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Effect of Calcination on Mechanical Strength Characteristics of Pellet Catalysts from Bandung Natural Zeolite Materials

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Abstract. The use of heterogeneous catalysts in the form of pellets can overcome the problems that occur in powder heterogeneous catalysts and homogeneous catalysts. Synthetic heterogeneous catalysts experience an increasing trend in prices due to increased demand. Natural materials such as natural zeolite have the potential to be used as catalysts. Catalyst pellets can have good performance in terms of activity, selectivity, and stability if they have adequate mechanical strength according to the process in which the catalyst will be used. The mechanical strength of the pellets can be obtained by considering the manufacturing process, manufacturing method, and catalyst raw materials. In this study, a study was conducted on the utilization of Bandung natural zeolite material to be used as catalyst pellets. The production of zeolite pellets was carried out by crushing, sifting 250 mesh, and non-calcining and calcining treatment at 550 C for 3 hours. Manufacture of the catalyst with a 5mm dies diameter with a compaction pressure of 1.5 tons. The pressure strength test with ASTM D4719 standard and the soluble resistance test on the catalyst with variations in time of 30 minutes, 60 minutes, and 90 minutes were applied. The results showed that the calcination treatment on the catalyst made the compressive strength value of the catalyst lower than the variation without calcination. The calcination treatment of Bandung natural zeolite pellet catalyst can increase the resistance to dissolve in the fluid by 73.03 %.

INTRODUCTION

Indonesia's geological condition which is at the meeting point of the continental plate and oceanic plate (subduction zone) makes Indonesia rich in mineral resources. Various types of minerals can be found, including zeolite rock minerals. Zeolite is a type of porous crystalline natural rock that usually consists of Si, Al, and O atoms, and is a catalytic material that can be widely applied. Natural zeolite is a porous material with good physicochemical properties, such as high cation exchange capacity, cation selectivity, and large pore volume [1]. One of the uses of natural zeolite rock is for the manufacture of catalysts. A catalyst is a chemical compound that causes a reaction to reach equilibrium more quickly without undergoing a chemical change at the end of the reaction. The catalyst does not change the equilibrium value and plays a role in lowering the activation energy. Zeolite catalysts have been widely developed and applied for industrial applications [2]. Catalysts are divided into two, namely homogeneous catalysts and heterogeneous catalysts. Homogeneous catalysts are catalysts that are soluble in reactants and/or reaction products. Meanwhile, heterogeneous catalysts are insoluble in reactants and/or reaction products [3]. Research on the potential of catalysts to react transesterification of oil into biodiesel, both homogeneous and heterogeneous catalysts are used to speed up the process [4]–[6]. Previous studies reported that the application of homogeneous catalysts has

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high catalytic activity but cannot be reused, the cost of separation makes it too expensive, high corrosion properties, and the residue can have a negative impact [7], [8]. In addition, heterogeneous catalysts have lower catalytic activity than homogeneous catalysts, but heterogeneous catalysts are very environmentally friendly, have low fabrication costs, and can be reused several times with simple separation and recovery [6], [9]. Catalysts in powder and liquid form can undergo rapid deactivation, which negatively affects the performance of the catalyst. In the use of liquid catalysts, it is difficult to separate the initial product from the final product, so further processing is required if the liquid catalyst is to be separated from the initial product. The use of pellet-shaped catalyst products can overcome the problems that occur in homogeneous catalysts and powder catalysts and also can produce catalysts with various advantages and uniqueness, commercially pellet-shaped catalysts can provide mechanical stability, varied shapes, repeated use, and the control function are easier. The mechanical strength of the pellets can be obtained by observing the manufacturing process, manufacturing method, and catalyst starting material. The mechanical strength of a zeolite catalyst depends on the strength of its components, the texture of its secondary structure, and the method of synthesis. When selecting heterogeneous catalysts for industrial use, mechanical strength and reliability are important operational characteristics, which are included in the list of parameters to be considered.

Calcination is an important part of catalyst preparation. The calcination process can change the chemical valence of the catalyst, the crystal phase structure, the specific surface, and the pore structure [10]. Research [11], [12] showed that pretreatment of calcination can cause changes in zeolite such as decreased crystallinity and greater amorphous content which may be beneficial for zeolite reactivity. The calcination process usually uses heating with a temperature range of 300°C-950°C and with a time span of 2-5 hours. The calcination process can result in a loss of crystallinity in the zeolite and mineral impurities, creating a better amount of amorphous reactive material. The literature shows that the crystal structure of zeolite becomes unstable after calcination at 300°C [13], [14]. Research conducted by [15] concluded that calcination resulted in a decrease in the crystallinity of zeolite. This decrease in zeolite crystallinity is directly proportional to the significant increase in amorphous content when the temperature reaches more than 500°C, so calcination at temperatures higher than 500°C is required to reduce the crystallinity of natural zeolite.

This study aims to determine the potential utilization of Bandung natural zeolite material to be used as catalyst pellets that have good mechanical strength and good fluid soluble resistance. Zeolite will be distinguished by the presence of non-calcined and calcined treatment at 550°C for 3 hours.

METHOD

Research Variables

The fixed variables in this study were natural zeolite from Bandung, 250 mesh sieving, 5 mm pellet diameter, and 1.5-tonne compaction pressure. The independent variables in this study were calcined and non-calcined variations. Calcination was carried out at 550°C for 3 hours. The outcome variables in this study were the value of the crushing strength test and the solubility test.

Pelletizing and Test

Zeolite in the form of stone is ground to a powder. The natural zeolite powder was then dried in an oven to remove the moisture content at 110°C for 3 hours. The dried zeolite powder was sieved with a size of 250 mesh (63 microns). Zeolite powder was then divided into non-calcined groups and calcined at 550°C for 3 hours. The powder is ready to be molded into pellet form. Pelletizing is done by compaction method on dies with a diameter of 5 mm. Compaction is carried out with a pressure of 1.5 tons for 20 seconds.

The compression test uses the side crush strength method with a single pellet test. The standard used is ASTM D4719. The single zeolite catalyst pellet was compressed between two rigid plates, using a mechanical testing device at a constant speed, with a compressive speed of 0.5 mm/min. The maximum force and compression strength before breakdown were recorded as SCS values with newton (N) and MPa values.

The dissolution time test was carried out to obtain the value of the dissolution rate (grams/minute). Zeolite pellets were placed in a dissolution apparatus with water fluid which was given a magnetic stirrer with a rotation of 250 rpm and the water temperature was set at 55°C. The initial mass before being tested was measured, then the mass after treatment for 30 minutes, 60 minutes, and 90 minutes was also measured by previously drying in the oven at 105°C for 1 hour.

RESULTS AND DISCUSSION

In the compression test, the graph displays each test and the results of the maximum force and compression strength are displayed. In the soluble test, the design results obtained data for the dissolving speed of each pellet tested. In this test, pellets with uniform specifications were selected, namely 250 mesh and 1.5 tons of compaction

Physical Measurement

Table 1 and Table 2 present the measurement data on the physical specifications of catalyst pellets from Bandung natural zeolite that have been made, both calcined and non-calcined.

TABLE 1. Physical Measurement of Non-Calcined Variations

Number	**Diameter (mm)**	**High (mm)**	**Volume (mm^3)**	**Massa (g)**	**Density (g/mm^3)**
1	5,36	4,20	94,7695	0,1419	1,49 x 10-3
2	5,24	4,46	96,1806	0,1488	1,54 x 10-3
3	5,34	4,44	99,4387	0,1497	1,50 x 10-3
Average	5,31	4,37	96,7963	0,1468	1,51 x 10-3

TABLE 2. Physical Measurement of Calcined Variations

Number	**Diameter (mm)**	**High (mm)**	**Volume (mm^3)**	**Massa (g)**	**Density (g/mm^3)**
1	5,10	5,20	106,2267	0,1558	1,46 x 10-3
2	5,10	5,60	114,3980	0,1658	1,44 x 10-3
3	5,10	5,70	116,4408	0,1715	1,47 x 10-3
Average	5,10	5,50	112,3551	0,1643	1,46 x 10-3

From the results of dimensional measurements, different results were obtained between those that were calcined and those that were not. The difference in dimensions is caused by the process to remove the pellet product from the dies and the absence of additional material that functions as a lubricant. Friction between the material and the wall of the die causes deformation of the pellet shape. The difference in composition after calcination indicates that there is an effect on changes in pellet shape.

For density, random data was obtained, but if it was averaged from the samples taken, it was obtained that the trend without calcination process, was greater in density. The impurities that normally fill the pores may be still present, while the calcined material has disappeared

Compression Test

From the results of the crushing strength test using the ASTM D4179 method that can be seen in Figure 1 and Table 3, data obtained that the uncalcined Bandung natural zeolite catalyst pellet has greater strength than the calcined, this is in line with the data on the density of the uncalcined catalyst pellet which is also higher than the calcined catalyst pellet. The presence of more pores results in a reduced density and lowers the strength of the calcined catalyst pellet.

The compressive strength of the calcined variant tends to be stable/constant, in contrast to the non-calcined variant which tends to fluctuate. Calcination of zeolite can change the crystal structure in the form of increasing the amorphous content in it [16], the amorphous structure can reduce the strength of the material because of its irregular shape. Calcination at 550°C for 3 hours for Bandung natural zeolite catalyst changed the crystal structure to an amorphous structure so that the compressive strength decreased compared to the uncalcined variant.

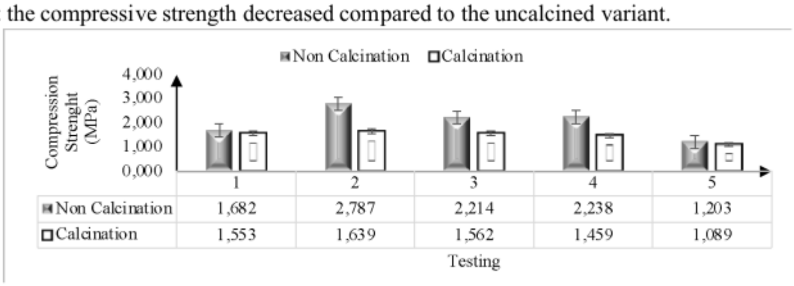

FIGURE 1. Compression Strength Variation of Non-Calcined and Calcined.

TABLE 3. Compression Test Results of Non-Calcined and Calcined Variations.

Method	Number	Force Peak (N)	Compression Strenght (MPa)
	1	35,716	1,682
	2	59,193	2,787
Non-Calcination	3	47,013	2,214
	4	47,523	2,238
	5	25,556	1,203
	Average	51,243	2,413
	1	32,990	1,553
	2	34,814	1,639
Calcination	3	33,166	1,562
	4	30,989	1,459
	5	23,124	1,089
	Average	33,657	1,585

Soluble Time Test

Table 4 and Table 5 present data on the results of the uncalcined and calcined uncalcined and calcined soluble natural zeolite catalyst pellets in Bandung. The uncalcined catalyst pellets had mass-loss rates of 2.2%, 28%, and 32.4% for 30 minutes, 60 minutes, and 90 minutes, respectively. The mass-loss rates for calcined pellets were 4.7%, 5.2%, and 4.8% with the same time variance as those without calcined. The average results of the weight loss value and the rate of dissolution rate on the catalyst pellets from the entire test time, for the non-calcined variant pellets, have an average weight loss value of 3.57 x 10^{-2} grams and a dissolution rate of 5.97 x 10 gram/minute. The calcined variant pellets had an average weight loss value of 0.81 x 10^{-2} grams and a dissolution rate of 1.61 x 10^{-4} grams/minute.

From the results of the solubility test, it can be seen that the pellet catalyst without calcination has a higher level of solubility in the liquid than the calcined one, this is possible due to the presence of dissolved impurities and conditions where the hydrophobic nature of the material without calcining is lower. The hydrophobic nature is influenced by the value of the Si/Al ratio which can be obtained from the XRF test results. The higher the value of the Si/Al ratio, the hydrophobic properties will increase. From the XRF test results obtained data that the calcination at a temperature of 550 c for 3 hours did not change the composition much, but there was a change in the Si/Al ratio, from 7.5 to 7.7.

TABLE 4. Test Results of Non-Calcinated Variation Solubility Test.

Testing Time	Number	First Mass M_1 (g)	Last Mass M_2 (g)	M_1-M_2 (g)	% of Soluble Mass	Soluble Rate (g/minutes)
	1	0,1538	0,1362	0,0176	11,4%	5,87 x 10^{-4}
30 Minute	2	0,1305	0,1135	0,0170	13,0%	5,67 x 10^{-4}
	3	0,1415	0,1261	0,0154	10,9%	5,13 x 10^{-4}
	Average	0,1419	0,1253	0,0167	11,8%	5,56 x 10^{-4}
	1	0,1530	0,1301	0,0229	15,0%	3,82 x 10^{-4}
60 Minute	2	0,1370	0,0834	0,0536	39,1%	8,93 x 10^{-4}
	3	0,1563	0,1075	0,0488	31,2%	8,13 x 10^{-4}
	Average	0,1488	0,1070	0,0418	28,4%	6,96 x 10^{-4}
	1	0,1530	0,1224	0,0306	20,0%	3,40 x 10^{-4}
90 Minute	2	0,1626	0,0818	0,0808	49,7%	8,98 x 10^{-4}
	3	0,1335	0,0990	0,0345	25,8%	3,83 x 10^{-4}
	Average	0,1497	0,1011	0,0486	31,8%	5,40 x 10^{-4}
	Average of All	**0,1468**	**0,1111**	**0,0357**	**24,0%**	**5,97 x 10^{-4}**

TABLE 5. Test Results of Calcinated Variation Solubility Test.

Testing Time	Number	First Mass M_1 (g)	Last Mass M_2 (g)	M_1-M_2 (g)	% of Soluble Mass	Soluble Rate (g/minutes)
30 Minute	1	0,1575	0,1495	0,0080	5,1%	2,67 x 10^{-4}
	2	0,1549	0,1481	0,0068	4,4%	2,27 x 10^{-4}
	3	0,1550	0,1477	0,0073	4,7%	2,43 x 10^{-4}
	Average	0,1558	0,1484	0,0074	4,7%	2,46 x 10^{-4}
60 Minute	1	0,1517	0,1439	0,0078	5,1%	1,30 x 10^{-4}
	2	0,1526	0,1449	0,0077	5,0%	1,28 x 10^{-4}
	3	0,1930	0,1825	0,0105	5,4%	1,75 x 10^{-4}
	Average	0,1658	0,1571	0,0087	5,2%	1,44 x 10^{-4}
90 Minute	1	0,1681	0,1604	0,0077	4,6%	8,56 x 10^{-5}
	2	0,1537	0,1454	0,0083	5,4%	9,22 x 10^{-5}
	3	0,1926	0,1837	0,0089	4,6%	9,89 x 10^{-5}
	Average	0,1715	0,1632	0,0083	4,9%	9,22 x 10^{-5}
	Average of All	**0,1643**	**0,1562**	**0,0081**	**4,9%**	**1,61 x 10^{-4}**

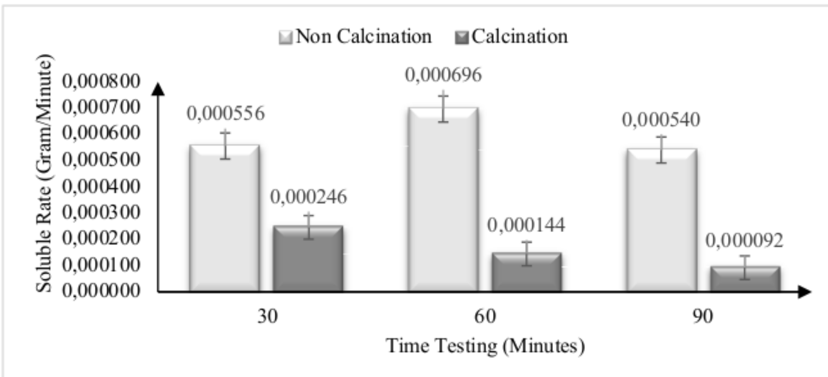

FIGURE 2. Comparison Diagram of the Percentage of Solubility Test Results of Dissolution Time of Non-Calcinated and Calcined Treatment Methods.

CONCLUSION

Based on the results of compression testing and dissolving time of natural zeolite catalyst pellets from Indonesia with compressive strength and dissolution rates, it was concluded that the load that was accepted by the uncalcined natural zeolite pellets from Bandung was greater than the calcined pellets.

The calcination process affects the mechanical strength of Bandung natural zeolite pellets, where the calcination treatment of 550°C for 3 hours makes the compressive strength value decrease compared to non-calcined which is 34.49%. The calcination process causes changes in the composition of the main elements and impurities in the zeolite material and can affect the strength of the pellet in the fluid, as evidenced by the calcination treatment of Bandung zeolite catalyst pellets can increase the mechanical strength at the value of the dissolution rate of the catalyst in the fluid by 73.03%.

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