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CHARACTERIZATION ZSM-5 MEMBRANE BASED ON VARIATION OF GAUZE BY TREATMENT SOAKED WITH NITRIC ACID AND ACETONE TO DECREASE CO ONCENTRATION

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Abstract

CO gas is a very toxic gas, because the ability of hemoglobin to bind CO gas to COHb is greater (300 times) than oxygen, causing the blood to lack of oxygen supply, which can in some cases lead to death. Therefore, it is necessary to decrease CO gas such as ZSM-5 zeolite membrane. This study aims to determine the growth of ZSM-5 zeolite on surface gauze surface steel. The problem is whether the immersion treatment in 1% nitric acid solution at 333K then continued to be soaked in acetone on the variation of gauze type and stainless steel size has effect to sticking precursor ZSM-5 to synthesis ZSM-5 membrane on the low temperature. The resulting membranes were characterized by XRD, FTIR, and SEM-EDX and the ZSM-5 membrane was applied to decrease CO gas concentration in the room. Results showed that the percentage of CO levels based on variation of gauze types (304 and AISI 316) and sizes (100, 200 dan 400 mesh) soaked treated with 1% nitric acid and and acetone at 363 K was 15.07 ± 1.05 %. The most significant was the highest decrease of CO gas aobserved on Stainless Steel gauze of AISI 316 type. One way anova analysis showed that there was influence of treatment given in the syntesis process of zeolite ZSM-5 membrane by coating to the decrease levels of CO gas and adsorption capacity of zeolit ZSM-5 membrane obtained.

Keywords: *Gas CO, Zeolit ZSM-5 Membrane, Variation gauze of stainless steel, Treatment was soaking in nitric acid and aceton*

1. Introduction

CO gas is a very toxic gas, and because the ability of hemoglobin to bind CO gas to COHb is greater (300 times) than oxygen, causing the blood to lack of oxygen supply, which can in some cases lead to death [1]. CO gas can be adsorbed by using zeolite. Zeolite is a tetrahydrate silica alumina crystalline mineral having a three-dimensional skeletal structure, formed from tetrahydral between aluminate (AlO_4^{5-}) dan silicate. (SiO^{4-}) are interconnected by the oxygen atoms to form a three-dimensional framework, containing the cavities in them filled by metal ions ie alkali or alkaline earth metals and freely moving water molecules [2].

ZSM-5 zeolite membrane synthesis using stainless steel gauze of various types and sizes previously immersed in HNO_3 and acetone solutions [3]. The problem is whether the variation of gauze type variation and stainless steel treatment on ZSM-5 zeolite membrane synthesis coating at low temperature has an effect on the decrease of CO gas concentration? The objectives of this study synthesized ZSM-5 zeolite membranes coating at low temperatures with various types of gauze with initial treatment soaked in HNO_3 and aceton solutions, characterizing ZSM-5 zeolite membranes by X-ray (XRD), SEM-EDX, and FTIR applying as adsorbent to decrease of CO gas in the room.

Zeolite ZSM-5 is a zeolite with medium pore size (5,1-5-6Å) with a three-dimensional pore structure. The acid properties possessed by ZSM-5 cause this zeolite is often used as a gas conversion catalyst in the petroleum and petrochemical fields [4]. Zeolite ZSM-5 has a large surface area and has a channel that can filter ions or molecules. The benefits of zeolite can be as a filter molecule, ion exchange, filter material, catalyst [5].

Several studies have reported that the physical structure and chemical composition of the buffer greatly affects the growth of the zeolite membrane. The choice of buffer is very important considering the price, thermal conductivity, anti-corrosion quality, and mechanical strength. Stainless steel gauze is heavily promoted as a buffer for micro-pore membranes, as stainless steel is an alloy of some metallic elements, corrosion-resistant, strong and resistant to oxidation reactions and is an environmentally friendly material [6]. [7] stated that the polypropylene container with the ratio of surface area to reactor volume 1.44 is the best container for synthesizing zeolite ZSM-5.

There are several types of stainless steel gauze, including 304 and AISI 316. stainless steel case contains 10.5% chromium element, so corrosion resistance is increased by forming chrome oxide film (Cr_2O_3), a thin layer that can protect itself and called the protective layer. The other metal content is Molibdeum (Mo), so it is more corrosion resistant especially in acidic environment, whereas 304 type gau contains chrome element less than 10,5% [8].

2. Method

This research type is Pre Experiment Design research. This study uses a stainless steel type AISI 316 size 180 mesh and 304 gauze sizes 100, 200, and 400 mesh with a treatment soaked in nitric acid and aceton and then dried at 383 K [3]. Membrane result were *characterized by XRD, FTIR, and SEM-EDX Decrease CO gas with CO meter,*

Treatment on *Stainless Steel* Gauze

Stainless steel gauze type 304 sized 100, 200 and 400 mesh as well as stainless steel gauze with type of AISI 316 sized 180 mesh were prepared cut at size of 3 cm x 3 cm and soaked in HNO_3 1% at 60 °C for 4 h, then continued for soaking in acetone for 1 h, and kept at 373 K]3].

Preparation of Zeolit ZSM-5 Precursor

Initially 0.136 g $NaAlO_2$ and 1,390 g NaOH 50% w/v were mixed in polypropylene 1 containers with surface area to volume ratio 1.44 (Mukaromah, et al., 2016), 1,549 g TPABr dissolved with 7,3788 g of distilled water in containers, and stirred with a magnetic stirrer for 5 minutes, then put into a polypropylene container 1, and added 24.490 g of ludox HS-40%, occurring semi-gel and stirring at a rate of 900 rpm for 6 h.

Synthesis of zeolit ZSM-5 membrane at low temperature for 4 h by *coating*

After the gauze buffer has been given treatment and then immersed in zeolit ZSM-5 prekusor, then heated in oven temperature 363 K for 4 days. Furthermore, the resulting ZSM-5 membrane was washed with distilled water, dried at 363 K overnight and then heated at 550 °C in a muffle furnace for 6 h.

Decrease of CO concentration indoors with ZSM-5 zeolite membrane

CO gas produced from cigarette smoke flowed for 3 minutes into an enclosed space with a capacity of 18 liters. Initial CO gas content was measured using a CO meter, then a Zx-5 zeolite measuring 3x3 cm^2 of known mass was inserted into the enclosure for 10 minutes, then the final CO gas content was recorded.

3.Results

The composition of the various buffer components before and after being soaked in a solution of HNO_3, then immersed in acetone characterized by EDX is shown in Table 1.

Table 1. Compositions of components of various buffer gauze before and after immersion in HNO_3 solution, then immersed in aceton.

No.	Type and Sizes gauze	Componen	Composition (%w/w) Before treatment	After treatment
1	304-100 mesh	C	5.05	8.10
		N	-	1.91
		Al_2O_3	0.47	0.58
		SiO_2	0.71	1.36
		Cr_2O_3	9.47	16.48
		MnO	16.18	14.82
		FeO	68.15	56.75
2	AISI-180 mesh	C	7.44	8.41
		SiO_2	0.89	0.82
		Cr_2O_3	19.12	19.54
		FeO	58.34	58.42
		NiO	9.97	10.53
		MoO_3	2.15	2.26
		MnO	1.38	-
3	304-200 mesh	C	6.12	5.86
		SiO_2	0.82	0.82
		Cr_2O_3	20.67	21.47
		FeO	63.25	68.85
		NiO	6.30	7.00
4	304-400 mesh	C	11.1	8.65
		Al_2O_3	-	0.43
		SiO_2	0.57	0.75
		Cr_2O_3	18.63	19.52
		MnO	1.32	-
		FeO	55.89	58.15
		NiO	9.92	10.17
		MoO_3	2.45	2.33

Table 1 shows that by immersion in the HNO_3 solution, then immersed in aceton increased Cr_2O_3 and Fe_2O_3 levels. Sample characterization with XRD is shown in Figure 1, FTIR is shown in Figure 2, and SEM in Figure 3.

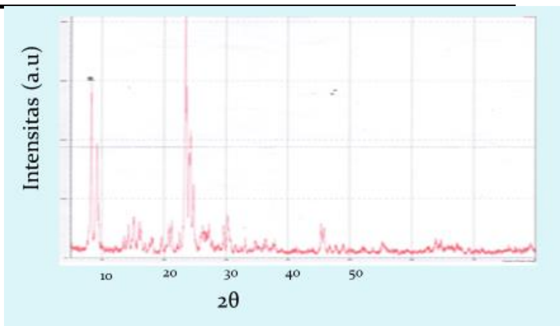

Figure1. XRD of ZSM-5 on stainlees steel support

Figure 1 shows that crystallization of ZSM-5 at low temperature (363 K) was successfully achieved in 4 days. It should be noted that the TPABr used in this study was much lower than that of the typical syntheses with TPABr/Si ratio around 0.1-0.3 [9.10.11]. Figure 1 depicts the XRD patterns of the products from exhibited a bump around a 2θ angle of 8 and 23°, which is a characteristic feature intense reflections observed crystallin ZSM-5.

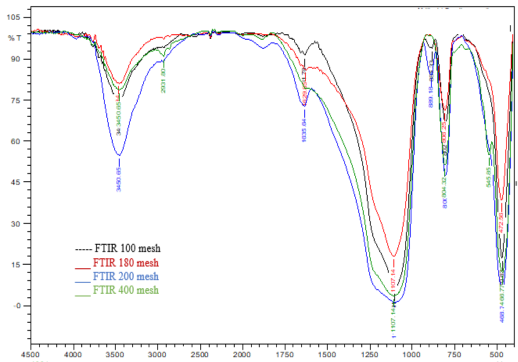

Figure 2. FTIR spectra of various types and sizes of gauze by being soaked in nitric acid then soaked in acetone

Figure 2 shows the FTIR spectra of the ZSM-5 samples. The information about the Si–O bonds is found in the wavenumber range from 1400 cm-1 to 400 cm-1. The most intense band at 1100 cm-1 is attributed to the transversal mode of asymmetric stretching of Si–O–Si bonds. Another band at 1225 cm-1 corresponds to Si–O–Si asymmetric stretching vibration between SiO4 tetrahedral, which is typical for zeolitic phases. The spectra of the products from gauze 304 200 mesh showed the least intense band at this wavenumber due to the significant presence of the amorphous part. Furthermore, a small shoulder at 962 cm-1 is attributed to the in-plane stretching vibration of Si–OH (silanol) bonds. The band at 800 cm-1 corresponds to the symmetric stretching vibration of Si–O–Si bonds and its bending vibration appears at 450 cm-1.

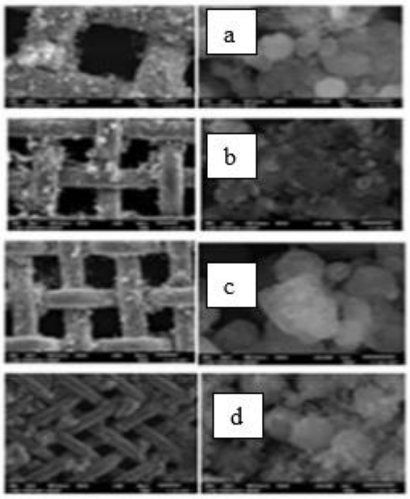

Figure 3. SEM images of various types and gauze sizes by treatment are soaked in nitric acid then immersed in aceton a) 304-100 mesh, b) AISI 316-180 mesh, c) 304-200 mesh, d). 304-400 mesh.

The decrease of CO gas concentration on the variation of gauze treatment type was soaked in 1% HNO_3 at 333 K for 4 h, then soaked in acetone for one hour shown in Figure 4.

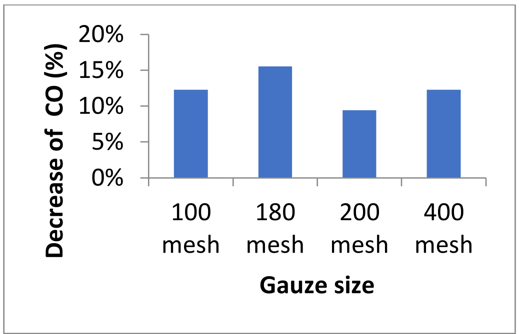

Figure 4. Graph of reduction of CO (%) gas content to variations of type and size of gauze immersed in nitric and acetone acids.

Figure 4 shows the percentage decrease of CO gas content with 10 min. contact time was the highest result was 15.07% ± 1.05 type of AISI 316 size 180 mesh. The average yield data of percentage reduction of CO gas content was tested normalized using Shapiro Wilk test, obtained by normal distribution data that is p (sig) or p value> 0,05. 180 mesh obtained p value = 0.787, 200 mesh gau obtained p value = 0.272, and 400 mesh gauze obtained p value 0.353> 0.05 which means the distribution of data is normal distribution. The result of data processing with One Way Anova test obtained obtained p value <0,05 0.000 where this result stated

4. Discussion

Treatment to the gauze prior to use as a buffer for the zeolite membrane (silicate) ZSM-5 can grow on the surface of the screen. The process of forming ZSM-5 at 363K lasted for 4 days, then continued calcination for 823K. This process is a heat treatment in order to rearrange unstable silica alumina into a more stable form and produce better crystal arrangement. The band at 550 cm-1 corresponds to the asymmetric stretching vibration of the double fivemembered rings (d5r) in the pentasil building units, which is a characteristic of ZSM-5 frameworks. The information about the crystallinity of the products was estimated by the intensity ratio between the bands at 550 cm^{-1} and 450 cm^{-1} [12]. All of the band assignments were in accordance with the previous literatures [13, 14]. Figure 4 shows the percentage decrease of CO gas content with 10 min. contact time was the highest result was 15.07% ± 1.05 type of AISI 316 size 180 mesh.

5.Conclussion

Treatment to the gauze prior to use as a buffer for the zeolite membrane (silicate) ZSM-5 can grow on the surface of the screen. The highest of percentage of CO levels based on variation of gauze types (304 and AISI 316) and sizes (100, 200 dan 400 mesh) soaked treated with 1% nitric acid and and acetone at 363 K was AISI 316 decrease CO gas 15.07 ± 1.05 %. There is influence of ZSM-5 zeolite membrane which is synthesized coating at temperature 90 ° C based on variation of type of gauze size of zeolite membrane ZSM-5 to the decrease in CO gas content.

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7. References

1. Zulfah, Wibowo, A, and Hartoni, U, C,. 2011. Analysis of the Effect of Use of Catalytic Converter on a four-step

engine on the reduction of flue emissions, Thesis, Pancasakti University of Tegal.
2. Lestari, D,. 2010. Study of Modification and Characterization of Natural Zeolite from Various Countries. oceedings of the national seminar on Chemistry and Chemistry Education, Yogyakarta.
3. Lopes, F., Bernal, M. P., Mallada R., Coronas, J., dan Santamaria, J. 200 5. Preparation . of Silicalite Membrane on Stanless Steel Grid Supporots. Ind. Eng. Chem. Res. 44, 7627-7632
4. Cejka, J,H,Van Bekkum, 2005, 'Zeolite and Ordered Mesoporous Materials : Progress and Prospect', Czech republic : The 1st FEZA School on Zeolites, Pague Studies in Surface Science and Catalysis Volume 157
5. Nurropiah, P., Mukaromah, A.H., Sitomurti D.H. (2015): Decrease in chromium (VI) in water using ZSM-5 zeolite with concentration variation and length of immersion time. National Seminar on Mathematics and Health The 2nd University Research Colloquium 2015. ISSN 2407-9189, 445-450
6. Holmbergh, B, 2008, 'Stainless Steels: Their properties and suitability to welding, Avesta Polarit, Sweden, Vol. 4, No. 2 hal: 52-56.
7. Mukaromah, A.H, Kadja, G.T.M Mukti, R,R, Pratama, I,R, Zulfikar, M,A, &Buchari. 2016. Surface-to-volume Ratio of Synthesis Reactor Vessel Governing Low Temperature Crystallization of ZSM-5. Journal of Publisher ITB,48 (3). Page :241-251.
8. Konno, H., Tago, T., Nakasaka, Y., Ohnaka, R., Nishimura, J. & Masuda, T., Effectiveness of Nano-scale ZSM-5 Zeolite and Its Deactivation Mechanism on Catalytic Cracking of Representative Hydrocarbons of Naphtha, Microporous Mesoporous Mater., 175, pp. 25-33, 2013.
9. Petushkov, A., Yoon, S. & Larsen, S.C., Synthesis of Hierarchical Nanocrystalline ZSM-5 with Controlled Particle Size and Mesoporosity, Micropororous Mesoporous Mater., 137, pp. 92-100, 2011.
10. Yokomori, Y. & Idaka, S., The Structure of TPA-ZSM-5 with Si/Al=23, Microporous Mesoporous Mater, 28, pp. 405-413. [22] Al-Oweini, R. & El-Rassy, H., Synthesis and Characterization by FTIR Spectroscopy of Silica Aerogels Prepared using Several Si(OR)4 and R′′Si(OR′)3 precursors, J. Mol. Struct., 919, pp. 140-145, 2009.
11. Shukla, D.B. & Pandya, V.P., Estimation of Crystalline Phase in ZSM-5 Zeolites by Infrared Spectroscopy, J. Chem. Tech. Biotechnol., 44, pp. 147-154, 1989.
12. Al-Oweini, R. & El-Rassy, H., Synthesis and Characterization by FTIR Spectroscopy of Silica Aerogels Prepared using Several Si(OR)4 and R′′Si(OR′)3 precursors, J. Mol. Struct., 919, pp. 140-145, 2009.
13. Figueiredo, A.L., Araujo, A.S., Linares, M., Peral, Á., García, R.A., Serrano, D.P. & Fernandes Jr., V.J., Catalytic Cracking of LDPE Over Nanocrystalline HZSM-5 Zeolite Prepared by Seed-assisted Synthesis from an Organic-template-free System, J. Anal. Appl. Pyrol., 117, pp. 132-140, 2016.