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John Regazzi, Chair

John Regazzi's career in the digital information industry is distinguished by a series of industry breakthroughs. This tech pioneer was the driving force behind the first commercially available CDROM text database and first professional web community for engineers and was the first Microsoft CDROM conference keynote address speaker.

An expert in information sciences and service, John is the former CEO of Ei Inc. After its purchase by Reed Elsevier, Regazzi was named CEO of Elsevier Inc., responsible for designing and marketing ground-breaking net-based services for the professional scientific, technical, and medical communities, including ScienceDirect, Scirus, Scopus, Engineering Village, and Medical Consult.

The Dean of the College of Information and Computer Science of Long Island University (LIU) from 2006 to 2008, John currently serves as Professor and Director of the Scholarly Communications and Information Innovation Lab at LIU. He was also recently appointed by the US Department of Commerce Secretary as Chairman of the Board of Advisors, National Technical Information Service (NTIS), a division of the Commerce Department. John also serves as Chairman of the boards of Research Solutions (OTC: RSSS), as well as Inflexxion and LawLogix, two of Akoya Capital Partners portfolio companies.

John S. Haynes, Chief Executive Officer, AIP Publishing

John Haynes received a Ph.D. in Chemistry from the University of British Columbia, followed by postdoctoral research at the University of Oxford. After two decades in the STM publishing industry, including senior positions with the Institute of Physics Publishing and Royal Society of Chemistry, John joined the American Institute of Physics in 2009 as Vice President, Publishing. In 2013, he was appointed Chief Executive Officer of the newly established AIP Publishing.

Alan Singleton, Secretary

Alan Singleton originally qualified in Physics from the University of Oxford. After several years in the electronics industry, he earned a Masters in Information Science and went on to the Institute of Physics (IoP) on a grant studying communication in physics, then worked as a Commissioning Editor at Elsevier and a Research Fellow in research communications at Leicester University. From 1985-98, Alan held increasingly responsible positions at IoP Publishing, beginning as a Research Officer and culminating his tenure there as Journals Director. He was science, medicine (books) and electronic Publishing Director at Oxford University Press for three years and, in 2001, became Managing Director of the publishing arm of the Institution of Mechanical Engineers in the UK. Since 2009, he has been active as a consultant to the scholarly publishing industry, primarily managing and assisting bid processes for learned societies seeking a journal publisher. He was Editor-in-Chief of the journal Learned Publishing until the end of 2014.

David K. Campbell

David K. Campbell received his B.A. in Chemistry and Physics from Harvard University in 1966, and after a period as a Marshall Scholar, his Ph.D. from Cambridge University in Theoretical Physics and Applied Mathematics in 1970. He held postdoctoral positions at the University of Illinois Urbana-Champaign (UIUC) (1970-72) and the Institute for Advanced Study in Princeton (1972-74) before joining Los Alamos National Laboratory in 1974 as the first J. Robert Oppenheimer Fellow. At Los Alamos, David co-founded and later directed the Center for Nonlinear Studies.

In 1992, David became Professor and Head of the Department of Physics at UIUC. In 2000, he moved to Boston University (BU), where he served as Dean of the College of Engineering from 2000-2005 and as University Provost from 2005-2011. He is currently Professor of Physics and Electrical and Computer Engineering and Materials Science and Engineering at BU. An international leader in the field of "nonlinear science," David received the American Physical Society's 2010 Julius Edgar Lilienfeld Prize for his research and scholarly contributions. He is the founding Editor-in-Chief of the AIP journal Chaos: An Interdisciplinary Journal of Nonlinear Science, a Fellow of the APS and AAAS, and is Past-Co-Chair of the Science Board of the Santa Fe Institute.

Wolfgang Christian

Wolfgang Christian is the emeritus Brown Professor of Physics at Davidson College where he taught for 33 years. He is currently serving as the elected national Secretary of the American Association of Physics Teachers. Wolfgang is a fellow of the American Physical Society and the American Association of Physics Teachers. He is the author or co-author of nine books including: Open Source Physics: A User's Guide with Examples (Addison Wesley 2006), An Introduction to Computer Simulation Methods: Applications to Physical System (Addison Wesley 2006), Physlet Quantum Physics (Prentice Hall 2005), Physlet Physics (Prentice Hall 2004), Physlets: Teaching Physics with Interactive Curricular Material (Prentice Hall, 2001), Just-in-Time Teaching (Prentice Hall, 1999). He has been the books editor of the American Physical Society (APS) journal Computers in Physics. Wolfgang served as the co-chair of the 2008 Gordon Research Conference on Physics Research and Education. His current research is in computational physics and in internet-based interactive curriculum development.

Judith Flippen-Anderson

Judith Flippen-Anderson spent 35 years as a small molecule crystallographer at the Naval Research Laboratory (NRL) in Washington, DC. After retiring from NRL, she took a quantum leap in molecular weight and accepted a position with the Protein Data Bank (2003 – 2015).

For a number of years, Judith served as the American Crystallographic Association (ACA) representative to the AIP Executive Committee and Governing Board. In November of 2013, she was elected as the AIP Corporate Secretary by the AIP Governing Board. In cooperation with AIP Publishing, she is helping to ensure that the ACA journal Structural Dynamics, launched in 2013, will be a success.

Judith is Co-Editor of ACA RefleXions and a former Editor of the International Union of Crystallography newsletter. She is also a member of the ACA meetings planning and finance committees, Past-Chair of the US National Committee for Crystallography, Past-President of the ACA, and was among the first class of ACA Fellows.

Susan E. Fox

Susan E. Fox is Executive Director of the Acoustical Society of America. A Fellow of the

American Society of Association Executives, Susan earned her M.S. in Public Affairs from the McCormack Graduate School of Policy and Global Studies, University of Massachusetts, Boston. She specializes in institutional change, governance, strategic positioning, and organizational development. Previously she served as Executive Director of the Arctic Research Consortium of the U.S., the American Association of Law Libraries, the Society of American Archivists, and as Director of Programs at the Harvard Kennedy School Belfer Center for Science and International Affairs.

Gerald Fuller

Gerald Fuller is the Fletcher Jones Professor of Chemical Engineering at Stanford University. He joined Stanford in 1980 following his graduate work at Caltech where he acquired his MS and PhD degrees. His undergraduate education was obtained at the University of Calgary, Canada. Professor Fuller's interests lie in studies of rheology and interfacial fluid mechanics. His work has been recognized by receipt of the Bingham Medal of The Society of Rheology, membership in the National Academy of Engineering, election to the American Academy of Arts and Science, and honorary doctorates from the Universities of Crete, Greece, and Leuven, Belgium.

Alan Jeffrey Giacomin

Editor-in-Chief of the AIPP journal Physics of Fluids, Jeffrey Giacomin holds the NSERC Tier 1 Canada Research Chair in Rheology and is Professor of Chemical Engineering and of Mechanical & Materials Engineering at Queens University at Kingston. Jeffrey is President of the Canadian Society of Rheology, former President of The Society of Rheology and former Associate Editor for Business of the Journal of Rheology, the archival journal of The Society of Rheology. For nearly 20 years, Jeffrey directed the Rheology Research Center of the University of Wisconsin-Madison. He has been named Professor of the French Academy of Sciences and he holds the title of Honorary Associate Member of the Institute of Non-Newtonian Fluid Mechanics in Wales.

Marsha I. Lester

Marsha I. Lester received her Ph.D. from Columbia University in 1981. She has risen through the academic ranks at the University of Pennsylvania, where she is currently the Edmund J. Kahn Distinguished Professor in the Department of Chemistry of the School of Arts & Sciences. She completed a four-year term as Chair of the Department of Chemistry in 2009.

Marsha has published extensively in a broad range of scholarly journals in the physical sciences. She has received many honors and awards, including her election to fellowship in the American Academy of Arts & Sciences, the Garvan-Olin Medal of the American Chemical Society, the Bourke Lectureship of the Faraday Division of the Royal Society of Chemistry, a John Simon Guggenheim Memorial Foundation Fellowship, Fellow of the American Association for the Advancement of Science, the American Chemical Society, and the American Physical Society, an Alfred P. Sloan Research Fellowship, and the Dreyfus Teacher-Scholar Award. In late 2008, Marsha was appointed Editor-in-Chief of The Journal of Chemical Physics, the preeminent journal in her field.

Ivan Petrov

Ivan Petrov is Principal Research Scientist at the Frederick Seitz Materials Research Laboratory, Adjunct Professor of Materials Science, and 1998-2010 Director of the Center for Microanalysis of Materials at the University of Illinois at Urbana-Champaign. He has

been Professor of Physics at Linköping University, Sweden since 2010 and was Visiting Professor of Surface Engineering at Sheffield Hallam University, UK from 2000-2012.

Ivan earned his Ph.D. in Physics from the Institute of Electronics, Bulgarian Academy of Sciences and received the Doctor Honoris Causa degree from Linköping University. He has published 270+ refereed papers cited over 10,500 times. Ivan is an Associate Editor of Surface Science Spectra and Surface and Coatings Technology.

A Fellow of the AVS, Ivan currently serves as the 2015 AVS President. He received the 2009 Bunshah Award and Honorary Lecture from the Advanced Surface Engineering Division of AVS and the 2013 AVS John A. Thornton Memorial Award/Lecture. He served as AVS Publication Chair from 2010-2014 and has been elected as the Chair of the Surface Engineering Division of the International Union of Vacuum Science Technology and Application from 2009-2017.

Greg Tananbaum

Greg Tananbaum serves as a consultant to publishers, libraries, universities, and information providers as owner of ScholarNext. Clients include Microsoft, SPARC, PLOS, AIP, the University of California, and Annual Reviews. He has been President of The Berkeley Electronic Press, as well as Director of Product Marketing for EndNote.

Greg writes a regular column in Against the Grain covering emerging developments in the field of scholarly communication. He has been an invited speaker at dozens of conferences, including the American Library Association, the Society for Scholarly Publishing, the Association of Professional and Learned Society Publishers, and Online Information UK. Greg holds a Master's degree from the London School of Economics and a B.A. from Yale University.

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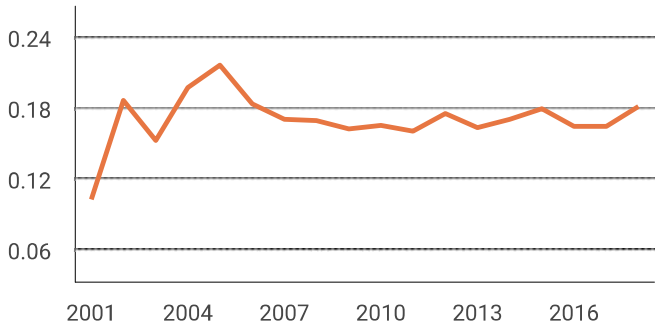

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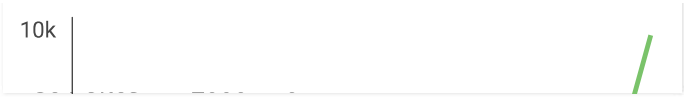

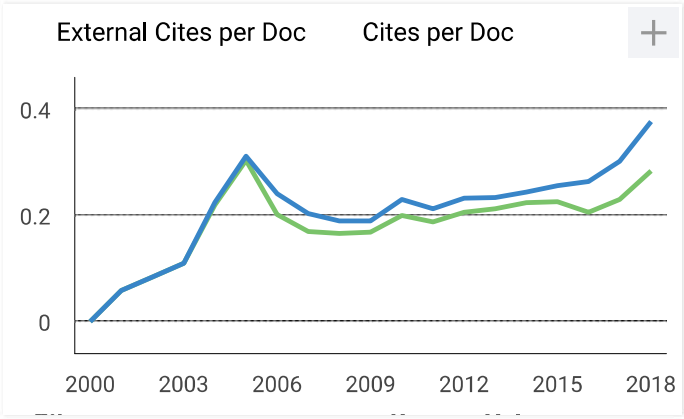

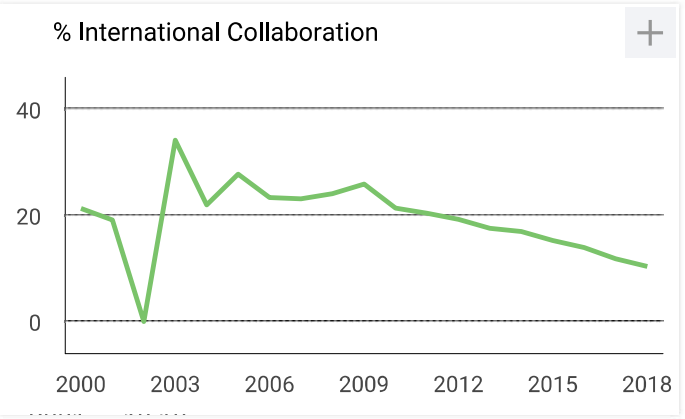

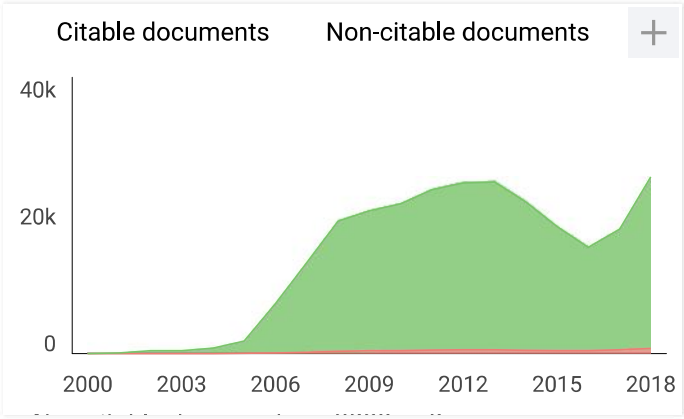

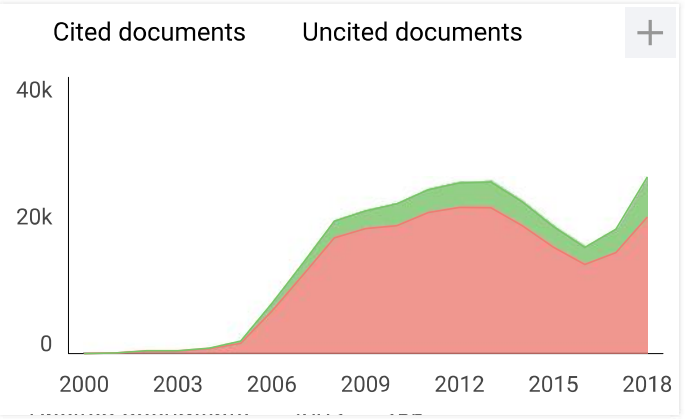

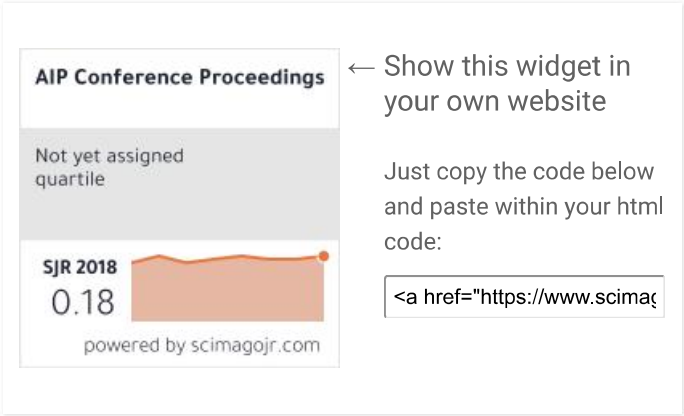

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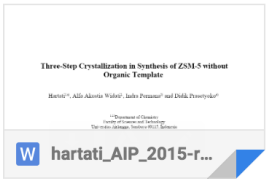

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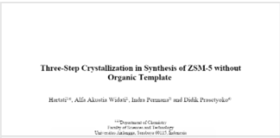
Three-Step Crystallization in Synthesis of ZSM-5 without Organic Template

Hartati[1*], Alfa Akustia Widati[2], Indra Permana[3] and Didik Prasetyoko[4]

[1,2,3]Department of Chemistry
Faculty of Sciences and Technology
Universitas Airlangga, Surabaya 60115, Indonesia


Respons for reviewer comments (ONPM08)

1. *Some researchers have been pursued on same field and similar topic that mean this manuscript reach low novelty. One of them on this link (http://www.sciencedirect.com/science/article/pii/S0376738899000691)*

   Thank you for your comment. We want to clarify about the journal on your link (http://www.sciencedirect.com/science/article/pii/S0376738899000691). In that journal "ZSM-5 membranes synthesized without organic templates using a seeding technique", the authors developed the synthesis method of ZSM-5 without organic template, but still used the seed in 1999. Actually, the using of seed in the synthesis still need a high cost process. Now, many authors including us, are developing the novel method that easy and cheap. We made a synthesis technique via stage crystallization, without organic template and seed. In our results, we have been successfully produce ZSM-5 zeolite.

2. *N2 adsorption/desorption analysis on the others samples was not furnished without any*

Three-step crystallization in synthesis of ZSM-5 without organic template

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Citation: AIP Conference Proceedings **1718**, 060002 (2016); doi: 10.1063/1.4943324
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Three-step Crystallization in Synthesis of ZSM-5 without Organic Template

Hartati[1,*], Alfa Akustia[1], Indra Permana[1], Didik Prasetyoko[2]

[1]Department of Chemistry, Faculty of Science and Technology, Universitas Airlangga, Surabaya, 60115, Indonesia

[2]Department of Chemistry, Faculty of Mathematics and Sciences, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia

*Email: hartati@fst.unair.ac.id

Abstract. ZSM-5 was synthesized without organic template through three steps crystallization by adding seed solution. ZSM-5 crystals was characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy and nitrogen adsorption/ desorption technique. XRD and FTIR showed that this method produced the ZSM-5 and quartz as byproduct. Nitrogen adsorption/desorption technique was as certained the formulation of micro-meso porosity of hierarchical ZSM-5.

Keywords : *ZSM-5, without organic template, micro-meso porosity*

1 Introduction

Commonly, ZSM-5 is synthesized through the addition of organic template as organic structure-directing agent (SDA), namely TPAOH (Xue [1]; Wang [2], Zhu [3]; Tao [4]) or TPABr (Xiao [5]; Pan [6]; Schmid [7]). Prasetyoko [8] synthesized pure ZSM-5 from rice husk ash with the addition of silicalite-1 through the crystallization at 175 ° C for 24 hours. Pan [6] studied the synthesis of ZSM-5 from dealuminated metakaolin with crystallization at 180 °C for 72 hours organic template free. The results as well as ZSM-5 synthesized by using TPABr as a template.

Hence, much research has been directed to develop ZSM-5 synthesis method by the gradual crystallization. Kim [9] reported that ZSM-5 can be observed without the addition of the organic template through a two-step process of crystallization. Crystallization at high temperature (190 °C) were intended to accelerate the nucleation and crystallization at a lower temperature (150 °C) that aims to control the size of crystals and crystal size distribution. The results showed a rapid nucleation and crystallization at high temperatures can form large crystals. Xianliang [10] also studied about the control of crystallization condition, at temperature of 150 °C and 190 °C. They added a nucleation solution before crystallization. The results showed that crystal of ZSM-5 is formed if nucleation solution is made at high temperature (190 °C) and crystallization is done at a lower temperature (150 °C) with mole ratio of $SiO_2/Na_2O < 0.18$. The lower mole ratio of $SiO_2/Na_2O > 0,18$ produced a mordenit materual. Mostafa [11] synthesized ZSM-5 without organic template by gradual crystallization at 180-°C which continued at 120-°C and then at room temperature with the addition of ZSM-5 seed. The results showed that the ZSM-5 formed is partial microporous crystalline nanosize with high surface area. The gradual crystallization in synthesis of zeolite also intended to acquire mesoporous particles, as done by Xianliang [10] to gain a hierarchical porous zeolite NaY particles without organic matter as a mesoporous structure directing agent. The use of organic template and organic species as mesophase directing agent (usually use hexadecyltrimethyl ammonium bromide (CTAB)) have many problems, such as high production costs and the results of thermal decomposition of the species can cause environmental pollution.

In this study, we use three step crystallization in the synthesis of ZSM-5 using seed solution without the addition of an organic template. The results obtained are compared with the synthesis without seed solution and hierarchical ZSM-5 which was synthesized using TPAOH as organic template and CTAB as mesophase directing agent.

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2 Experimental

2.1. Zeolite synthesis

All chemicals directly used without purification, include tetraethyl orthosilicates (TEOS, Merck, 99%); sodium aluminate, $NaAlO_2$ (Sigma Aldrich, $Al_2O_3 = 50 – 55\%$); sodium hydroxide (NaOH, Merck, ≥ 98%); tetrapropylammonium hydroxide (TPAOH, Merck, 40% wt solution in water), hexadecyltrimethyl ammonium bromide (CTAB, AppliChem).
The synthesis of ZSM-5 in gradual crystallization using seed solution (A) performed by dissolving NaOH with distilled water by stirring then sodium aluminate gradually add while stirring. Thereafter, the TEOS was incorporated into the mixture dropwise, so that obtained the molar composition of mixture: $10Na_2O$: $100SiO_2$:1, $25Al_2O_3$:$1800H_2O$. The mixture was divided in two parts (A1 and A2). The first mixture (A1) was allowed for 19 h at room temperature and then the mixture put in a stainless steel outoclave and heated in an oven at 190 °C for 4 h (Xianliang [10]). The second mixture (A2) was allowed at room temperature for 24 h. After that, A1 was blended with A2. The mixture was heated gradually at 100 °C for 24 h, 120 °C for 24 h, and 150 °C for 24 and 36 h. The product isolated by centrifugation, rinsed with distilled water, and then dried at 105 °C for 24 h.

As a comparison, the synthesis of ZSM-5 also carried out gradually without seed solutio with similar molar composition with synthesis ZSM-5 using seed solution. The mixture was stirred for 5 h without seed solution, then allowed for 24 h at room temperature. The mixture was inserted into stainless steel outoclave and then closed tightly and heated in an oven at 100 °C for 24 h, 120 °C for 24 h, and 150 °C for 24 h (B). Synthesis of zeolites in the gradual crystallization without seed solution also performed at low temperature, starting at 60 °C for 48 h and then at 80 °C for 48 h, and at 100 °C for 24 h (C). The solid obtained at each of the crystallisation washed up to neutral pH and dried overnight at 105 °C.

Hierarchical ZSM-5 (Hie-ZSM-5) also used as a comparison was synthesized following the modified method of Eimer [12] and Goncalves [13]. Sodium aluminate was mixed with TEOS through stirring for 30 min, then added TPAOH and aquades so obtained composition: $1SiO_2$: xAl_2O_3:0.2TPAOH: $38H_2O$ ($x = (SiO_2/Al_2O_3)^{-1}$, x-= 1/40 or mole ratio of Si/Al = 20). The mixture was stirred for 15 min, then hydrothermal process was conducted by entering the mixture into a polypropylene bottle, and heated in an oven at 80 °C for 48 h. The mixture was cooled to room temperature and then was added CTAB gradually up to a mole ratio of SiO_2/CTAB = 3.85. The mixture was stirred about 30 min then allowed for 3 h. The solids formed washed with distilled water until neutral pH, then dried at 60 °C for 48 h. The dried solid was calcined at 550 °C for 1 h in N_2 and continued in air for 6 h. The catalyst was previously reported by Hartati [20].

2.2. Characterization

X-ray diffraction patterns (XRD) was measured using a Philips X'pert XRD instrument with Cu Kα radiation in the 2θ range from 5 to 50° in steps of 0.02°. Infrared spectra measured on an infrared spectrophotometer (FTIR) Shimadzu using a KBr pellets techniques in range of wavenumbers of 400 − 4000 cm^{-1} with a spectral resolution of 4 cm^{-1}, 45 scans, at room tempetature. Nitrogen adsorption/desorpsi done at 77 K using Quantachrome Nova version 10. Sampels were degassed at 250 °C before measured. The micropore surface area of samples were measured using Brunauer – Emmett – Teller (BET) method on P/Po = 0.3, while the mesopore surface area, pore volume, and pore diameter were determined by Barrett – Joyner – Halenda (BJH) method.

3. Results and discussion

The crystalline phase and type of zeolite formed were identified by X- ray diffraction (XRD). Figure 1 shows the XRD pattern of samples synthesized by some variation of temperature in the gradual crystallization. It can be seen that the sample that synthesized without organic template and without the addition of seed solution (Figure. 1.A) clearly shows the characteristic peaks of MFI were detected on 2θ around 7-9 º and 23 – 25º as well as the peaks of Hie-ZSM-5. However, in sample A appears peaks at 2θ = 20,92 º and 26,72 º corresponding to peaks of α-SiO_2 (quartz) (Wang [2]). The formation of quartz together in the results of the synthesis of ZSM-5 can be caused by a fairly high alkaline levels and weak template (Sang [14]; Yue-ming and Wang-ming, [15). The formation of the impurities such as quarts can also be occured when the synthesis is done in a long time (Guth and Kessler [16]), as was done in this study. Synthesis of zeolites through gradual crystallization was done using the same material composition with mixture used in synthesis using seed solution. The XRD patterns in Figure 1.B shows that the crystal of ZSM-5 has been formed, however there are other peaks at 2θ around 22 ° and at around 27.5 ° indicate α-cristobalite (Shinohara and Kohyama [17], Prasetyoko [8]). Difraction pattern in Figure 1.B also shows peak at 2θ near 26,72 º indicate of α-SiO_2, while the peaks at 2θ around 6.5º and 10,5º show of mordenite (Xianliang [10]). The XRD pattern in Figure 1.C shows that the results of the synthesis of zeolites without the organic template and without the addition of seed solution a like big hum typically of the amorphous solids. This indicates that the ZSM-5 is not formed.

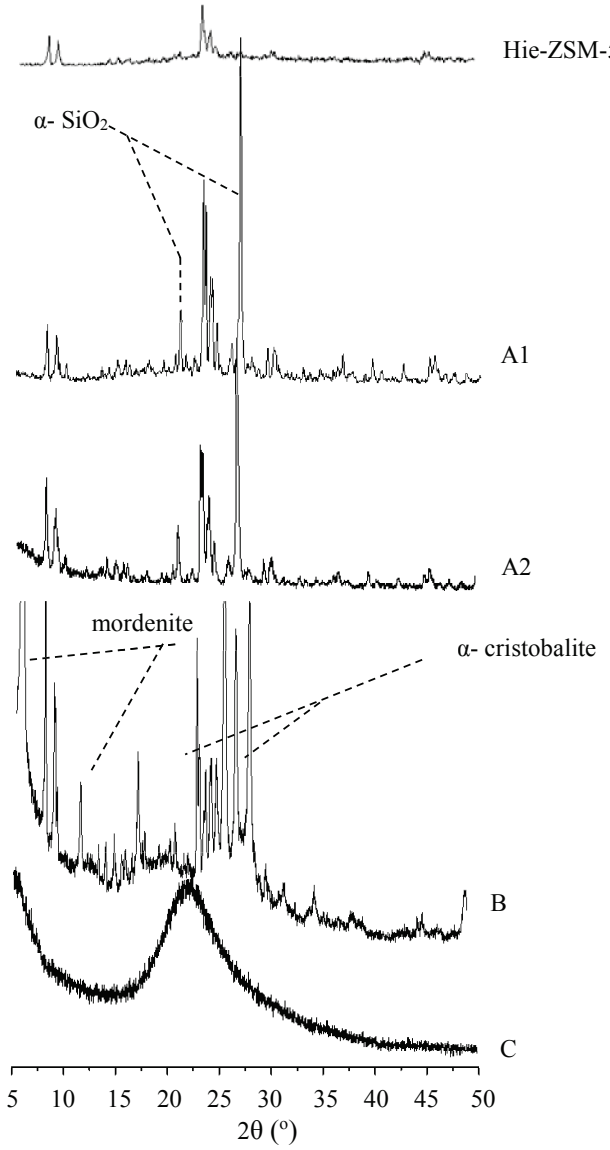

Figure 1. XRD pattern of samples

FTIR spectra at wavenumbers 400-2000 cm^{-1} is shown in Figure 2. Samples A and B in Figure 2 show a typical peak D5R at wavenumbers around 550 cm^{-1} and the vibration of external asymmetric at around 1223 cm^{-1} which became the structure identifier for MFI structure of ZSM-5 as also appear in Hie-ZSM-5 spectra. This is in accordance to the results of Kim [9] which shows that the gradual crystallization begins with warming at high temperature up to 190 ºC can accelerate the formation of the crystal nuclei, so that it can generate ZSM-5. However, that peaks are not appear in sample C. The shoulder band at 1223 cm^{-1} is an asymmetric external vibration (← OTO →) which is characteristic peak of ZSM-5 (Khatamian [18]). A strong absorption at about 1100 cm^{-1} is an asymmetric internal vibration (← OTO →), a weak absorption at about 795 cm^{-1} shows the vibration of external symmetry (← OTO →), and absorption at about 450 cm^{-1} shows the vibrational of T-O from TO_4 (T = Si or Al) (Li and Wu [19]).

Table 1. Wavenumber of streching and bending vibration of FTIR spectra

Sample	Wavenumber (cm^{-1})				
	External Asimetry	**Internal Asimetry**	**External Simetry**	***D5R***	**T-O Bending**
A1	1223	1084	799	546	453
A2	1223	1084	799	546	453
B	1230	1086	783	544	445
C	-	1100	797	-	463
Hie-ZSM-5	1223	1110	797	545	453

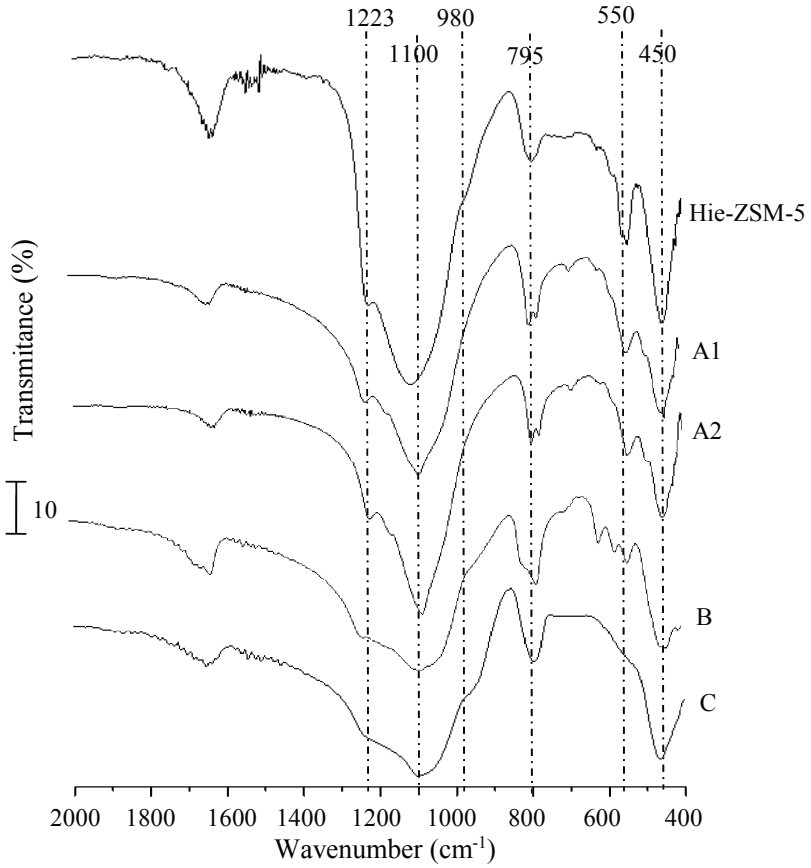

Figure 2. FTIR spectra of samples

The FTIR spectra support the XRD data that show the ZSM-5 can be synthesis by gradual crystalization at high temperature and by addition of seed solution. Figure 1.B shows FTIR spectra of sample C does not show clear peak at 2θ around 550 cm^{-1} and 1223 cm^{-1}. FTIR spectra at Figure 1.C does not show the bands at arround 550 cm^{-1} and 1223 cm^{-1} which are in accordance with the XRD pattern of an amorphous solid (Figure 1.C).

Figure 3 shows that isotherm curve of N_2 adsorption/desorption of zeolite ZSM-5 (A2) is lower than Hie-ZSM-5 which shows that A2 has a smaller porosity and surface area. This is consist with the data shown in Table 2. Table 2 also shows that the sample A2 has a hierarchical porous but the dominant of its surface is microporous. It is different from Hie-ZSM-5, which has a hierarchical porous with large meso-micro porous surface area. From the Figure 3 (insert), it can be stated that both samples showed hierarchical porous structure.

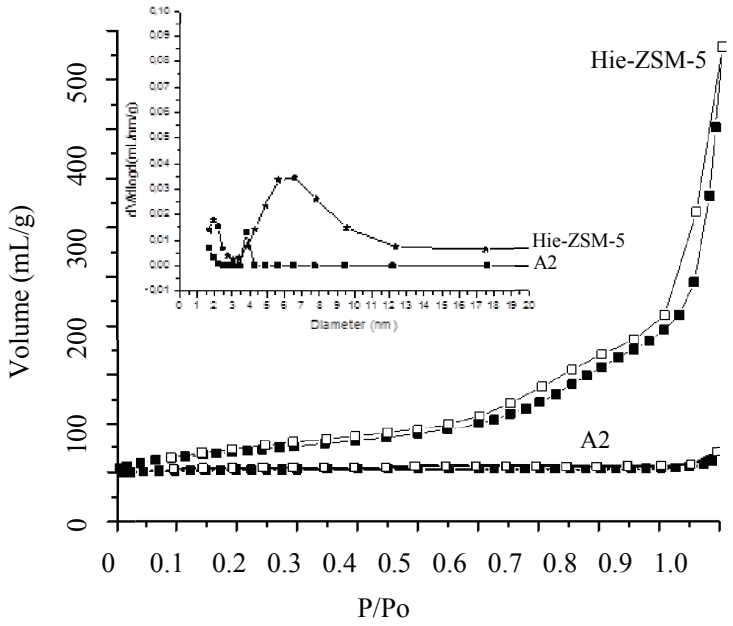

Figure 3. Isotherm N_2 adsorption/desorption of samples and pore diameter of samples (insert)

Table 2. Pore structure properties of samples

Sample	Surface Area Mesoporous[a] (m^2/g)	Surface Area Microporous[b] (m^2/g)	Pore Volume[a] (mL/g)	Pore Diameter[a] (nm)
Hie-ZSM-5*)	177.233	241.756	0.701	6.575
A2	13.015	213.844	0.031	3.808

a. determined by BJH
b. determined by BET method at $P/P_0 = 0.3$
*) as reported previously by Hartati [20]

4. CONCLUSIONS

Synthesis of ZSM-5 with step crystallization can be performed at a high temperature (100 ° C for 24 h, 120 ° C for 24 h, and 150 ° C for 24 or 36 h) with the addition of seed solution. ZSM-5 obtained has a hierarchical pore with the majority of micropores than ZSM-5 synthesized with the organic template and mesophase template. This method also produced the quartz mineral in small quantities.

Acknowledgement

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