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by Setia Budi Sasongko

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Setia Budi Sasongko, Didi Dwi Anggoro, Luqman Buchori, Rico Febrianto, and Ester Alninta Basa Siagian

The Effect of Dealumination Process on Zeolite Y Acidity and Morphology

Setia Budi Sasongko[a)], Didi Dwi Anggoro, Luqman Buchori, Rico Febrianto and Ester Alninta Basa Siagian

Department of Chemical Engineering, Faculty of Engineering, Diponegoro University, Indonesia

[a)]Corresponding author: sbudisas@live.undip.ac.id

Abstract. Zeolite Y is well-known as hydrated alumino-silicate minerals, which are used in catalysis, adsorption, and separation. The catalytic properties of zeolites depend on acidity and surface area. It can be modified by Si/Al ratio. The main objective of this paper study the effects of the dealumination process of zeolite Y on acidity and morphology. Dealumination of zeolite Y used H_2SO_4, drying process at 100 °C for 1 hour, then the calcinating process at 500 °C for 3 hours. After dealumination, characterization attempts through surface area analyzer using BET (Brunauer-Emmett-Teller) method to determine the surface area of the catalyst and acidity analysis using NH_3 and pyridine to determine a total of acidity and total of acidity at the surface of the catalyst. Based on the result, dealuminated catalyst has less acidity number for both surface and total body (0.00006 mol/gram and 0.0031 mol/gram) with a greater number of surface areas (668.694 m^2/gram) compares to all the zeolite Y without dealumination. It is caused by dealuminated zeolite Y has more active sites due to decreasing number of alumina and caused a bigger porosity. Bigger porosity indicates a smaller surface area of catalyst.

Keywords: catalyst, zeolite Y, dealumination

INTRODUCTION

The Zeolites are known as hydrated alumino-silicate minerals, which are used in catalysis, adsorption, and separation. They made from interlinked tetrahedra of alumina (AlO_4) and silica (SiO_4), some contain more alumina, while others contain more silica. The catalytic properties of zeolites depend on acidity, which can initiate carbenium ion reactions [1]. The acidity and acid strength of the zeolite can be modified by Si/Al ratio. Modified dealumination of zeolite will change the surface area and will affect the efficiency of catalyst [2].

The dealumination process increases the acidity of zeolite [3]. It changes in the electrostatic area of zeolite for adsorption, stability for low silica zeolite is 800-900 K and high silica zeolite is 1300 K, and high acidity level [4].

The main objective of this paper study the effects of the dealumination process of zeolite Y on acidity and morphology.

METHOD

The dealumination process consists of three steps, that are chemical treatment, washing, and calcination. The chemical treatment is processed through chemical treatment using H_2SO_4 in Magnetic stirring heater and a series of boiling 3-neck and Liebig column.

The dealumination process runs in several combination variables between temperature, time and concentration (N) of H_2SO_4. The first step of the dealumination process was creating a solution of H_2SO_4 with several concentration variables. Secondly, 10-gram zeolite Y was added to the solution. Thirdly, the

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dealumination process was taken place with temperature and time based on the combined variable. Further, it washed and filters the solution by a vacuum pump. The zeolite Y filtered was dried at 110 °C. The calcination process of zeolite Y was carried out using the furnace at a constant temperature of 550 °C over 3 hours. As a result, zeolite Y as the catalyst has been modified.

The Catalyst that has been made was being tested through the acidity test and surface area test. Acidity test was carried out using pyridine and NH_3 by flowing the gas into catalysts, to check out the amount of active site inside zeolite Y. The surface area test as a morphology test was carried out using the Surface Area Analyzer with the Brunauer-Emmett-Teller (BET) method to determine the surface area of zeolite Y. The surface area analyzer utilized the quantachrome instruments from Central-Laboratorium Diponegoro University.

RESULTS AND DISCUSSIONS

In this study, the dealuminated zeolite Y catalyst was analyzed for its effect on the characterization of the dealuminated zeolite Y. The dealumination process involves three dealumination variables, they are dealumination temperature, the concentration of the sulfuric acid solution (H_2SO_4), and time of dealumination. Observed characterization of the changes in dealuminated zeolite Y includes two things, the amount of zeolite Y acidity and zeolite Y morphology. The amount of zeolite Y acidity can be determined through the total acidity of zeolite Y and surface acidity of zeolite Y, while the morphology of zeolite Y can be determined by analyzing the surface area of zeolite Y.

Effect of Dealumination on the Acidity of Zeolites Y

The acidity of dealuminated zeolite Y can be determined by conducting an ammonia and pyridine absorption test. The ammonia absorption test validates the value of total acidity of zeolite Y and the pyridine absorption test determines the amount of surface acidity of zeolite Y [5]. The results of the dealuminated zeolite Y acidity test can be seen in **TABLE 1**.

TABLE 1. Total and Surface Acidity based on the dealumination of Zeolite Y

Run	Temperature (˚C)	Concentration (N)	Time (hour)	Total Acidity (mol/gram)	Surface Acidity (mol/gram)
1	50	5.5	4	0.0167	0.0011
2	60	8	3	0.02	0.0016
3	60	3	3	0.0061	0.000091
4	66	5.5	4	0.0173	0.0015
5	60	3	5	0.006	0.000081
6	33	8	5	0.0136	0.0004
7	40	8	5	0.0148	0.0011
8	60	8	5	0.0142	0.0006
9	40	3	5	0.0127	0.00012
10	40	3	3	0.0132	0.0005
11	50	9.1	4	0.0247	0.0024
12	50	5.5	5.8	0.0088	0.000099
13	33	5.5	4	0.0065	0.0000765
14	50	1.1	4	0.0064	0.0000778
15	50	5.5	2.2	0.0076	0.000095
16	40	8	3	0.0151	0.0009
17	non dealumination			0.0031	0.00006

The catalyst acidity is the result of the number of moles per unit weight or unit surface area of the catalyst. The catalyst acidity test is carried out to determine the number of acidic sites that exist on the catalyst, both on the whole catalyst body and the catalyst surface. Ammonia absorption tests able to determine the total number of acid sites with ammonia as an adsorbate base. Ammonia gas molecules that are relatively basic in size are smaller than the zeolite Y catalyst cavity and it is very possible for the ammonia molecule to enter into all acidic catalyst cavities [5]. Therefore, ammonia not only interacts with the acidic sites on the surface but also with the acidic sites in the catalyst cavity, which is called total acidity.In the pyridine absorption test, pyridine as an alkali adsorbate will be able to determine the amount of acidity of the catalyst surface. Pyridine molecules that are basic in nature are relatively larger than the catalyst cavity so that pyridine will enter only reaching the acidic sites on the catalyst surface [5].

Based on these results, it can be compared that the acidity of dealuminated zeolite Y and without dealumination. The dealuminated zeolite Y catalyst in all variables has a total acidity value and surface acidity greater than the total acidity and surface acidity value on the zeolite Y catalyst without dealumination. This can occur because the zeolite Y catalyst which has been dealuminated has more active side as a result of the reduction of alumina through the dealumination process in zeolite Y [6]. Furthermore, the reduction of alumina causes a larger pore size on the catalyst thus allowing greater absorption of ammonia and pyridine gas into the dealuminated zeolite Y catalyst [7].

Effect of Dealumination on the Surface Area of Zeolites Y

The surface area of dealuminated and without dealuminated of zeolite Y can be identified by characterizing the Brunauer-Emmett-Teller (BET) test. The BET test can be used to determine the amount of surface area on a zeolite Y catalyst [8]. The results of the BET test for surface area values on the zeolite Y catalysts can be seen in **TABLE 2**.

TABLE 2. Surface Area of Zeolite Y Catalyst

Run	Temperature (°C)	Concentration (N)	Time (Hour)	Surface Area (m^2/g)
1	50	5.5	4	314.562
2	60	8	3	63.631
3	60	3	3	67.827
4	66	5.5	4	66.045
5	60	3	5	43.981
6	33	8	5	40.125
7	40	8	5	69.624
8	60	8	5	50.651
9	40	3	5	41.062
10	40	3	3	54.454
11	50	9.1	4	58.908
12	50	5.5	5.8	49.998
13	33	5.5	4	58.479
14	50	1.1	4	40.875
15	50	5.5	2.2	66.043
16	40	8	3	44.505
17	non dealumination			668.694

Based on the results of the surface area obtained that dealuminated zeolite Y has a smaller surface area compared to the zeolite Y without dealumination, This phenomenon has similarities with research conducted by [9], where the results of zeolite Y which has been dealuminated has a smaller surface area compared to zeolite Y without dealumination.

Changes in the surface area of both treatment on zeolite Y proves that there is a change in the structure of the zeolite Y catalyst. This structural change occurs due to changes in the pore size of the zeolite Y catalyst caused by the dealumination process. The acidity of the dealuminated zeolite Y catalyst to be greater than the initial zeolite Y catalyst (**TABLE 1**). This is caused by a change in the pore size of the dealuminated zeolite Y. With the dealumination process, the pore size is larger so that ammonia and pyridine gas can more easily enter the pores. Large pore size changes are shown by the surface area of the dealuminated catalyst is smaller than catalyst without dealumination. In general, particles that have smaller pores have larger specific surface areas. The small pore size indicates the number of small surfaces or very small capillary cavities in the catalyst body. These very small capillary cavities in the zeolite Y catalyst open into larger cavities due to a dealumination process that can eliminate various minerals, especially alumina in the catalyst, so the amount of catalyst surface area is reduced from the previous size. From this statement, it can be explained that the smaller surface area on the dealuminated zeolite Y catalyst happens due to the greater pores of the catalyst.

CONCLUSION

The dealumination process greatly affects the acidity and morphology of the zeolite Y catalyst. The catalyst that has been dealuminated will change structurally by opening the capillary cavity in the catalyst and increasing the amount of acidity due to the greater active sites on the catalyst.

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