PROCEEDING BOOK I

CHEMMPRO 2014

MINERAL AND MATERIAL PROCESSING

International Seminar on Chemical Engineering
in conjunction with
Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) 2014

"Minerals and Materials Processing Toward Sustainable Development"

Bandung, Indonesia
30 - 31 October 2014

Organized by :
Department of Chemical Engineering
Faculty of Industrial Technology
Institut Teknologi Bandung

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PREFACE

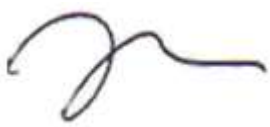
International Seminar on Chemical Engineering in conjuction with Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) was held at East Hall, Institut Teknologi Bandung during 30 – 31 October 2014. This international conference had a theme of "Mineral and Material Processing" which was applicable with the enactment of Law No.4 of 2009 on Mineral and Coal Mining where the ban on the export of unprocessed minerals has been applied since January 2014. Chemical engineers hold a significant role in this area, especially to develop and implement appropriate processing technologies to the mineral resources, which also should considering the sustainable development.

There were five plenary lectures in this two-days conferences, with theme "Sustainable Mineral and Metal Processing" and "The Advancement of Chemical Engineering Technology" along with plenary discussion about "Overcome the Challenges in Indonesia's Mineral and Materials Processing Industry". This proceeding comprises the summary of these outstanding speech and the collected papers that has been presented in the parallel sessions. These papers are divided into several general themes: mineral processing, material processing, material refining and recovery, advance materials, nanotechnology, catalyst, polymers, and others.

The international conference provides an opportunity to publicize research works which done or in ongoing ones in many research institution and showcase their latest advancement and technologies. We have expectation in this occasion is not only a good place to exchange and discuss the progress of their research in chemical engineering that applicable to material and mineral processing, but also a venue to collect and to disseminate the most updated technologies and the researches of regional issue and public interest in order to contribute to the community and to draw support from the industrial and the governmental sectors.

We would like to grateful to all participants and sponsors who has contributed to the conference, to the organizing committee for their commitment in their busy days so that the conference is possible to be held and conducted successfully.

Thank you,



Dr. Dendy Adityawarman
Conference Chairman

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PROGRAM

Day 1: Thursday, 30 October 2014	
	Plenary Session 1
	Sustainable Mineral and Metal Processing
07.30-08.00	Registration
08.00-08.25	CheMMPRO Opening : Vice Rector of Communications, Partnerships, and Alumni Prof.Dr.Ir. Hasanuddin Z. Abidin, M.Sc.
08.25-08.50	Plenary Speaker: Prof. Geoffrey Brooks, Swinburne University of Technology
	Title: Development of Solar Thermal Processing of Minerals
08.50-09.15	Plenary Speaker: Assoc. Prof. M. Akbar Rhamdhani, Swinburne University of Technology.
	Title: Metals from Urban "Ores": Opportunities, Challenges and Technology
09.15-09.35	Discussion
09.35-10.00	Morning Tea / Coffee Break
	Plenary Session 2: Plenary Panel - Business Forum
	Overcome the Challenges in Indonesia's minerals and materials processing industry
10.05-10.25	R. Sukhyar (Directorate General of Mineral & Coal (Dirjen Minerba, Kementerian ESDM)
10.25-10.40	Ir. Hendra Santika, M.M. (PT. ANTAM)
10.40-10.55	Dr.Ir. Rozik B. Soetjipto (Freeport Indonesia)
10.55-11.10	Dinar Aryasena (PT Newmont Nusa Tenggara)
	Title: Throughput Prediction Model Development at Batu Hijau
11.10-11.25	Mr. Graham Brock (Direct Nickel)
	Title: Direct Nickel - Breakthrough Technology
11.25-12.30	Discussion
12.15-13.20	Lunch

Paralel Session 1: 30 October 2014 , 13.20-15.00

13.20-15.00	Room A Advanced material	Room B Industry	Room C Mineral & Material Processing	Room D Mineral Processing
	AM.02	AM.01	MAP.05	MIP.13
	AM.07	IN.02	MAP.06	MIP.05
	AM.04	IN.03	MAP.04	MIP.09
	AM.10	MRY.02	MIP.07	OT.01
	AM.06	MIP.10	MIP.08	MIP.06
15.00-15.30	Afternoon Tea / Coffee Break			

Parallel Session 2

15.30-17.10	Room A Advanced Material & Material Recovery	Room B Advanced Material & Material Processing	Room C Mineral & Material Processing	Room D Others
	AM.05	AM.14	MIP.01	AM.13
	AM.11	AM.12	MIP.03	OT.09
	MRY.07	AM.16	MRY.01	OT.11
	MRY.04	MAP.02	MRY.03	OT.06
	MRY.06	MAP.03	MAP.01	MRY.05
18.00-20.30	Gala Dinner. Venue: House - Sangkuriang			

Day 2: Friday, 31 October 2014				
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The Advancement of Chemical Engineering Technology				
07.30-08.00	**Registration**			
08.00-08.25	**Plenary Speaker: Prof. Ka Ming NG (HKUST)**			
08.25-08.50	**Plenary Speaker: Assoc. Prof Wuled Lenggoro (TUAT, Japan)**			
	Title: Assembly of Fine Particles Synthesized from the Gas-Phase			
08.50-09.15	**Plenary Speaker: Assist Prof. Manabu Miyamoto (Gifu University)**			
	Title: Shape Selectivity of MFI type Core-Shell Zeolite Catalysts			
09.15-09.30	**Discussion**			
09.35-10.00	**Morning Tea / Coffee Break**			
Parallel Session 3				
10.00-11.10	**Room A** **Catalyst**	**Room B** **Process Modelling**	**Room C** **Others (Extraction)**	**Room D** **Polymer**
	AM.03 CT.02 CT.05 OT.15	MRG.01 MIP.02 MIP.04 MRG.02	AM.08 AM.09 OT.02 OT.03	PL.01 PL.02 PL.03 AM.15
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Parallel Session 4				
13.30-15.00	**Room A** **Catalyst/Process Kinetics**	**Room B** **Nanotechnology**	**Room C** **Others (Biorenewable)**	**Room D** **Polymer**
	CT.04 CT.03 OT.07 OT.08 OT.12	NP.01 NP.02 NT.01 NT.02 NT.03	OT.04 OT.05 OT.16 OT.10 OT.13	PL.05 PL.06 PL.09 PL.04 OT.14
14.30-15.00	**Afternoon tea / coffee Break**			
15.00-16.00	**Closing Ceremony**			

International Seminar of Chemical Engineering in Minerals and Materials Processing
In conjunction with Seminar Teknik Kimia Soehadi Reksowardojo 2014

The Effect of Ultrasonic Irradiation on Preparation Zeolite Catalyst from Natural Mineral

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Abstract. The objective of the research is to study ultrasonic radiation in preparation of zeolite catalyst. Natural zeolite that obtained from Gunung Kidul District Indonesia was contain components like SiO_2, Al_2O_3, CaO, MgO, Na_2O, K_2O that potency decelop as catalyst. This material used for synthesis of zeolite catalyst with dealumination, neutralization, drying and calcination processes. In dealumination process did with ultrasonic assisted with time variation 1-6 hours. The zeolite catalyst was analyzed X-ray Diffraction and specific surface area. The results shown crystal percentage decreased due ultrasonic radiation and type of zeolite change complex type to sillicalite. The surface area of zeolite catalyst increased with ultrasonic radiation.

Keywords: *natural zeolite, dealumination, ultrasonic radiation, surface area, type of zeolite*

1 Introduction

Indonesia has a several natural zeolite that contain silica and aluminum. The deposits of natural zeolite can be found in Lampung, Tasikmalaya, Gunung Kidul Malang, etc [1,2]. The natural zeolite has composition like as presented in Table 1. The natural zeolite has content of silica minimum 60%. Table 1 shown that the components are containing in natural zeolite have potency as catalyst. The components can catalytic in chemical reaction like as Al_2O_3 and TiO_2. The utilization of natural zeolite just is limited for water treatment, agriculture, foods for animal like cow and buffalo, carrier in fertilizer and herbicide and medium in planting. So, the mineral is potential for used as raw material in catalyst production. Commonly, zeolite mineral in Indonesia classified as mordernite zeolite[1-3]. Mordenite has been identified as suitable acid catalyst in several industrial processes, such as cracking and isomerization of hydrocarbons. The classification of zeolite mineral according Collela (2007) involve Analcime, Chabazite, Clinoptilolite, Erionite, Faujasite, Ferrierite, Heulandite, Laumontite, Mordenite and Philipsite[4].

Takahara and workers (2005) was developed H-mordernite catalyst that had ratio SiO_2/Al_2O_3 20-90 and ratio SiO_2 to Al_2O_3 90 is more stable [5]. Widayat and workers (2009) reported influencing of time dealumination and solvent concentration in zeolite catalyst preparation that used ethanol dehydration. Zeolite catalyst preparation use chemical and physical treatment that hydrochloride acid concentration 4 M has best performance in ethanol dehydration[1,3]. Ferrierite type zeolite was processed with dealumination process by using hydrochloride acid. Ferrierite catalyst that used for m-xylene tranformation to p-xylene[6]. Boveri and workers report that steaming and acid treatment also increasing performance mordenite catalyst[7] and Chung use acetc acid for activation of mordenite type zeolite[8].

TABLE 1. The composition of natural zeolite in West Java and Banten

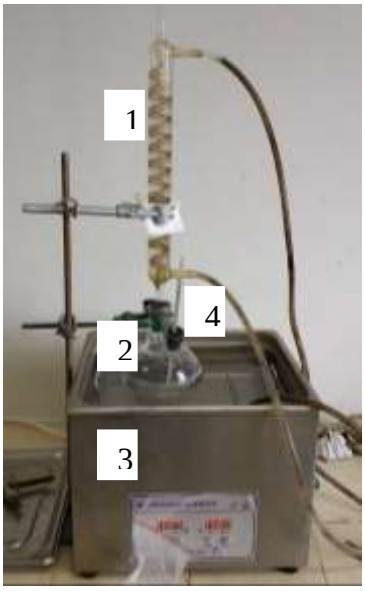
Parameter (%)	District			
	Bayah, Lebak Banten	Karangnunggal, Tasikmalaya	Cikembar, Sukabumi	Nanggung, Bogor
SiO_2	64,55	61,40 – 70,60	68,0 – 69,8	61,39 – 66,16
Al_2O_3	12,83	11,49 – 3,84	11,85 – 13,16	12,04 – 14,12
Fe_2O_3	1,38	1,15 – 5,30	1,52 – 2,39	1,18 – 1,98
CaO	1,64	1,88 – 4,16	1,54 – 2,23	1,75 – 3,78
MgO	0,71	0,40 – 2, 77	0,27 – 0,52	0,55 – 0,90
K_2O	2,81	0,90 – 4,01	2,59 – 5,0	0,30 – 1,78
Na_2O	0,33	0,90 – 2,53		
TiO_2	0,22	0,06 – 0,85	0,03 – 0,19	
H_2O		1,98 – 4,46		1,00 – 1,65

The others treatment in activation of natural zeolite include ultrasonic and microwave irradiation [9-12]. Combination acid treatment and microwave was done on zeolites beta, mordenite and ZSM-5. The results showed higher mesoporosity, higher surface area, and Brønsted acid sites with higher strength than the mordenite treated by thermal treatment[9,10]. Run and workers (2004) use ultrasonic irradiation for Mesoporous Molecular Sieve (MMS) synthesis. Specific surface area of MMS increase similar with increasing of ultrasonic power, time, temperature [11]. Baoyu and workers (2007) use ultrasonic irradiation for MCM-36 preparation. MCM-36 is a zeolite material that has dual porosity i.e microporosity and mesoporosity. A microporosity formed inside its crystalline layers and mesoporosity in the space between the interlayers formed by pillaring the layers with polymeric inorganic oxides. This research use tetramethylammonium silicate as the pillaring agent[12]. Mosaddeg (2013) also use ultrasonic irradiation for nano eggshell powder that a biodegradable catalyst.

The ultrasonic irradiation has potency for catalyst or mineral zeolite preparation. The paper focused of ultrasonic irradiation effect on characteristic zeolite catalyst where preparation with chemical and physical treatment. The catalyst characteristic includes crystallite, specific surface area and morphology.

2 Material and Methods

The material was used a natural zeolite that obtained from Wonosari Gunung Kidul district. So, this material were reduced of size until obtained 100 mesh, then was analyzed composition, water content and crystallography [2]. Hydrochloride acid has technical specification that used as solvent in chemical treatment and found in Brataco CV. Hydrochloride acid solution has 4 M concentration. Silver nitrate solution has concentration 0.1 M that used as indicator in washing of catalyst product. The equipment that used in this research a three-neck flask equipped with a thermometer and a condenser. This equipment presented in Figure 1.The equipment is placed an ultrasonic cleaner that has 40 kHz frequency. An ultrasonic cleaner, type JP-060S was produced by Skymen International (HK) Limited. The electrical power of the generator is 250 W and the temperature can be controlled from 303 to 333 K.



Annotation:
1. Condenser
2. Three neck bottle
3. Ultrasonic cleaner
4. thermometer

Figure 1. Equipment experiments

The chemical treatment was carried out with 40 gram natural zeolite added 800 ml of 4 M hydrochloride acid solution. Temperature operation was fixed 323 K and operation time varied 0.5-6 hours. Catalyst product was filtered and washed with distilled water until total chloride removal. After that, the samples were

dried and calcined in inert condition. The calcination of catalyst product was done on isothermal temperature at 500^{o}C until five hours.

Catalyst Characteristic

The catalyst product was analyzed of characterization consist of specific surface area, total pore volume (BET analysis), and crystallite. The analysis crystallography was used x-ray diffractometer, XRD-7000S model Shimadzu brand with X-ray tube target Cu, voltage 30 kV, current 30 mA, and Kα radiation. XRD data was analyzed with PCXRD software. Morphology of catalyst was analized with JEOL PC Scanning Electron Microscope (PCSEM) model JSM-6510LA with magnification x5000. The BET analysis by using *Quantachrome* with Po = 711.65 mmHg and nitrogen as carrier gas. The analysis process did in Center of Research and Service Diponegoro University (CORES DU).

3 Results and Discussion

3.1. Natural zeolite characteristic

The natural zeolite was treated by size reduction and analysed crystalograph by using XRD analysis. The result was presented in figure 2. XRD pattern show that natural zeolite has crystalline. XRD data was did matching with library database. The library database of material that used include mordenite, aluminum oxide, silicon oxide calcium sulfate, sodium aluminum silicate, kaolinite and barium sulfate. Semi qualitative analysis with XRD software was resulted a mounts mataerial. The material like presented in Table 2. Figure 2 is a result matching XRD pattern of natural zeolite with silicon oxide (red line), calcium sulfate (green and black line), sodium aluminum silicate (blue line) and barium sulfate (pink line). The results compared with Widayat et.al., (2009, 2010) that natural zeolite has a mordenite type. This research natural zeolite is a mixture mineral like as silicon oxide, barium sulfate, calcium sulfate and sodium aluminum silicate. The components have variety composition. Barium sulfate has composition bigger than silicon oxide, calcium sulfate and albite. Calcium and barium is metallic II A in periodic system. These metallic do not need in catalyst. So natural zeolite need pretreatment processing if use as catalyst.

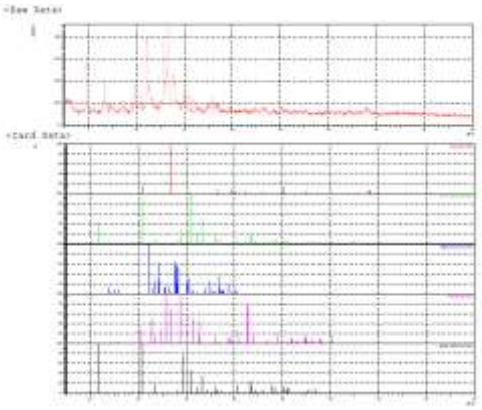


Figure 2. XRD Pattern of natural zeolite and matching with library database

Table 2. Analysis type of natural zeolite

Mineral Name	Line Colour	Chemical Formula	Composition (%w/w)
Silicon oxide (Quartz)	Red	SiO_2	33.35
Calcium Sulfate Hydrate (Gypsum)	Green and black	$CaSO_4.2H_2O$	22.77
Sodium Aluminum Silicate (Albite)	Blue	$NaAlSi_3O_8$	5.47
Barium Sulfate (Barite)	pink	$BaSO_4$	38.40

Catalyst Product Characteristic

The natural zeolite and catalyst product was compared in crystallography. The catalyst product was treated by chemical treatment- ultrasonic irradiation, and physical treatment. The XRD pattern was presented in figure 3. XRD pattern of natural was compared with XRD pattern of catalyst product. The figure 3 shows that natural zeolite has peak higher than peak of catalyst product. Natural zeolite has metallic contents bigger than catalyst product for calcium, barium and aluminum. Chemical treatment processing with irradiation causes changes reaction between metallic with chloride ion. This was caused the process of dissolving the components Calcium sulfate $CaSO_4$, Barium sulfate $BaSO_4$ and albite $NaAlSi_3O_8$. Reactions were happened like presented in this reaction:

$$Ca^{2+}_{aq} + 2Cl^{-}_{aq} \rightarrow CaCl_{2(aq)}$$

$$Ba^{2+}_{aq} + 2Cl^{-}_{aq} \rightarrow BaCl_{2(aq)}$$

$$Al^{3+}_{aq} + 3Cl^{-}_{aq} \rightarrow AlCl_{3(aq)}$$

Compounds of barium chloride, calcium chloride and aluminum chloride are a solution that followed in filtrate at filtration process.

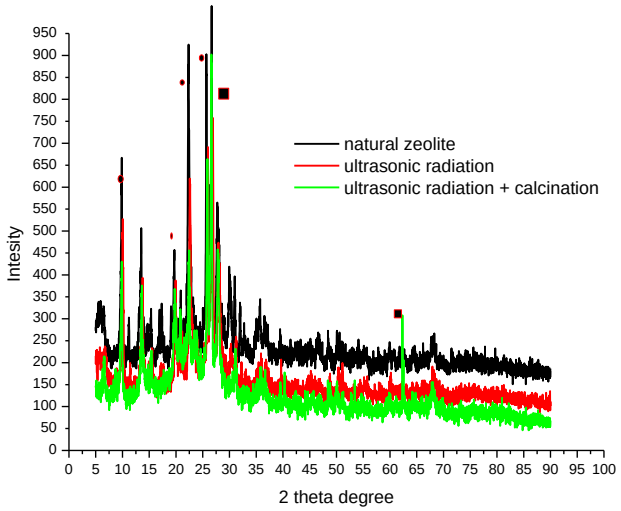


Figure 3. XRD pattern of natural zeolite and catalyst product

The products catalysts commonly have grey color. The calcination processing cause color of catalyst becomes brown whiteness. The catalyst products have a yellow and brown color because the catalysts contain Fe, Zn, Pb and Cu components [13]. Fe component can be cause colour in catalyst product brown whiteness[1,3]. Figure 3. was also indicate that chemical treatment-ultrasonic irradiation and physical treatment influence in preparation of zeolite catalyst. XRD patterns have peaks as similar in 2 theta degree 5-90. Intensity of natural zeolite pattern has peak higher than catalyst with ultrasonic irradiation and combining ultrasonic irradiation with physical treatment. This indicates that the concentration of barium, calcium and aluminum is higher than the product of the catalyst. Chemical treatment –ultrasonic radiation led to a decrease in these metals.The circle signs in Figure 3 as indicate this phenomena. Overall, the intensity decreases with the chemical treatment, ultrasonic irradiation and physical treatment. In the following 2 theta degree 9.8, 20.8, 23.32, 25.6, showed a significant decrease in intensity.

Pattern XRD with green line is a CRD pattern for zeolite catalyst that preparation with chemical-ultrasonic irradiation and physical treatment. The others results, intensity in 2 theta degree 25 and 62.34 increase. It is indicating increasing of components composition like silica (Si). The physical process that use in the research is calcinations process. This process causes the reactions to residual calcium sulfate and barium sulfate. The reaction is as follows.

$$CaSO_4 \rightarrow CaO + SO_{2(g)}$$

$$BaSO4 \rightarrow BaO + SO_{2(g)}$$

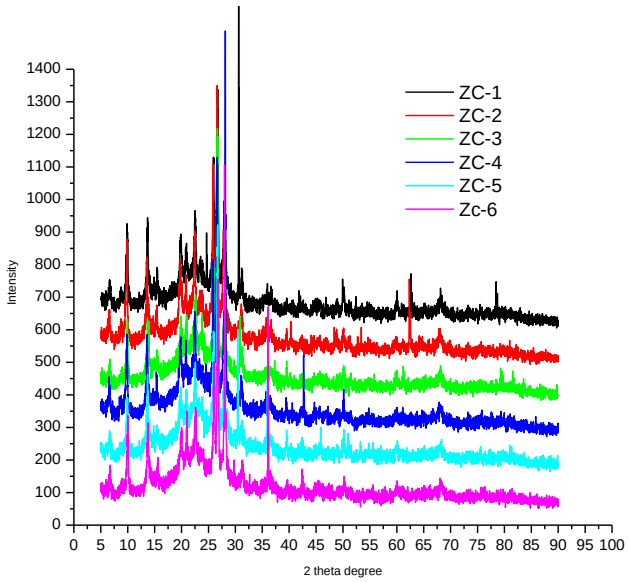


Figure 4. XRD pattern of zeolite catalysts (ZC)

Fugure 4 is XRD pattern of zeolite catalyst that preparation catalyst with chemical – ultrasonic irradiation and physical treatment. Ultrasonic irradiation varied in time 1-6 hours. ZC-1 is zeolite catalyst that preparation by using chemical –ultrasonic irradiation 1 hours and calcinations processing. ZC-2, ZC-3, ZC-4, ZC-5 and ZC-6 are a zeolite catalyst that preparation with ultrasonic irradiation for 2, 3, 4, 5 and 6 hours. Figure 4 was indicating that time ultrasonic irradiation was influencing crystallography of catalyst product. All Zeolite catalyst have diffractogram similar. Differences peak in Fig 4 is located at 2 theta degree 26, 32 and 35.4.

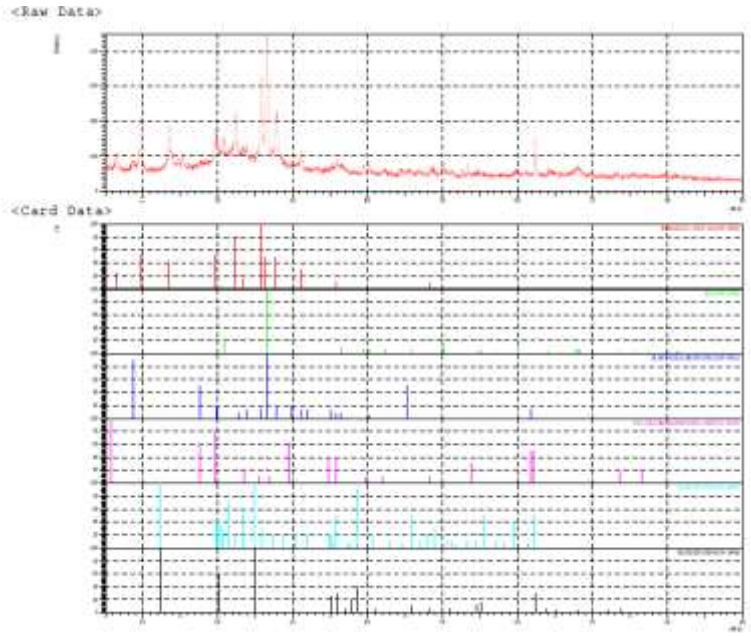


Figure 5. XRD Pattern of zeolit catalyst and matching with library database

The XRD data of zeolite catalyst also was analysed crystallography and crystallite percentage by using PCXRD 7000ver. The result was presented in figure 5. The library database of material that used include mordenite, aluminum oxide, silicon oxide calcium sulfate, sodium aluminum silicate, kaolinite and barium sulfate. This research natural zeolite is a mixture mineral like as silicon oxide, barium sulfate, calcium sulfate and sodium aluminum silicate. The results of the data processing software showed that the catalyst is a kind silicalite. Barium sulfate, calcium sulfate and albite / sodium aluminum silicate is not there in the catalyst. This happens because the dissolution reaction /dealumination and calcination. The others researchers reported that ultrasonic radiation can increase specific surface area and crystallography, proportionate with ultrasonic power, time, temperature [11,12]. Similar results with other researchers [11,12] showed that ultrasonic irradiation can increase the specific surface area. In this experiment showed that ultrasonic radiation 1 hour to obtain a specific surface area of 79.09 m^2/gram and 2 hours obtain specific a surface area of 107.378 m^2/gram. Increasing in surface area is very significant. Natural zeolite generally has a surface area of about 20 m^2/gram[1,3].

The results of degree of crystallization presented in Figure 6. Figure 6 shows that degree of crystallization was not significantly affected by the ultrasonic irradiation time. But there is a reduction in the degree of crystallization in the presence of ultrasonic radiation treatment.

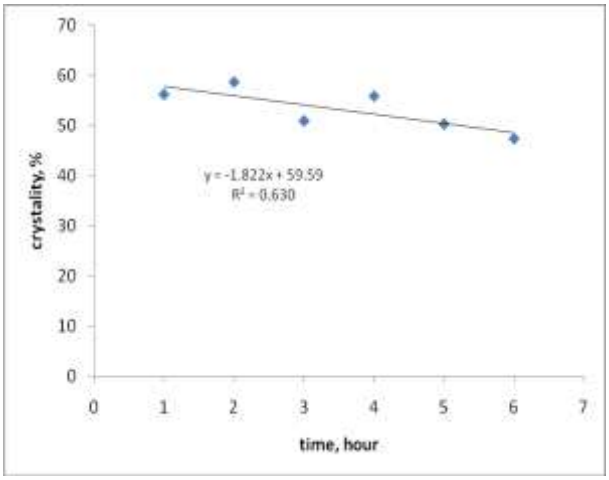


Figure 2. Correlation of crystallite percentage vs irradiation time.

4 Conclusion

Synthesis of zeolite catalysts has been done using ultrasonic assisted. Natural zeolite obtained from Gunung Kidul district is a mixture Barium sulfate, calcium sulfate, silicon oxide and albite. With the chemical treatment - ultrasonic irradiation followed physical treatment turned into a type of zeolite. Type of zeolite change from a mixture mineral to silicalite type. The specific surface area of zeolite catalysts increased significantly. Zeolite catalyst has the potential to be developed particularly from minerals.

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CHEMMPRO 2014

MINERAL AND MATERIAL PROCESSING

CERTIFICATE

is hereby presented to

Widayat

The Effect of Ultrasonic Radiation on Preparation Zeolite Catalyst from Natural Mineral

as

Paper Presenter

International Seminar on Chemical Enginnering
in conjunction with
Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) 2014
"Minerals and Materials Processing Toward Sustainable Development"
30-31 October, 2014 at East Hall, Institut Teknologi Bandung, Indonesia

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Conference Chairman