolite NaX structure is rather surprising due to in general the synthesis of mesoporous zeolite requires the presence of surfactant as structure directing agent. We suggested the formation of mesoporous structure is due to the arrangement of silica and alumina in the slit-pore of zeolite NaX. Apart from the presence of mesopores, analysis of zeolite NaX SF method as shown in Fig. 8 also indicated the presence of the microporous structure. The micropore size distribution showed the highest pore diameter of ~ 1.47 nm with a micropore volume of ~ 0.006 cm³/g contrary to kaolin that only consisted of relatively small micropore size within 0.36 and 0.90 nm of diameter.

The surface acidity was performed by infrared spectroscopy using pyridine as a probe molecule. The amount of adsorbed pyridine on the zeolite NaX was analysed in the 1700-1400 cm⁻¹ of wavelength region. Protonated pyridine molecule in the form of pyridium ion (C₅H₅NH⁺) interacting with Brønsted acid sites shows a specific adsorption band at a wavenumber of ~ 1540-1545 cm⁻¹. The pyridinium ion is a result of bond formation between pyridine with a proton from the surface hydroxyl group of zeolite X. Interaction between pyridine with Lewis acid site on the surface formed by coordinated bonding interaction between the free electron pairs of pyridine molecule with the empty orbital of extra-framework alumina in the zeolite. This interaction leads to the appearance of the absorption band at wavenumber ~1440-1452 cm⁻¹ [19].

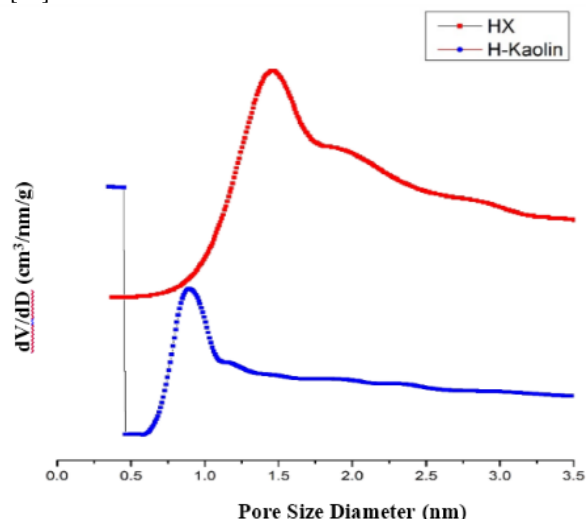


Fig. 8 The micropore distribution of as-synthesized HX from the SF method

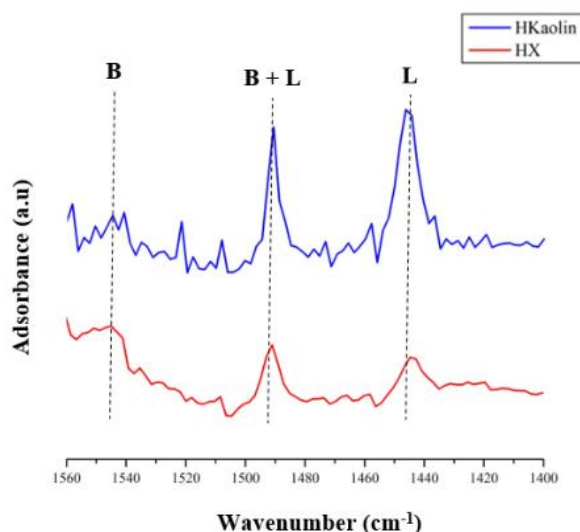


Fig. 9 The infrared spectra of pyridine adsorption of synthesized HX

The infrared spectra of the pyridine upon adsorption on zeolite HX showed pyridine peak vibration appeared ~ 1440-1452 cm^{-1} indicating the presence of Lewis acidity. The band appeared at 1545 cm^{-1} revealed Brønsted acidity and the band at 1490 cm^{-1} corresponded to the total adsorption of pyridine on both Lewis and Brønsted acid sites [20]. The peak area of adsorbed pyridine was determined using the Gaussian method to indicate the amount of surface acid site on the as-synthesised zeolite X. The calculated data showed the zeolite X has a high number of Brønsted acidity in comparison to Lewis acidity to give about ~0.18 mmol/g of the Brønsted acid site and 0.05 mmol/g of Lewis acid site.

CONCLUSION

We investigated the potential use of abundantly available silica and alumina riched kaolin to be transformed into zeolite X. The synthesis occurs in three steps involving the preparation of seed gel, the formation of feedstock gel using kaolin and the mixture of these two gels to form the overall gel. The crystallization process was carried out under a hydrothermal condition at 105°C which produced zeolite NaX with both microporous and mesoporous structure. The zeolite NaX also consisted of high purity and crystallinity with high surface acidity which ideal as a catalyst in many catalytic applications.

ACKNOWLEDGEMENT

The authors would like to acknowledge the Ministry of Research and Higher Education, Indonesia, under “PBK” Research 2017-2019 with contract number of 525/PKS/ITS/2017.

REFERENCES

- [1] S. Chandrasekhar, P.N. Pramada, Investigation on Synthesis of Zeolite NaX from Kerala Kaolin, *Journal of Porous Materials* (2004) 6:283-297.
- [2] Y. Liu, C. Yan, X. Qiu, D. Li, H. Wang, A. Alshameri, Preparation of Faujasite Block from Fly Ash-Based Geopolymer via in-Situ Hydrothermal Method, *Journal of the Taiwan Institute of Chemical Engineers* 433-39 (2016) 59.
- [3] R.M. Mohamed, I.A. Mkhaldid, M.A. Barakat, Rice husk ash as a renewable source for the production of zeolite NaY and its characterization, *Arabian Journal of Chemistry* 48-53 (2012) 8.
- [4] J.-Q. Wang, Y.-X. Huang, Y. Pan, Hydrothermal Synthesis of High Purity Zeolite A and X from Natural Kaolin without Calcination *Microporous and Mesoporous Materials* 50-56 (2014) 199.
- [5] C. Belviso, F. Cavalcante, A. Lettino, S. Fiore, A and X-Type Zeolites Synthesised from Kaolinite at Low Temperature, *Applied Clay Science* 162-68 (2013) 80-81.
- [6] J-Q. Wang, Y. Pan, New Hydrothermal Route for the Synthesis of High Purity Nanoparticles of Zeolite X from Kaolin and Quartz, *Microporous and Mesoporous Material* 77-85 (2016) 232.
- [7] Y. Ma, C. Yan, A. Alshameri, X. Qiu, D. Li, C. Zhou, Synthesis and Characterization of 13X Zeolite from Low-Grade Natural Kaolin, *Advanced Powder Technology* 495-99 (2014) 25.
- [8] V. Garshasbi, M. Jahangiri, M. Anbia, Equilibrium CO₂ Adsorption on Zeolite 13X Prepared from Natural Clays, *Applied Surface Science* 225-33 (2017) 393.
- [9] K.-H. Chung, D.-R. Chang, B.-G. Park, Removal of Free Fatty Acid in Waste Frying Oil by Esterification with Methanol on Zeolite Catalysts, *Bioresourc Technology* 38-43 (2008) 99.
- [10] K.M. Roghayeh, S.A. Fakhry, Study on the Thermal Behavior of Low Silica X-type Zeolite Ion Exchanged with Alkaline earth Cation, *Microporous and Mesoporous Material* 285-293 (2009) 120.
- [11] Z. Xu, T. DaQing, Z. JingJing, L. XingYang, Synthesis of NaX Zeolite at Room Temperature and Its Characterization, *Material Letters* 80-83 (2013) 104.
- [12] W. Jin-Quan, H. Ya-Xi, P. Yuanming, X. Jin, Hydrothermal Synthesis of High Purity Zeolite A from Natural Kaolin without Calcination, *Microporous and Mesoporous Material* 50-56 (2014) 199.
- [13] M. Davis, Zeolite and Molecular Sieve Synthesis, *Chemistry Material* 756-768 (1992) 4.
- [14] C.W. Purnomo, C. Salim, H. Hinode, Synthesis of Pure Na-X and Na-A Zeolite from Bagasse Fly Ash, *Microporous and Mesoporous Materials*, 6-13 (2012) 162.
- [15] W. Mozgawa, M. Krol, Barczyk, FTIR Studies of Zeolites from Different Structural Groups, *CHEMIK* 667-674 (2011) 65.
- [16] D. Chen, X. Hu, L. Shi, Q. Cui, H. Wang, H. Yao, Synthesis and Characterization of Zeolite X from Lithium Slag, *Applied Clay Science* 148-51 (2012) 59-60.
- [17] J.M. Gómez, E. Diez, I. Bernabé, Deoxygenation of M-Toluic Acid over Hierarchical X Zeolite, *Catalysis Communications* 55-58 (2016) 78.
- [18] H.J. Lee, Y.M. Kim, O.S. Kweon, I.J. Kim, Structural and Morphological Transformation of NaX Zeolite Crystals at High Temperature, *Journal of the European Ceramic Society* 561-64 (2007) 27.
- [19] K.A. Layman, M.M. Ivey, J.C. Hiemminger, Pyridin Adsorption and Acid/Base Complex Formation on Ultrathin Films of $\gamma\text{-Al}_2\text{O}_3$ on NiAl (100), *Journal Physic Chemistry* 8538-8546 (2003) 107.
- [20] S. Bendenia, I. Batonneu-Gener, J. Camparot, K. Marouf Khelifa, H. Hammoudi, A. Khelifa, Acidity Study of X Zeolites Modified by Nickel And/or Chromium Cations in the Case of Binary and Ternary Exchanges, *Microporous and Mesoporous Materials* 111-18 (2012) 159.