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Kinetic Studies of Preparation of ZSM-5 Catalyst

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Abstract: ZSM-5 is a synthetic zeolite which contains silica (Si) and alumina (Al) with the ratio of silica greater than the alumina. The catalyst is known as ZSM-5 because it has a pore diameter of 5^0A (angstroms) and it has more than five Si/Al ratios. The catalyst has a high production cost and a complex production process which is highly influenced by time and temperature. Based on that fact, the focus of this research is studying the production process of ZSM-5 and investigating the effect of time and temperature on the crystallinity and the morphology of ZSM-5. In this research, Latourette *et al*. method was used, followed by the calcination at different temperatures (500 °C, 600 °C, 700 °C, 800 °C) in 5 and 7 hours. Subsequently, ZSM-5 was characterized using XRD (X-ray Diffraction) and SEM (Scanning Electron Microscope). Calcination time affects the XRD peak intensities by increasing the ZSM-5 crystal cores. The longer the calcination time, the higher the peak intensity.

Keywords: ZSM-5, Calcinations, Complex production, Crystallinity

I. INTROUCTION

Cracking is a process that is widely used in research and industry, especially in hydrocarbon-related ones. However, the hydrocracking process traditionally requires high operating temperatures and can cause environmental problems. Therefore, the use of transition metals, silica, zeolite-modified zeolite, mesoporous mixed oxide catalyst, and metal organic frameworks, was developed in the catalytic cracking process [9]. The catalytic cracking process can break down complex hydrocarbons to simpler ones at low temperature and pressure [11]. Zeolite is the best catalyst for oil cracking process because it has a high thermal stability, has microcrystalline structure, and able to concentrate the reactants that are contained in the pores [9]. Synthesis of ZSM-5 catalyst (Zeolites Socony Mobil-5) was first performed by Argauer and Landolt which is patented by Mobil Oil Corporation in 1972 [1]. ZSM-5 is a synthetic zeolite which contains silica (Si) and alumina (Al) with the ratio of silica greater than the alumina. The existence of aluminium and Si/Al ratio of ZSM-5 catalyst causes ZSM-5 to have acidic property [6]. This property makes the catalyst has the function as a catalyst support, adsorbent, etc. [7]. It has pore diameter range of 5 Å (angstroms) and it has more than five Si/Al ratio. ZSM-5 has a medium pore size (0.54 nm × 0:56 nm) and has two intersecting three-dimensional channels with one straight parallel channel and the other running parallel defined by sinusoidal 10-membered ring openings of 5.3 Å × 5.6 Å and 5.1 Å × 5.5 Å [9].

ZSM-5 catalyst can be used to manufacture a variety of products through a catalytic cracking process. Types of products that can be produced by ZSM-5 catalyst are influenced by physical properties of the catalytic material and constituent of ZSM-5 catalyst. There are several parameters that can affect the properties of ZSM-5 catalysts. These parameters are the silica source, the alumina source, template, and crystallization conditions [5].

In this research, synthesis of ZSM-5 catalyst with Latourette et al. method [4] at the various temperatures, calcination time, and catalyst supports was conducted. After that, the produced catalysts were analysed by using SEM and XRD to understand their characteristics.

II. STRUCTURE OF ZSM-5

ZSM-5 is composed of several pentasil units linked together by oxygen bridges to form pentasil chains. A pentasil unit consists of eight five-membered rings. In these rings, the vertices are Al or Si and an O is assumed to be bonded between the vertices. The pentasil chains are interconnected by oxygen bridges to form corrugated sheets with 10-ring holes. Like the pentasil units, each 10-ring hole has Al or Si as vertices with an O assumed to be bonded between each vertex. Each corrugated sheet is connected by oxygen bridges to form a structure with "straight 10-ring channels running parallel to the corrugations and sinusoidal 10-ring channels perpendicular to the sheets." Adjacent layers of the sheets are related by an inversion point. The estimated pore size of the channel running parallel with the corrugations is 5.4–5.6 Å. The crystallographic unit cell of ZSM-5 has 96 T sites (Si or Al), 192 O sites, and a number of compensating cations depending on the Si/Al ratio, which ranges from 12 to infinity. The structure is orthorhombic (space group Pnma) at high temperatures, but a phase transition to the monoclinic space group P21/n.1.13 occurs on cooling below a transition temperature, located between 300 and 350 K. ZSM-5 catalyst was first synthesized by Argauer and Landolt in 1969 [8].

III. METHODOLOGY

3.1 Materials

Materials that are used in this research are: Aluminium Sulphate (Al2(SO4)3.18H2O), Cobalt Nitrate (Co(NO3)2.6H2O), Nickel Nitrate (Ni(NO3)2.6H2O), Sulfuric Acid 98% (H2SO4), Sodium Silicate and Sodium Hydroxide (both were supplied by Merck Ltd.), distilled water (obtained from Laboratory of Membrane Research Centre Diponegoro University), Methanol, and Glycerol (both were supplied by Indrasari Chemical Store).

3.2 Catalyst synthesis

Catalyst was synthesized according to Latourette et al. [7] method. Firstly, Solution A as the source of alumina was prepared. The solution contains 26.7 grams of aluminium sulphate (Al2(SO4)3.18H2O), 56 grams of 98% Sulfuric Acid (H2SO4), and 15 grams of distilled water. After that, Solution B as the source of silica was prepared. The solution contains 56 grams of Sodium Silicate and the same amount of 40% Sodium Hydroxide (NaOH). Subsequently, Solution A and Solution B were mixed. After that, the mixture was homogenized by a homogenizer at a speed of 12000 rpm. After obtaining a homogeneous mixture, crystallization is done by using an autoclave reactor at 200 °C in 6 and 8 hours. After the crystallization, ZSM-5 catalysts as products were washed with distilled water and then dried overnight. The ZSM-5 products crystals were analysed by using XRD to determine their degree of crystallinity. After that, ZSM-5 undergone calcination process at various temperatures of 500 °C, 600 °C, 700 °C and 800 °C in 5 and 7 hours. After this process, the product was analysed using XRD to determine their crystallinity and their catalyst's structure. SEM analysis was also conducted to find out the morphology of the catalysts.

3.3 Catalyst characteristic

The ZSM-5 catalyst products were characterized with morphology and crystallinity analysis. The analysis of crystallography conducted by using XRD-7000S Shimadzu with Copper X-ray tube target, 30 kV voltage, 30 mA current, and Kα radiation. XRD data was analyzed with PCXRD software. Morphology of catalyst data was analyzed with JEOL PC Scanning Electron Microscope (PCSEM) model JSM-6510LA with x5000 magnification. These analysis processes were conducted in Center of Research and Service Diponegoro University (CORES DU).

IV. RESULTS AND DISCUSSIONS

4.1 The effect of crystallization time

In the production of the ZSM-5 catalyst variations of crystallization time of 6 and 8 hours was performed. Crystallization time is one parameter that plays an important role on the properties of the catalyst since the catalyst products were harvested at different times. During the variation of crystallization time, the crystallization takes place at temperatures of 200 °C[12].

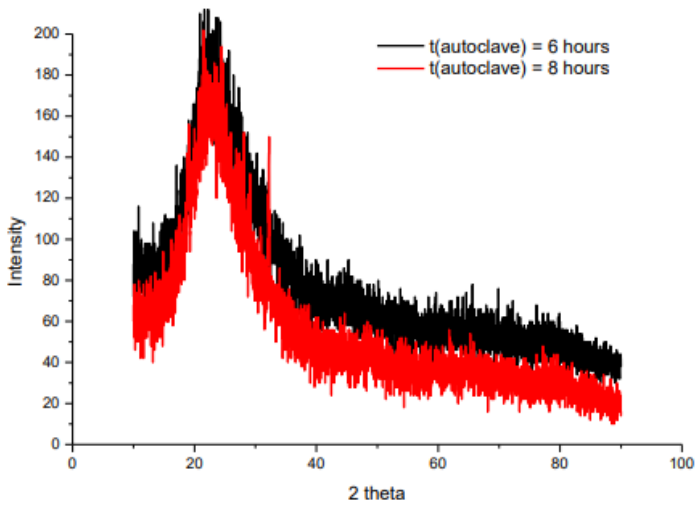

Figure 1.XRD pattern of ZSM-5 Synthesis on difference crystallization times.

Figure 1 shows the results of the XRD analysis of the ZSM-5 catalyst in the autoclave for 6 and 8 hours. The graph shows that the longer the crystallization time, the more compact the catalyst structure is. This fact can be seen from the intensity at 2 theta degree 21 and 23. Furthermore, the long crystallization time also affects the degree of crystallinity [12].

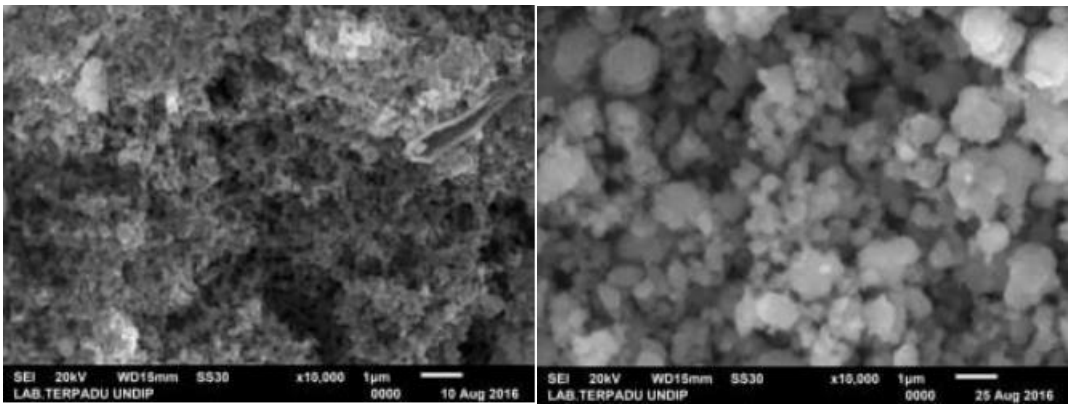

Figure 2.Photographs of ZSM-5 catalyst (a. Synthesized; b. Standard ZSM-5)

Figure 2 describes a pentasil unit consists of eight five-membered rings. In these rings, the vertices are Al or Si and an O is assumed to be bonded between the vertices. The pentasil chains are interconnected by oxygen bridges to form corrugated sheets with 10-ring holes. Like the pentasil units, each 10-ring hole has Al or Si as vertices with an O assumed to be bonded between each vertex. Each corrugated sheet is connected by oxygen bridges to form a structure with "straight 10-ring channels running parallel to the corrugations and sinusoidal 10-ring channels perpendicular to the sheets.

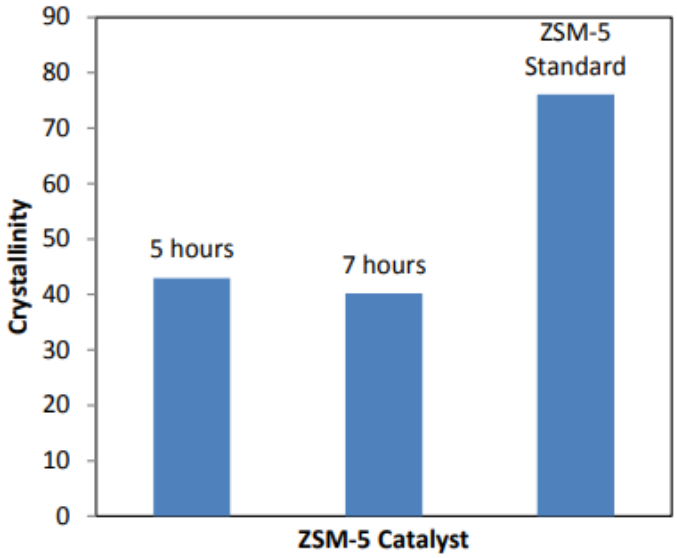

Figure 3. ZSM-5 crystallinity in difference calcination times [12]

V. CONCLUSION

In conclusion, by the Latourette et al. method it can be resulted that, crystallization time affects the physical properties of ZSM-5 catalyst. In addition, the calcination temperature on ZSM-5 catalyst production also affects the crystallinity of the catalyst. The higher the temperature is, the better the catalyst's The higher the temperature is, the better the catalyst's Crystallinity. Subsequently, ZSM-5 was characterized using XRD (X-ray Diffraction) and SEM (Scanning Electron Microscope). Calcination time affects the XRD peak intensities by increasing the ZSM-5 crystal cores. The longer the calcination time, the higher the peak intensity.

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