Preparation and Characterization of Catalyst Zn/Al₂O₃ Catalyst using Dry and Wet Impregnation Method

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Abstract

Metal supported catalysts are used in many catalytic cracking processes. Experiments have shown that the increasing of metal loaded within the catalyst support may affected the increasing of cracking process conversion and selectivity. Therefore, a fundamental understanding of increasing the metal loaded is needed. In this study, Zinc on Alumina supported catalyst was prepared using the dry and wet impregnation methods to obtain the best amount of Zn metal content in Al₂O₃ as catalyst support with good characteristics for catalytic cracking. The prepared precursor solution in wet impregnation method was varied in concentration. While the dry impregnation method was varied in drying temperature. The Atomic Absorption Spectroscopy test results the metal content of Zn on the variation of precursor solution concentration on 0.5; 2.5; and 4.5 M are 1.06; 9.65; 15.91% respectively, and in the variation of the drying temperature at 25, 50, and 80 °C respectively are 18.32; 12.48; and 39.07% respectively. Characteristics analysis through X-Ray Diffraction, Brunauer-Emmet-Teller, and Scanning Electron Microscopy tests have also been carried out on samples with the highest metal content of 39.07% and samples with the lowest metal content of 1.06%.

Keywords: Catalyst, Impregnation, Metal-Support, Catalytic Cracking

Abstrak

Katalis yang didukung logam banyak digunakan dalam proses perengkahan katalitik. Eksperimen telah menunjukkan bahwa peningkatan logam yang dimuat dalam pendukung katalis dapat mempengaruhi peningkatan konversi dan selektivitas proses perengkahan. Oleh karena itu, diperlukan pemahaman mendasar tentang peningkatan beban logam. Pada penelitian ini, katalis pendukung Zn pada Alumina dibuat menggunakan metode impregnasi kering dan basah untuk mendapatkan jumlah kandungan logam Zn terbaik dalam Al₂O₃ sebagai pendukung katalis dengan karakteristik yang baik untuk perengkahan katalitik. Larutan prekursor yang disiapkan dengan metode impregnasi basah divariasikan konsentrasinya. Sedangkan metode impregnasi kering divariasi suhu pengeringannya. Uji Spektroskopi Serapan Atom menghasilkan kandungan logam Zn pada variasi konsentrasi larutan prekursor 0,5 M; 2,5 M; 4,5 M adalah 1,06%; 9,65%; 15,91% dan pada variasi suhu pengeringan 25, 50, dan 80 ºC masing-masing sebesar 18,32%; 12,48%; dan 39,07%. Analisis karakteristik melalui uji *X-Ray Diffraction*, *Brunauer-Emmet-Teller*, dan *Scanning Electron Microscopy* juga telah dilakukan terhadap sampel dengan kandungan logam tertinggi sebesar 39,07% dan sampel dengan kandungan logam terendah sebesar 1,06%.

Kata Kunci: Katalis, Impregnasi, Metal-Pengemban, Perengkahan Katalitik

INTRODUCTION

The use of a catalyst during a catalytic cracking reaction benefits the reaction by the enhance of reaction rate and the decline of reaction temperature. Several studies have shown the catalytic cracking process can be carried out in a lower temperature and be able to produce a lighter hydrocarbon fraction with a better quality than the product resulting from a non-catalytic cracking process [1,17].

The characteristics of catalyst will determine the reaction process and the resulting product. The amount of metal loaded, the structure of the catalyst, and the

pore size of the catalyst are several factors that influence the characteristics of the catalyst. Pinto proved that pore size greatly determines the number of hydrocarbon chains produced from cracking reactions, because the larger the pore size, the longer the hydrocarbon chain length of the product produced [2]. Serrano also shows that the catalytic cracking process of polystyrene will only be reached by a micropore catalyst [3]. In consequence, modification of heterogeneous catalyst into a metal-supported system is needed in catalytic cracking process to obtain good cracking catalyst characteristics such as large surface area, good porosity, heat stability, chemical stability, and mechanical strength [4].

Modification of heterogeneous catalyst into a metal-supported system be carried out by the impregnation method which is the most widely used preparation because the process is easy, fast, and economical [1]. However, the amount of metal (active site) on the catalyst support frequently cannot reach the desired level. According to Fang, the lower the metal in catalyst component, the lower the conversion [5]. Therefore, studies are needed to obtain high levels of metal on the metal supported system, as well as the characteristics of big surface area and good morphology to obtain good cracking results.

Liu has shown that metal in a catalyst support is mainly determined by the impregnation and drying process [6]. A fundamental understanding of impregnation and drying becomes very important to achieve an optimal metal profile. The drying effect on metal distribution and catalyst properties has been studied by Wu who examined the impact of various preparation procedures on the mechanical strength of solid catalysts and showed that drying had a significant effect on the mechanical properties of catalysts [7]. However, there is no explanation about how it affects the characteristics of the catalyst. The concentration of precursor solutions in catalyst preparation can affect the characteristics of the catalyst and the metal content on the catalyst [8].

In this research, Zinc metal on catalyst support of Al_2O_3 (alumina) was prepared by the wet impregnation method with variations in the concentration of the precursor solution and the dry impregnation method with drying temperature variations. Variations are made to determine the best amount of metal (Zn) on Al_2O_3 as catalyst support. In addition, it is expected that catalyst with the highest amount of metal (Zn) may have a good characteristic in surface area, pore size, and morphology which are suitable for the catalytic cracking process.

MATERIALS AND METHODS

Alumina catalyst support was loaded with 40% of Zinc with this following technique:

Catalyst Preparation by Wet Impregnation Method

A precursor solution was made by solving the metal precursor of $ZnCl_2$ in a solvent of distilled water resulted concentration of 0.5M (Sample Code A1); 2.5M (Sample Code A2); and 4.5 M (Sample code A3). Then, the solution was added to alumina support and stirred the mixture at 60 ℃ for an hour followed by drying in oven at 110 ℃ for 6 hours. Then, the sample was calcined at 300 ℃ for 4 hours.

Catalyst Preparation by Dry Impregnation Method

A precursor solution was made by solving the metal precursor of $ZnCl_2$ in a solvent of distilled water with the ratio as same as the solubility of $ZnCl_2$. Then, the solution was added to alumina support and dried in two steps: with initial drying temperature vary of 25 ℃ (Sample Code B1), 50 ℃ (Sample Code B2), 80 ℃ (Sample Code B3) for 8 hours and with final drying at 110 ℃ for 6 hours. Then, the sample was calcined at 300 ℃ for 4 hours.

General Procedure

Catalyst was prepared by using wet and dry impregnation method which generally consist of three steps; impregnation, drying, and calcination.

Detection Method

The Zn content in the catalyst were analyzed using Atomic Absorption Spectroscopy (AAS), the pore properties and the surface area of the catalyst were determined using Brunauer-Emmet-Teller (BET) method, and the surface morphology of the catalyst were analyzed using Scanning Electron Microscope (SEM).

RESULTS AND DISCUSSION

Concentration of Precursor Solution Analysis

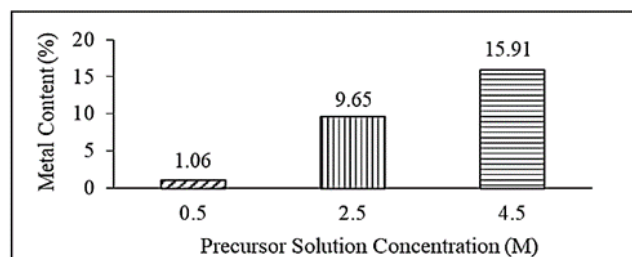The Zn content in samples from wet impregnation process is shown in **Figure** 1.

Figure 1. Result of AAS Analysis Data on the Sample Code A1 (0.5M), A2 (2.5M), and A3 (4.5M)

From Figure 1, it can be interpreted that the higher the concentration of the precursor solution is used, the greater the total metal content is absorbed. It shows that the quantity of $ZnCl_2$ in the precursor solution may affect the amount of Zn metal that can be carried on the Al_2O_3 supported catalyst. According to Nugrahaningtyas [8], the high concentration of precursor solutions can cause more substances to accumulate on the surface of catalyst support. Thus, displacement by diffusion increases together with the increasing of precursor solution concentration used. The A1 sample shows the lowest percentage of Zn metal at 1.06% with the use of the lowest concentration of precursor solution of 0.5 M. The low concentration of precursor solutions may affect the outer condition of catalyst support pore surface cannot provide a significantly high concentration gradient. Thus, the diffusion process that occurs is also low. As a result, there is only a slight Zn migrates into the supported catalyst pore. The A3 sample shows the highest percentage of Zn metal at 15.92% with the use of the highest concentration of precursor solution of 4.5 M. It can be caused by diffusion process phenomenon. A high precursor solution concentration may result a high concentration gradient between the outside and inside of the catalyst support surface. The high concentration gradient causes the transfer of a big amount of precursor solution. While the Zn content in A2 sample gives a linear relationship between the precursor solution concentrations on the metal content of Zn obtained. The metal content of Zn in sample A2 is greater than sample A1 and fewer than sample A3 with the metal content in sample A2 of 9.65%.

Through a theoretical calculation, 83 g of $ZnCl_2$ and 60 g of Al_2O_3 was used to reach the Zn content of 40% from the total weight of the catalyst resulted (40 g of Zn metal in 100 g of the Zn/Al_2O_3 catalyst obtained). Based on this, variations were carried out on the amount of distilled water used wherein the precursor solution concentration for 0.5 M; 2.5 M; and 4.5 M each obtained with 1195 mL, 216 mL, and 107 mL distilled water. The use of distilled water as much as 107 mL with a reactant mass of 143 g resulted in a very concentrated mixture looks alike slurry. Whereas in distilled water use as many as 1195 mL and 216 mL give a very dilute mixture.

In wet impregnation process, filtration was carried out after the impregnation step and a big amount of Zn were lost through the filter together with distilled water in the form of ion. This might be the reason of the low Zn content found from the wet impregnation process. Linearly, the increasing use of distilled water in this study shows a decrease in the amount of metal

contained in catalyst samples. The use of distilled water as much as 1195 mL in the A1 sample provides a greater chance for the Zn loss in filtering process. While A2 has a higher Zn content from A1 due to the less amount of distilled water used compared with A1 sample. Therefore, the possibility of Zn content is also lower. However, A3 sample (4.5 M) has the lowest probability of Zn loss due to its slurry form is not resulted lots of filtrate.

Drying Temperature Analysis

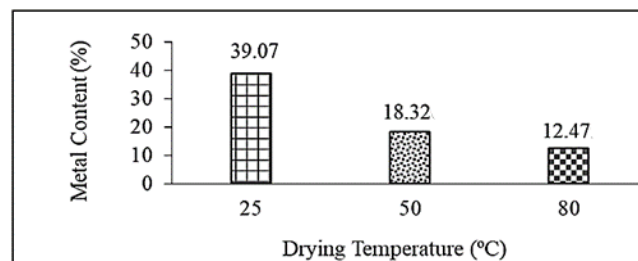The Zn content in samples from dry impregnation process is shown in Figure 2.

Figure 2. Result of AAS Analysis Data on the Sample Code B1 (25 ºC), B2 (50 ºC), and B3 (80 ºC)

Based on the data obtained in **Figure** 2, a high initial drying temperature used may lead to a low percentage of metal (Zn) resulted. According to Lee's review [9], during the drying period, the precursor solution around the catalyst support can migrate through the capillary flow and undergo the diffusion into the catalyst support pore. The dissolved metal is redistributed by the presence of desorption and absorption in the catalyst support pore. While the solvent evaporates and the solution becomes saturated, the solute is deposited on the catalyst support. According to this, drying process contributes to the distribution of metal in the catalyst support and provides different Zn content for the use of different drying temperature. B1 sample is resulted using the lowest initial drying temperature compared to the other sample, but it has the highest Zn level of 39.074%. This may be due to the low drying rate resulted from the low drying temperature; hence the Zn metal remains in the catalyst support after the undergoing of the drying process. In accordance with Kotter [10], who state that the low drying rate may lead to the formation of crystals which remain at the catalyst support pore or precisely in the core of the catalyst support particle. Warren [11], explained that the heat transfer by slow convection causes a slow solvent flow to evaporation surface. Thus, capillarity is maintained at each interface boundary and the solvent flow will not reach the evaporation surface. The solute is not transported

along with solvent and there is a diffusion process, therefore the concentration of solute is high.

The drying process in B3 sample was carried out using the highest initial drying temperature of 80ºC, but AAS analysis showed that it has the lowest Zn metal content compared to another sample, that is 12.479%. In according to Kotter [10], the high drying rate may lead to the escape of salt crystal from the pore. Thus, with a high initial drying temperature in the B3 sample, Zn metal in the catalyst support may come out from the alumina pore and cause the B3 sample has a low metal content. However, the adequate drying rate will only affect the salt crystals to be in the pore wall of the catalyst support. Therefore, in B2 sample with an initial drying temperature of 50ºC, Zn content is not higher than those in B1 sample nor lower than B3 sample.

The metal content that can be achieved using the dry impregnation method as shown in Figure 2 relatively is higher than using the wet impregnation method as shown in Figure 1. It indicates that dry impregnation method is better than wet impregnation method to obtain high metal content in metal supported catalyst. This is due to the ability to use a higher concentration in the dry impregnation process compared to the wet impregnation process.

The initial drying temperature used in all variables of the wet impregnation method is the same, namely at temperatures of 25ºC. The use of dry impregnation method, by the use of the same initial temperature, may result the highest amount of Zn metal from all samples in the study, namely in B1 sample. This is due to the precursor solution concentration in B1 sample is higher than the A1, A2, and A3 sample. Furthermore, dry impregnation can be carried out by only the use of 22 mL distilled water, while wet impregnation cannot be carried out with a less than 107 mL distilled water to get the reactants dissolved. Based on this, the precursor solution concentration used in the dry impregnation can be higher than the maximum of the precursor solution concentration in the wet impregnation method.

Catalyst Characterization

X-Ray Diffraction Analysis

The diffractogram pattern of alumina, zinc, samples with highest Zn metal content (sample B1; 39% and sample B2; 18%) and the lowest Zn metal content (Sample A1; 1%), and reference diffractogram are presented in **Figure** 3, 4, 5, and 6.

Qualitative analysis is carried out by comparing the diffractogram patterns of B1 (**Figure** 5) and B2 (**Figure** 6) with the reference alumina diffraction

pattern (**Figure** 3.a). It shows no significant difference pattern between them. This may indicate that there is no change in alumina structure of B1 and B2. In the range of (2θ) 15-25° on both samples, there is no peak of zinc referring to the reference diffractogram (Figure 3.b). According to Goula [12], it may be due to the dispersed of zinc into catalyst support. In consequence, zinc cannot be reflected by the x-ray through the catalyst support surface.
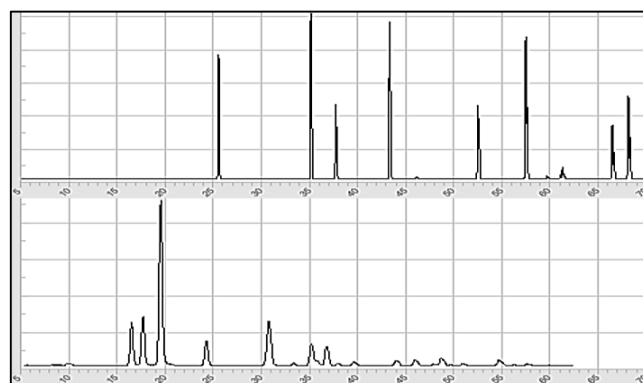

Figure 3. X-Ray Diffraction Diffractogram of (A) Al_2O_3 (B) Zn
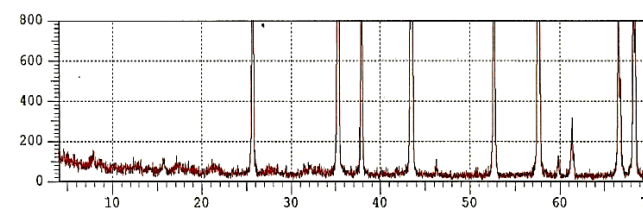

Figure 4. X-Ray Diffraction Diffractogram of A1 Sample (Wet Impregnation, 0,5 M)
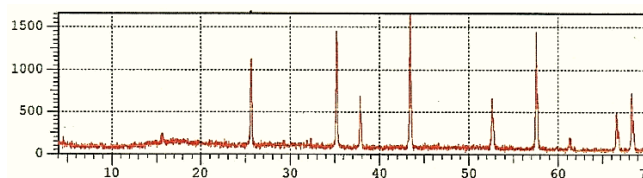

Figure 5. X-Ray Diffraction Diffractogram of B1 Sample (Dry Impregnation, 25ºC).
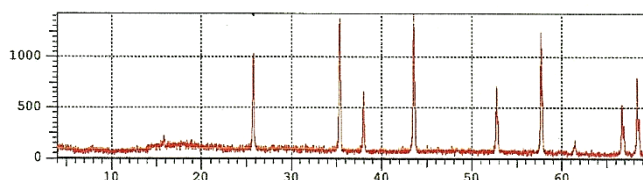

Figure 6. X-Ray Diffraction Diffractogram of B2 Sample (Dry Impregnation, 50ºC).

Therefore, the diffraction patterns of B1 and B2 do not form a high intensity peak. The diffraction pattern in the A1 sample (**Figure** 4) shows that there is one different peak from the reference alumina diffraction pattern in the range (2θ) 37-39°. This may indicate that zinc is not dispersed properly into the

catalyst support. According to Goula [12], the peak intensity is generated by metal that covers up the catalyst support surface. Thus, the metal may result a reflection of the beam when x-ray is passed through the catalyst surface and may result a high intensity peak in the diffractogram.

In B1 and B2 samples, by the range of 30-40°, it is observed that zinc may affect the increase of intensity. The diffraction pattern of B1 sample is higher than that of B2. In addition, in this range, the diffraction patterns of alumina and zinc have a similar peak pattern at some angles. Therefore, the zinc contained in sample B1 and B2 was observed as it did not contribute to the change of the pattern in catalyst.

Based on **Figures** 4, 5, and 6, it also can be seen that the material spectrum from the sample A1, B1, and B2 present a similar diffractogram pattern from the range (2θ) 5-70°. Even so, the peak intensity of the three samples is totally different. In **Figure** 5, sample B1 shows the highest spectrum intensity around 1650, while samples A1 and B2 as shown in **Figure** 4, and 6 are 800 and 1400. The difference in intensity of these three materials show differences in the amount of metal contained in the sample [13]. This reinforces the results of the AAS test on the three samples where B1 sample with the highest intensity does have the highest metal content compared to all samples. Conversely, in A1 sample with the lowest metal content of 1.06% it has a much lower intensity than the B1 sample intensity.

The data obtained from XRD characterization of the three samples shows that B1 has the best character compared to samples A1 and B2 if it is only observed by the diffraction pattern and intensity parameter. It is because B1 has a diffraction pattern which matches the reference Zn/Al_2O_3 diffraction pattern and has the highest intensity compared to the other sample. However, other parameters are still needed to find the best catalyst characteristic.

Surface Area and Pore Characteristic

BET analysis is carried out to analyze the catalyst surface area and pore. The analyzed catalyst is the sample with the highest Zn metal content (B1) and the catalyst sample with the lowest Zn metal content (A1). The result of the catalyst surface area analysis in sample B1 was 42.833 m^2/g, and the surface area of the A1 sample catalyst was 29.312 m^2/g. While alumina (Al_2O_3) used before the impregnation process has a surface area of 15 m^2/g. It shows that impregnation of metal into the catalyst support may increase the catalyst surface area. This result is in accordance with the study conducted by Shah [1], that the surface area of the Al_2O_3 catalyst will increase after the impregnation of metal.

Increasing the surface area of the Al_2O_3 catalyst in B1 and A1 corresponds to the embedded metal content of Zn, hence B1 which has the highest metal content of Zn (39.074%) may result a larger surface area of 42.833 m^2/g. While A1 which has the lowest metal content of Zn (1.06%) resulting in a smaller surface area of 29.312 m^2/g. The surface area enhancement of the catalyst also indicates that the quality of the catalyst is getting better, because the surface area contains active sites. In consequence, reactant compounds can be adsorbed in a bigger amount on the surface and the reactions can be fast due to the increasing of the reaction rate. In addition, one of the requirements for cracking catalyst is to have a surface area of between 10-1000 m^2/g [14]. Based on this, both sample B1 and A1 Zn/Al_2O_3 catalysts have fulfilled the requirement as a cracking catalyst.

Pore catalyst analysis was carried out by utilizing isothermal desorption adsorption which uses physical adsorption principle. Through this analysis, it can be obtained radius size or pore radius and the pore size distribution of the catalyst. The result of the pore analysis for B1 sample is shown by the isothermal desorption adsorption graph in **Figure** 7.
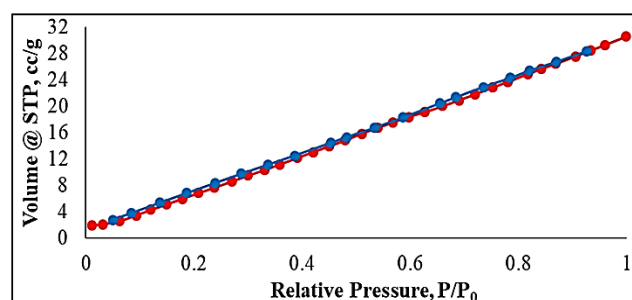
Figure 7. Adsorption of Isothermal Desorption with Nitrogen Gas

Figure 7 shows the analysis data in the form of the amount or volume of nitrogen gas (cc/g) to the relative pressure of nitrogen gas (P/P_0) adsorbed into the catalyst sample. In the initial condition that is before the catalyst sample is filled with nitrogen gas, the relative pressure is zero because there is no nitrogen gas contained in the pores of the catalyst sample. Then, along with the adsorption of nitrogen gas into the pores of the catalyst sample, the pressure will increase. So that the adsorption line in **Figure** 7 (red line) increases. This shows that the more volume of gas nitrogen adsorbed into the pores of the catalyst sample, the greater the relative pressure.

The increasing relative pressure indicates that all nitrogen gas has been adsorbed and filled the pores of the catalyst sample, therefore the nitrogen gas will slowly be absorbed and come out from the pores of the catalyst sample. Nitrogen gas desorption phenomenon

is presented by the desorption line (blue line) in Figure 7.

Based on **Figure** 7, it is observed that the catalyst belongs to type IV isothermal adsorption graph based on the classification of the International Union of Pure and Applied Chemistry (IUPAC). The characteristic of type IV isothermal adsorption graph is that there is a slight difference in the adsorption line and desorption line, wherein desorption will release the adsorbate with a greater quantity or volume than the amount of adsorbate adsorbed at adsorption at the same relative pressure. This phenomenon is better known as hysteresis. In **Figure** 7, hysteresis occurs when the relative pressure is 0.6 to 0.9. The hysteresis phenomenon indicates that when the physical adsorption process takes place on the catalyst, multi-layer adsorption or adsorption phenomenon occur [15].

The occurrence of multi-layer adsorption will also cause capillary condensation. Capillary condensation or pore condensation is a phenomenon of condensed gas being a liquid-like phase in a pore at a pressure lower than the saturation pressure of all its fluids [15]. The phenomenon of hysteresis, multi-layer physical adsorption, and capillary condensation all indicate that the catalyst has certain pore characters, namely mesoporous. Based on other classifications of IUPAC, a mesoporous structure catalyst has a pore size ranging from 2 to 50 nm or 20 to 500 Å (**Figure** 8).
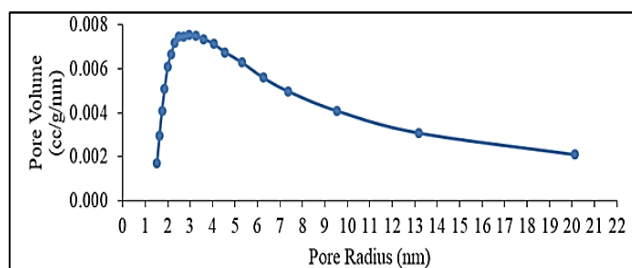

Figure 8. Catalyst Size Distribution

Figure 8 is a graph of the B1 catalyst sample pore size distribution which shows the amount of pore volume of the catalyst (cc/g/nm) at a specified pore radius (nm). Based on it, the overall pores contained in the B1 catalyst sample are in the size of the pore radius between 1-22 nm. Then, in the size range of 1-22 nm, most of the pore has a radius between 2-5 nm. This is appointed by the size of the pore radius between 2-5 nm, the amount of porous volume reaches 0.005 - 0.0075 cc/g/nm. While the size of the pore radius is more than 5 nm, the amount of porous volume shows a decrease which is smaller than 0.005 cc/g/nm.

Briefly, even though B1 sample catalyst has a pore radius that varies between 1-22 nm, most of the pores in the B1 catalyst sample have pore radius sizes

between 2-5 nm. This is also proven based on BET analysis which shows that the average value of the pores is 22.05 Å or 2.205 nm. The data has proved that the catalyst does have a mesopore structure.

The catalytic cracking reaction will produce different product quality based on the character of the catalyst used. In one type of plastic waste catalytic cracking, specifically polystyrene catalytic cracking, catalytic cracking will not occur if the catalyst used is micropore. This is because the structure of polystyrene has a phenyl group on the side of the main carbon chain so that it produces steric obstacles and causes the structure to be too thick or large to be able to enter into the micropore catalyst [3]. Thus, the catalyst cracking of polystyrene must be used which has a pore size greater than micropore.

Mesoporous characteristics obtained on the catalyst in this study may advantageous to be applied in polystyrene catalytic cracking. According to Shah [1], the desired main product of polystyrene catalytic cracking is styrene monomer (C_8H_8). However, in that study there were also several other products besides the main products of styrene monomers, including benzene, toluene, ethylbenzene, α-methylstyrene, naphthalene, 1.1'-(1,3-propanediyl) bis benzene, residues, and gases. The α-methylstyrene, naphthalene, and 1,1'-(1,3-propanediyl) bis benzene compounds are hydrocarbon chains that are longer than styrene monomer, with a molecular formula varies from C_9H_{10}, $C_{10}H_8$, and $C_{15}H_{16}$ respectively.

Whereas based on Pinto [2], the length of the catalytic cracking product is influenced by the pore size of the catalyst used. The greater the size of the pore, the longer the hydrocarbon chain of the product produced. Therefore, the application of a large pore size catalyst will increase the number of products with a longer chain and reduce the number of products with a lower chain. This was proven by Filip [16], who showed that the use of mesoporous catalysts may result a greater number of styrene monomer product than the number of styrene monomer product using a macropore catalyst. Furthermore, the use of a macropore catalyst may result in the number of a longer hydrocarbon chains product (α-methylstyrene and 1.1 '- (1,3-propanediyl) bis benzene). Based on this, the catalyst with mesoporous character obtained in this study when applied to polystyrene catalytic cracking may produce a lot of styrene monomer products.

Morphology Characteristic

SEM analysis for Zn/Al_2O_3 catalyst with the highest metal content of Zn (B1; 39%), the lowest metal content (A1; 1%), Al_2O_3 supported catalyst are presented in Figures 9, 10, 11, 12, and 13.
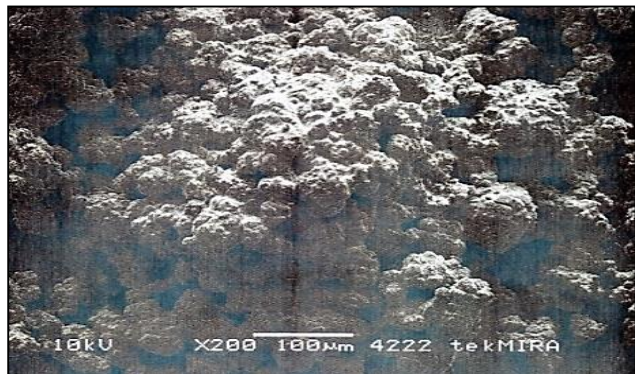

Figure 9. Morphology of Zn/Al_2O_3 Sample B1 Catalyst by SEM analysis of 200 times magnification.

Figure 10. Morphology of Zn/Al_2O_3 Sample B1 Catalyst by SEM analysis of 450 times magnification.
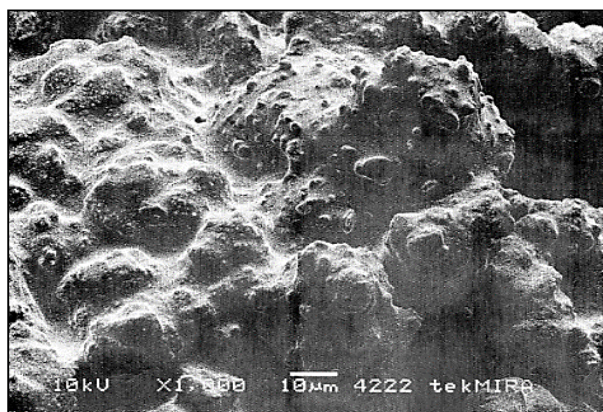

Figure 11. Morphology of Zn/Al_2O_3 Sample B1 Catalyst by SEM analysis of 1000 times.

Based on the characteristics of the surface area and porosity, the Zn/Al_2O_3 catalyst with the highest metal content (Sample B1, 39%) has a pore type of mesoporous structure. The structure of the mesopore means that the structure of the catalyst has a regular pore network and also has a high surface area. This kind of pore size is not too small or narrow (micropore type) nor too large (macropore type) [5]. This is proven by the result of SEM analysis as shown in Figure 9 and Figure 10.
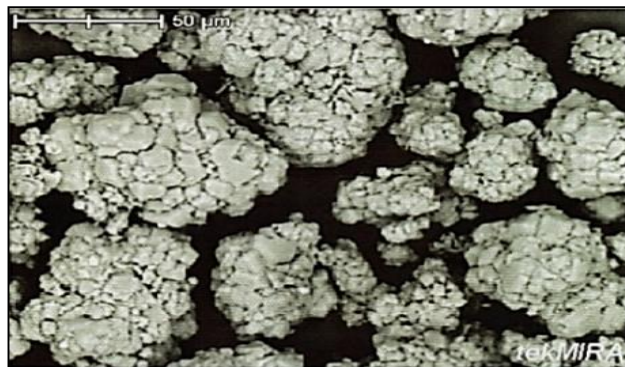

Figure 12. Morphology of Al_2O_3 Catalyst by SEM analysis of 1000 times magnification
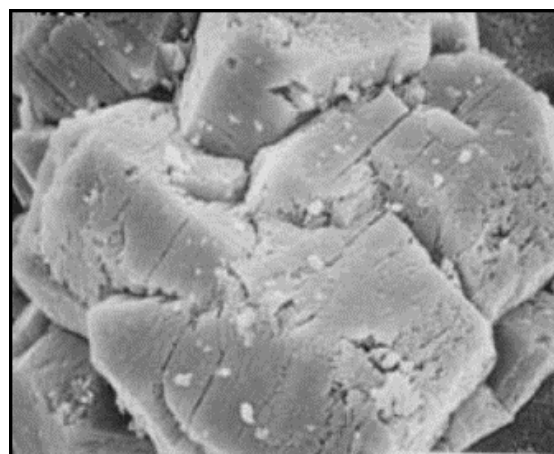

Figure 13. Morphology of Zn/Al_2O_3 Catalyst A1 Samples by SEM analysis of 450 times magnification

The morphology of the catalyst in **Figure** 9 looks like a cloud or interconnected agglomerates, consisting of almost uniform spheres with an adjacent pore. This is a characteristic of a catalyst that has a mesoporous structure and is caused by the occurrence of a hysteresis phenomenon. Based on Lowell [15], the phenomenon of hysteresis occurs when its relative pressure is more than 0.5, characterizes that the catalyst has a structure of agglomerates consisting of a uniform spherical shape. This is in accordance with the results of the BET analysis in **Figure** 7, where the phenomenon of hysteresis occurs when its relative pressure is close to 0.6.

Figure 12 shows the morphology of the Al_2O_3 supported catalyst before Zn was impregnated, with a magnification of 1000 times. By comparing **Figure** 11

with **Figure** 12, it is observed that there are significant morphological changes. In **Figure** 11, Zn covers up the Al_2O_3 supported catalyst surface. It looks slightly rough with a slightly protruding surface. This shows that Zn has been successfully impregnated in the catalyst support and Zn is distributed evenly, according to the characteristics obtained from XRD analysis.

Figure 13 shows the morphology of the Zn/Al_2O_3 (Sample Code A1) which has the lowest metal content (1.06%). By comparing Figure 10 with Figure 13, there are significant differences in structure or morphology. In Figure 10 it can be seen that the Zn has covered almost all part of the Al_2O_3 catalyst support surface due to the interconnected agglomerates. Whereas in Figure 13, Zn does not look covered by the Al_2O_3 catalyst support surface due to the separate agglomerates. It shows that a low content of Zn may affect the catalyst surface characteristic.

CONCLUSION

1. A higher drying temperature in the dry impregnation process lead to a lower metal content in the catalyst.
2. A higher concentration of the precursor solution in the wet impregnation process results in a higher metal content in the catalyst.
3. XRD characterization of the highest metal content (Zn) sample showed that alumina and zinc structure are observed, and a high diffraction intensity is obtained, while the XRD characterization of the lowest metal content sample showed that there were one different peak due to the reference of diffraction pattern and a low diffraction intensity is obtained.
4. BET analysis showed an increase in surface area subsequent to the impregnation wherein catalyst with a high metal content has a high surface area and mesoporous structure.
5. SEM analysis on sample with the highest metal content showed a good distribution of Zn metal and sphere-like shape with mesoporous structure observed on its structural morphology, while SEM analysis on sample with the lowest metal content did not show significant signs of Zn distribution.
6. The results of AAS, XRD, BET, and SEM showed that the B1 code sample of Zn/Al_2O_3 catalyst preparation met the characteristic targets needed for cracking.

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