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The Effect of Metal Impregnation of Fe, Cu, and Co on Surface Area of Zsm-5 Catalyst Analyzed Using Surface Area Analyzer (SAA)

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Abstract: *ZSM-5 is a heterogeneous catalyst commonly used for petroleum cracking reactions and gas conversion processes, ZSM-5 has a pore size of 0.54 nm × 0.56 nm with a surface area of 306.178 m^2/g. To improve the performance of the ZSM-5 catalyst, it is necessary to impregnate it so that the surface area of the catalyst becomes smaller. One of the impregnation methods used is wet impregnation with the added metal of 2.5% wt, then characterized by using a Surface Area Analyzer (SAA). The results of the analysis reveled that the surface area of the ZSM-5 catalyst impregnated with Fe, Cu, and Co, was 291,853 m^2/g, 302,390m^2/g, and 291,504m^2/g, respectively. The diameter of the pores of the ZSM-5 catalyst impregnated with Fe, Cu, and Co were 3.3896nm, 3.2688nm, and 3.2790nm, and the total volume of the pores of the ZSM-5 catalyst impregnated with Fe, Cu and Co metals, are 0.24732 cc/g, 0.24712cc/g, and 0.23896 cc/g. Therefore, the addition of Fe, Cu, and Co metals on the surface of the ZSM-5 catalyst will reduce the surface area of the catalyst, reduce the total pore volume and reduce the diameter of the pores of the ZSM-5 catalyst.*

Keywords: *ZSM-5; Impregnation; Surface areas; Total pore volume and pore diameter.*

INTRODUCTION

Zeolites are widely used in the petrochemical industry as heterogeneous catalysts, especially in petroleum cracking reactions and gas conversion processes. One of the zeolites applied in the reaction is ZSM-5 (Krisnandi et al., 2012). ZSM-5 is a synthetic zeolites containing silica (Si) and aluminum (Al) with a higher Si content in ZSM-5 compared to Al, thus making the ZSM-5 catalyst have acidic properties. The acidic nature of the ZSM-5 catalyst has functioned as support as an adsorbent. ZSM-5 has a diameter of about 5Å, and ZSM-5 has a pore size of 0.54nm × 0.56nm (Widayat dan Annisa, 2017). The size of ZSM-5 < 2 nm belongs to the microporous structure which makes this material have a large surface area.

Small pore size will have a positive and negative impact. The positive impact given by the micro-sized catalyst is that the ZSM-5 catalyst has high selectivity while the negative impact given is the speed of access of the adsorbed molecules into the ZSM-5 material being low due to the size being too small (Krisnandi et al., 2012). To overcome this the researchers made ZSM-5 have a mesoporous size (2-50nm). Mesoporous type ZSM-5 has a large surface area, large porosity, and good hydrothermal stability and can be applied at high temperatures (Wang et al., 2010). The large surface area of the catalyst affects the speed of the reaction. The application of ZSM-5 as a catalyst has several

advantages including good adsorption capacity, high thermal stability, and good Lewis and Bronsted Lowry ion exchange capabilities.

Impregnation is generally defined as the total saturation of certain substances; this saturation aims to fill the pores of the catalyst with active metal by immersing the catalyst in a solution containing the metal. The function of the catalyst is to provide a large surface area so that it is easier to spread the active site and wider contact surface (Permana et al., 2020). Fe, Cu, and Co metals are selective metals for the oxidation of methane to methanol where these three types of metals have an oxidation number of more than +2. Thus, applying this metal can form C1-Oxygenate (Mansouri et al., 2013). The purpose of adding metal to the catalyst surface is to improve the performance of the catalyst and to minimize the pores of the catalyst in order to increase selectivity.

The surface analyzer was applied to determine the surface of the ZSM-5 catalyst impregnated with Fe, Cu and Co metals by using the BET (Brunauer-Emmet-Teller) method. The BET theory explains the phenomenon of the adsorption of gas molecules on the surface of solids. If the solid is in the form of fine particles/powder, the surface area for the solid is greater and vice versa if the solid is in the form of coarse particles, the surface area for the solid is getting smaller. The BET method provides information on the specific surface therefore this method can be applied to estimate the average size of particulate solids. (Abdullah dan Khairurrijal, 2009).

BET relies on the adsorption of gaseous nitrogen into the sample. The amount of gas adsorbed is a function of the surface of the solid. The data from the Surface Area Analyzer is processed with various theories and calculation models developed by the researchers to convert them into data on the surface area, pore volume, pore radii, and so on. For example, the BET theory and the Langmuir theory may be applied to calculate the surface area of solids.

The surface area of the catalyst is measured based on the amount of nitrogen gas adsorbed on the monolayer, while the pore size is measured based on the gas pressure in the pores, the total surface area can be used as an indicator of the active site of the catalyst and the transport of reactants known as catalytic activity. In essence, BET characterization requires the material to be characterized to have pores, for example, zeolite, silica, alumina, ceramic and carbon. When a BET analysis is carried out on the surface area of the catalyst, only the volume of gas absorbed by the pores/solid surface will be measured under isotherm conditions. (Sudarlin, 2012).

This study purpose to examine how the effect of metal impregnation of Fe, Cu, and Co on the surface area, total pore volume and pore diameter of ZSM-5, the best results from this study were applied as candidates for the conversion of methane gas into methanol through partial oxidation reactions. The method of impregnation used for this study is wet impregnation.

RESEARCH METHODS

Materials and Tools

The materials used in this research are aquades, Co $(NO_3)_2$. $6H_2O$ (MERCK), Fe $(NO_3)_3$. $9H_2O$ (MERCK), Cu $(NO_3)_2$. $3H_2O$ (MERCK), nano ZSM-5-H (ACS Material), liquid nitrogen dan nitrogen gases (SAMATOR). hot plate (Wisd) and magnetic stirrer, oven (Memmert), furnace (Carbolite CWF 1000), 50 mL beaker glass (iwaki), stand and burette, analytical balance (and), spatula, porcelain cup, 10 mL measuring pipette (iwaki), a set of surface area analyzer instruments SAA (Quantachrome Nova Touch LX2 Series).

Methods

ZSM-5 Catalyst Impregnation with Fe, Cu and Co

The metal was impregnated with 2.5% wt of the mass of ZSM-5, and to obtain it, weighed 0.3699g of $Co(NO_3)_2.6H_2O$, then dissolved using 10 ml of distilled water. After that 3g of commercial ZSM-5 were weighed and drained with 1.5 ml of distilled water while stirring with a magnetic stirrer. After that, the $Co(NO_3)_2.6H_2O$ solution was put into the burette and a distinctive was installed, then the $Co(NO_3)_2.6H_2O$ solution was dripping from the burette slowly while continuing to stir. After all the $Co(NO_3)_2$. $6H_2O$ solution was used up in the burette, the mixture was continuously stirred for 24 hours at room temperature conditions, and the stirring rate was set at 150 rpm. The mixture that had been stirred for 24 hours was baked in the oven at 60^0C until the mixture was completely dry and a ZSM-5 catalyst impregnated with metal Co was obtained. Then the catalyst that formed is charred at 550^0C for 3 hours, the purpose is to activate the catalyst Co/ZSM-5. The same treatment was applied for 0.541g $Fe(NO_3)_2$. $9H_2O$ and 0.2853g Cu $(NO_3)_2.3H_2O$.

Sample preparation for SAA analysis

Some impregnated catalysts are added to the sample cell that was previously weighed. The cell sample used is round since the test sample is powdered. Each cell sample is labeled for port station 1 and 2, cell sample 1 is labeled (BE1) having a mass of 17.9201 and a catalyst that contains 0.1464g $Fe(NO_3)_2.9H_2O$. Cell sample 2 is labelled (BE2) with a mass of 19.0721g and the analysed sample is $Co(NO_3)_2$. $6H_2O$ of 0.1448g. As for the Cu/ZSM-5 catalyst samples, they were carried out separately because the SAA instrument could only analyze 2 samples. For the Cu/ZSM-5 sample, 0.9320g was used with a sample cell of 17.1990g.

After determining the mass of the catalyst to be analyzed, degaussing is done to remove the remaining gas from the catalyst. The degassing process is carried out by placing the sample cell in the degaussing port, after which the cell sample is wrapped in a heating mantle. The degassing process itself is carried out at a temperature of 300^0C for 3 hours.

Sample Analysis

After the degaussing process is complete, the filler road is inserted into the sample cell to close the powder sample so that it does not fly away when nitrogen gas is applied. The cell sample is mounted on the station-specific port analysis that has been tagged. When installing the sample cell on the port analysis, it must be perpendicular to the point that the sample cell is not broken. The diver is loaded with liquid nitrogen and placed on the automatic lift. Then, the analysis process is conducted with the parameters that have been configured on the monitor. When analyzing nitrogen gas is flowing to the analysis port and will be adsorbed on the sample.

RESULTS AND DISCUSSION

Impregnation in general can be defined as the saturation of the ZSM-5 catalyst in an activation metal solution, where this activation metal will fill the pores of the catalyst. Impregnation is conducted by soaking the catalyst for some time. To determine the surface area of the catalyst, the total pore volume and the radius of the pores, a surface area analyzer (SAA) instrument was applied. The surface area of the catalyst is measured based on the amount of nitrogen gas adsorbed on the monolayer section, while the pore size is measured based on the gas pressure in the pores, the total surface area can be used

as an indicator of the active side of the catalyst and reactant transport which is known as catalytic activity.

A B C

Figure 1. A. Co/ZSM-5 catalyst, B. Fe/ZSM-5 catalyst and C Cu/ZSM-5 catalyst

Figure 1 reveals the results of the impregnation of each sample where ZSM-5 impregnated with metal Co has a gray color while ZSM-5 impregnated with metal Fe has an orange color and ZSM-5 impregnated with metal Cu has a bluish-white color. The color that emerges from all samples comes from the impregnated metal. The reason for selecting these metals was based on research conducted by Chen et al (2010). Fe metal has the role of increasing the surface area of the catalyst and has high selectivity, while Co metal was chosen because it has a large surface area and has many species of Co_3O_4 and CoO that are selective for methanol (Beznis et al., 2011). Meanwhile, copper can oxidize methane to methanol at low temperatures but has low selectivity. ZSM-5 catalysts impregnated with Cu metal can increase the reaction rate at low temperatures (Narsimhan et al., 2016). The impregnated catalyst was analyzed with SAA; before being analyzed, the sample underwent a degassing process, which aims to remove gas trapped in the catalyst so that the catalyst is in a vacuum. After the degassing process is complete, the sample is weighed to determine the actual weight.

Table 1. The mass of the catalyst after the degassing process.

Sample	Catalyst mass before degassing (g)	Catalyst mass after degassing (g)	Loss of catalyst mass (g)
ZSM-5	0.1925	0.1732	0.0193
Fe/ZSM-5	0.1464	0.1285	0.0179
Cu/ZSM-5	0.2141	0.1910	0.0231
Co/ZSM-5	0.1448	0.1269	0.0179

From Table 1, there is a missing catalyst mass, indicating that the gas in the sample has disappeared and is ready to be analyzed. In addition to the gas lost from the sample, according to Sudarlin (2012), the degassing process is carried out by flowing nitrogen gas into the sample cell so that it will crowd out the impurities contained in the catalyst. Degassing is carried out according to the temperature of the material to be analyzed. If the temperature used is too high, it will cause the material to melt and damage the material so that it cannot be analyzed. If the degassing temperature is too low, the degassing process will be in vain, and there will be impurities in the material.

Table 2. Surface area of impregnated ZSM-5 catalyst.

Sample	Surface area (System calculation) (m^2/g)	Surface area (Manual calculation) (m^2/g)	Specific Surface Area (m^2/g)
ZSM-5 Komersial	306.178	306.173	1767.771
Cu/ZSM-5	302.390	302.388	1583.194
Co/ZSM-5	291.504	291.498	2297.116
Fe/ZSM-5	291.853	292.271	2271.230

From the table above, data is obtained in the form of the surface area of the ZSM-5 catalyst that is not impregnated with the impregnated ZSM-5 catalyst. The surface area calculated by the system and manual calculations reveals a less significant difference, which is 0.005 m^2/g. As a result, the surface area of the system calculation results can be said to be accurate. In the results of the analysis using the Quantachrome Nova Touch series surface area analyzer instrument, the non-impregnated surface area of the ZSM-5 catalyst was 306,178 m2/g. Based on information from the manufacturer of the commercially used H-ZSM-5 (ACS Material), it appears that the H-ZSM-5 belongs to the P-26 type.

Where the ZSM-5 catalyst, which was impregnated with Cu metal, had a surface area of 302,390 m^2/g. The ZSM-5 catalyst impregnated with Fe metal had a surface area of 291,853 m^2/g, and the ZSM-5 catalyst impregnated with Co metal had a surface area of 291,504 m^2/g. From Table 2, it can be observed that there was a decrease in the surface area of the catalyst impregnated with metal. For example, the ZSM-5 catalyst has a surface area of 306,178 m2/g, compared to 3,788 m^2/g for the Cu/ZSM-5 catalyst, which has a surface area of 302,390 m^2/g. According to Dewajani et al (2016), The more metal that is evenly distributed on the surface of the catalyst is expected to increase the specific surface area, but conversely, the increasing percentage of activating metal causes the surface area to decrease. This is due to the accumulation of metals at the pore mouths or pore channels or the activating metal being dispersed unevenly on the surface, causing less nitrogen gas to be adsorbed so that the specific surface area of the catalyst decreases with the increasing amount of metal.

From the results of the total surface area, the specific surface area can be determined. The specific surface area can be defined as the total surface area divided by the sample weight of the catalyst. From Table 2, it can be seen that the largest specific surface area is found in the ZSM-5 catalyst, which is impregnated with metal Co. The specific surface area reaches 2297.116 m^2/g, which indicates that the impregnation process with cobalt metal (Co) completely covers the surface of the ZSM-5 catalyst. The specific surface area describes the active surface on the catalyst that can be in direct contact with the reactants; the greater the specific surface of the catalyst, the greater the activity of the catalyst. (Sarifudin et al., 2020). The activity of the catalyst is strongly influenced by the surface area and acidity of the catalyst; however, a surface area that is too large will also have an impact on reducing the pore radius of the catalyst, so that the catalyst pores cannot be passed by the reactants (feed) in the adsorption process, resulting in interactions between the reactants. with the catalyst active sites on the catalyst surface being quantitatively reduced (Morina dan Sidjabat, 2009).

Table 3. Total pore volume, pore radius and pore diameter.

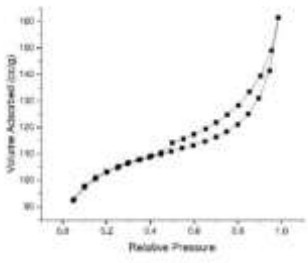
Sample	Total Pore Volume (cc/g)	Average Pore Radius (nm)	Average Pore Diameter (nm)
ZSM-5 Komersial	0.25049	1.6362	3.2724
Cu/ZSM-5	0.24712	1.6344	3.2688
Co/ZSM-5	0.23896	1.6395	3.2790
Fe/ZSM-5	0.24732	1.6948	3.3896

The total pore volume is obtained from the volume of nitrogen gas adsorbed at a certain pressure (Pednekar et al., 2017). Table 3 shows that the ZSM-5 catalyst impregnated with cobalt metal has the smallest total pore volume of 0.23896 cc/g and the largest total pore volume of Fe/ZSM-5, namely 0.24732 cc/g. The pore volume increases with the increase in the number of pores, and increasing the number of pores will increase the surface area of the catalyst (Aregawi, 2021).

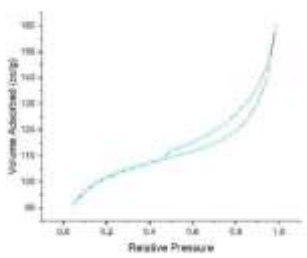
A large total pore volume will have high activity in product formation. The more volume a catalyst has, the more effective the reactants are at diffusing and being adsorbed into the pores of the catalyst, so that the bond-breaking reaction in the reaction is more effective (Morina dan Sidjabat, 2009) Therefore, the ZSM-5 catalyst impregnated with Fe metal has high catalytic activity in theory because it has a large total pore volume of 0.24732 cc/g when compared to the ZSM-5 catalyst impregnated with Cu and Co metals.

According to IUPAC itself, there are 3 classifications of porous materials, including (1) microporous materials with a pore size of 2 nm; (2) mesoporous or medium porous materials with pore diameters ranging from 2 nm to 500 nm; and (3) macroporous materials with a pore diameter > 50 nm. (Alothman, 2012). From the results of the analysis using the surface area analyzer brand Quantachrome Nova Touch series, the adsorption and desorption curves are presented in Figure 2.

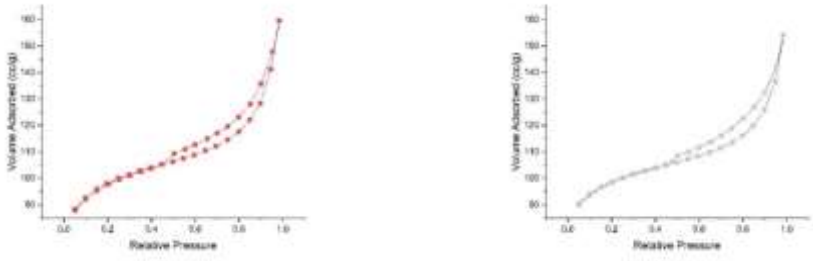
Figure 2 reveals that between each curve's adsorption and desorption, there is a space or gap known as a hysteresis loop. The literature-based classification of the aforementioned curve as type IV reveals that the ZSM-5 catalyst belongs to the mesoporous pore type, which contains pores with a diameter range of 2 nm to 50 nm. Complex pore systems in porous materials, notably pore networks shaped like ink bottles, prevent evaporation in the pores conducting through the equilibrium of open pores, causing delays because of restricted pore distribution that leads to capillary condensation. such that the evaporation that does occur slowly and makes the substance hysteretic (Cychosz dan Thommes, 2018).



A



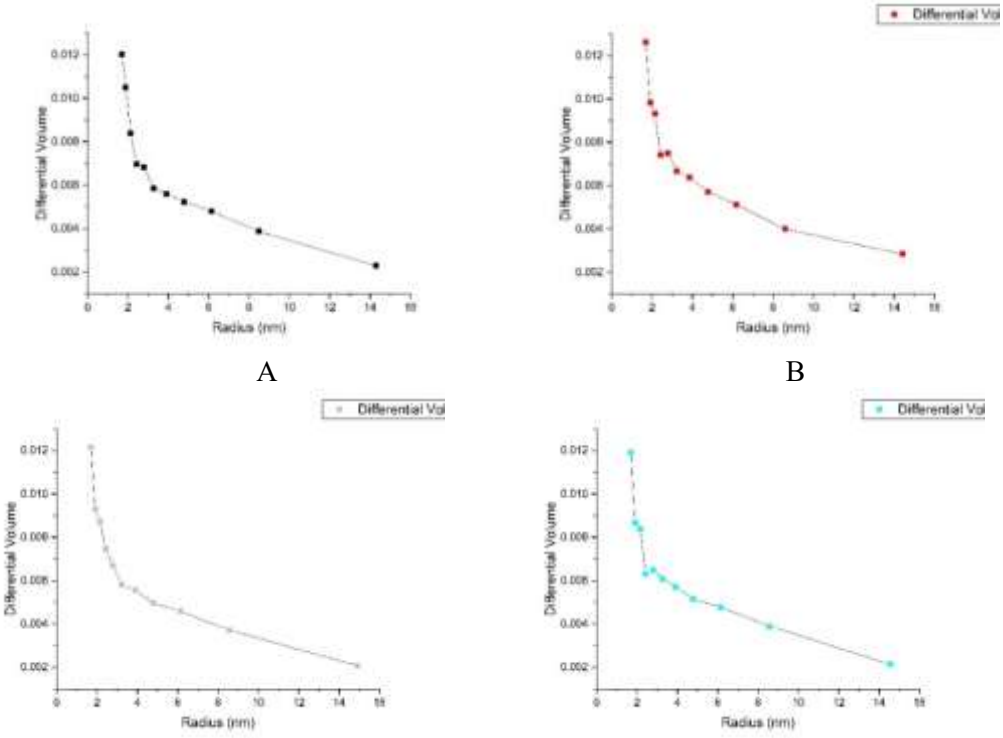
B



C D

Figure 2. A. Commercial ZSM-5 isotherm curve; B. ZSM-5 Isotherm Curve Impregnation With Cu metal; C. ZSM-5 isotherm curve of Fe impregnation; D. Isotherm curve of catalyst ZSM-5 impregnation with metal Co.

To determine the changes in the characteristics of the catalyst after the impregnation process, an analysis of the pore size distribution was carried out. In this case, the method used is Barret-Joyner-Halenda (BJH). From the distribution curve, it can be observed that the larger the pore volume, the smaller the pore radius, and the smaller the pore volume, the wider the pore radius. From the distribution curve, it can be observed that the larger the pore volume, the smaller the pore radius, and the smaller the pore volume, the wider the pore radius. From the radius and volume first derivative data, a BJH curve can be formed, and from the analysis results, a BJH curve is obtained for each catalyst.



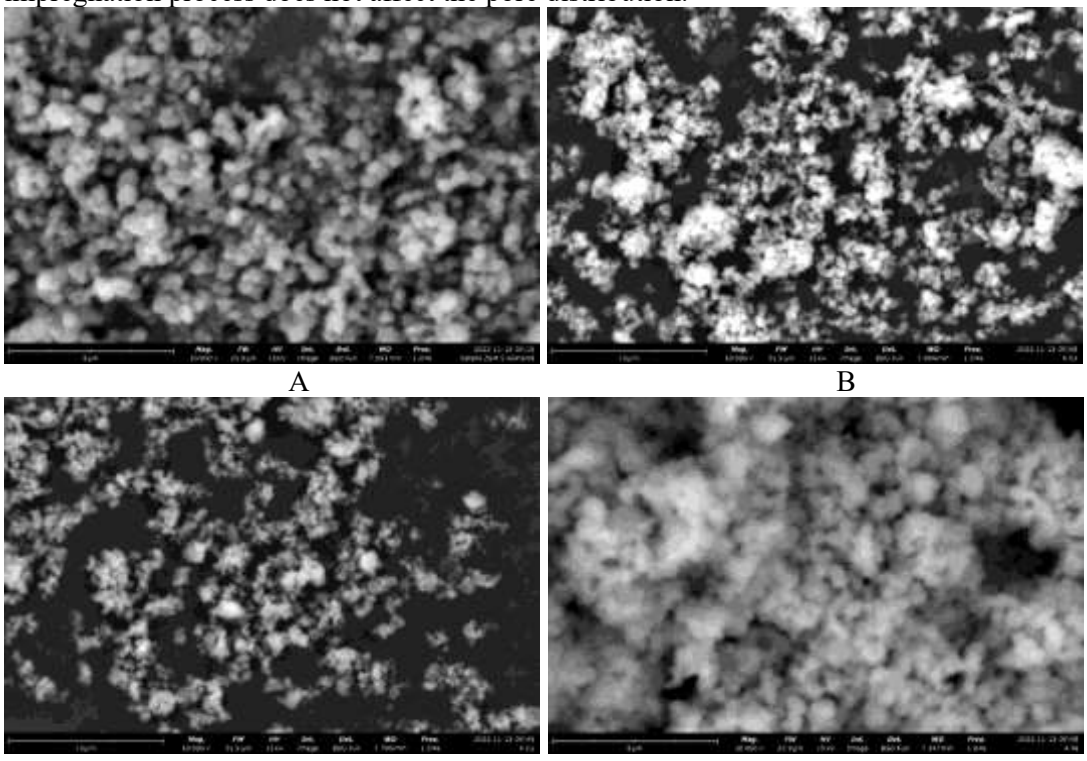
A B

C D

Figure 3. A. Commercial BJH ZSM-5 curve, B. BJH ZSM-5 curve impregnated with Fe C. ZSM5 curve impregnated with Co and D. BJH ZSM-5 curve impregnated with Cu metal

The pore distribution diagram presented in Figure 3 reveals that the ZSM-5 catalyst that is not impregnated and the ZSM-5 catalyst that is impregnated with Fe, Cu, and Co

metals have pores in the mesoporous region. The pore distribution curve reveals that the impregnation process does not affect the pore distribution.



A B C D

Figure 4. A. Commercial ZSM-5, B. ZSM-5 impregnated with Cobalt, C. ZSM-5 impregnated with Cu, D. ZSM-5 impregnated with Fe.

A characterization using SEM was carried out with the aim of identifying whether there was a change in the morphology of ZSM-5, which was impregnated with Fe, Cu, and Co metals. From Figure 4, it can be seen that there was no significant change from the impregnation process to the ZSM-5 crystals. It can be observed that the crystals before and after impregnation did not change and remained lumpy and round.

CONCLUSIONS

The effect of Fe, Cu, and Co metal impregnation resulted in a smaller surface area, total pore volume, and pore diameter of the catalyst, with the surface area of the catalyst impregnated with Fe, Cu, and Co metals being 291,853 m^2/g, 302,390 m^2/g, and 291,504 m^2/g, respectively. Meanwhile the pore diameters of the ZSM-5 catalyst impregnated with Fe, Cu, and Co were 3.3896 nm, 3.2688 nm, and 3.2790 nm, respectively, and the total pore volumes of the ZSM-5 catalyst impregnated with Fe, Cu, and Co, respectively, are 0.24732 cc/g, 0.24712 cc/g, and 0.23896 cc/g.

Fe metal can be applied as a candidate for a metal impregnated with ZSM-5 for partial oxidation reactions due to its specific surface area and total pore volume. Fe metal has a surface area of 291.853 m2/g and a total pore volume of 0.24732 cc/g. This is because the greater the total pore volume, the greater the activity in product formation. The activity of the catalyst is also affected by the surface area, but the small surface area will also have an impact on increasing the pore radius of the catalyst so that the catalyst pores can be passed through the reactants (feed) in the adsorption process.

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