CHROM IMPREGNATED ON ZEOLITE AS CATALYST FOR HYDROCRACKING OF LDPE-PLASTIC WASTE INTO LIQUIDS FUELS

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Abstract - Hydrocracking of LDPE-plastic waste into liquid fuels using Cr impregnated on Zeolite as catalyst has been performed. This research aimed to examine the effectiveness of Cr-Zeolite catalyst, to determine the yield of gasoline fractions, and to determine the weight of catalyst on the hydrocracking of plastic waste.This research began with pyrolysis process of LDPE using pyrolysis reactor. Cr-Zeolite catalyst for hydrocracking was prepared from natural zeolites activated by H_2SO_4 1 M. Chromium metal impregnation was performed by wet zeolite impregnation into $Cr(NO_3)_3$solution with an autoclave apparatus. Further, the resulting catalyst was calcined and reduced with hydrogen gas. The catalyst was characterized by Fourier Transform Infrared (FTIR), X-rayDiffraction (XRD), Surface Area Analyser (SAA), Atomic AbsorptionSpectrophotometer (AAS), andTransmission Electron Microscope (TEM), as well as acidity testing. The thermal cracking time varied for 15 and 30 minutes with a 20 mL/minute gas flow rate of H_2. The catalyst activity and selectivity test for hydrocracking of the plastic oils was carried out at optimum time with a temperature 300 ^oC using microreactor. Variation of catalyst:feed ratio used was 0.25; 0.5; and 1.0 (% w/w). The obtained liquid product was analyzed by GasChromatography-Mass Spectrometer (GC-MS).The results showed that Cr-Zeolite catalyst was effective in increasing the liquid product on pyrolysis oil hydrocracking. The largest liquid product and gasoline fraction were produced from the use of catalyst:feed ratio 1% (% w/w). On that ratio 52.2 % liquid product and 93.22% gasoline fraction were produced.

Keywords - Autoclave, Cr-Zeolite, hydrocracking, LDPE, pyrolysis

I.INTRODUCTION

The use of plastic is often found starting from the scale of household, industry, and offices. Almost in every place can be found items made of plastic such as fans, chargers, motor bodies, and so on. Based on these facts, as the increasing use of plastic will increase the waste of plastic, accordingly proper plastic waste management and processing are needed. Unprocessed plastic will have negative impact on the environment because it cannot be rapidly decomposed[1,2]. There are many ways that plastic can be used further so as not to cause negative impacts that frightencommunity, one of them is to recycle plastic waste. Recycling will turn plastic waste into new plastic materials. However, this recycling process will only convert plastic waste into a new form instead of overcoming the amount of plastic waste because when the plastic recycling product has lost its function it will return to plastic waste [3]. Only about 4% can be recycled, the rest are mounted in waste shelters [4]. The plastic-waste treatment that is currently widely studied and developed is converting plastic waste into fuel oil so as to reduce pollution to the environment [2]. Judging from the nature of the plastics composed of petroleum hydrocarbon components, the plastic waste is very potential to be converted into fuel oil. The technique that's used to restore the plastic material is by breaking the carbon chain or polymer to become hydrocarbon, one of them with pyrolysis technique [5]. Endang, et al [3]have converted PP (Polypropylene) and LDPE plastics by pyrolysis technique with natural zeolite catalyst that produces oil that has density valuesclose to kerosene's and diesel's. However, in this study, researcher focused only on the density test alone and did not measure other parameters such as flash point so that oil was equivalent to liquid fuel such as gasoline and so on.

Wardhana and Saptoadhi [6] have converted polyethylene plastic waste into fuel by pyrolysis method without catalyst resulting in 61% liquid fuel, 27% gasoline, and 12% of mass-based solid fuel. However, the results have not been tested with the GC-MS instrument so it cannot be seen how big the C5-C12 fraction thatequivalent to gasoline is.

It needs a study of plastic pyrolysis followed by hydrocracking with catalyst process to make better quality product. The selection of catalysts for hydrocracking of plastic pyrolysis oils is based on research by Sundaryano and Budiyanto [7] who have synthesized two Cr-H-Zeolite and Ni-H-Zeolite catalysts. Both catalysts were used to crush the palm oil liquid waste which produced yield 42% and 37.5%for each Cr-H-Zeolite and Ni-H-Zeolite catalysts. The Cr-H-Zeolite catalyst had the highest percentage value, but only about 7.34% ofliquid fuels fractionequivalent to gasoline was formed. With the above ideas, the author decided to do research on pyrolysis of plastic waste that produces oils and then proceed with hydrocracking process with zeolite catalyst carried by chromium metal. The pyrolysis of

oils prior to hydrocracking will be characterized by the standards prevailing in Indonesia or in the world as it approaches the nature of gasoline or not. The hydrocracking process makes it possible to break the double bond that's still inside the plastic pyrolysis oil to form a single branch without branches with quality equal to liquid fuel that is widely used today.

II.RESEARCH METHODS

Plastic pyrolysis process

The raw material of LDPE plastic was cut into small pieces and then put into a pyrolysis reactor. Pyrolysis was carried out to a temperature of up to 300-400 °C for 4 hours. The obtained oil from the pyrolysis process was analyzed by GC-MS and measured its density and immersion.

Synthesis and characterization of Cr-Zeolite catalyst

Zeolite that was obtained from C.V. Fruitanol Energy pounded until smooth and sifted 100 mesh. 100 grams of zeolites was fed into a glass beaker containing 200 mL aquades and stirred for 10 minutes. Then the mixture was allowed to stand for 24 hours.Furthermore the zeolite was separated from the mixture by the Buchner method. The result of zeolite solids plus 1 M H_2SO_4 and was stirred for 1 hour and then allowed to stand for 24 hours.The immersion was filtered and washed with aquadest up to pH 5 then tested with $BaCl_2$. Zeolite that was free of sulfuric ions ($SO4^{2-}$) was dried with oven at 100 °C for 24 hours. The dried zeolite was wet impregnated with $Cr(NO_3)_3$solution with percentage of metal versus the catalyst ie 1% (w/w) [8,9]. The process of loading was performed using an autoclave apparatus.The catalyst and chromium nitrate solution were introduced into the autoclave reactor and heated in an oven at 150 °C for 5 hours. Subsequently, the catalyst was separated from the solution by filtering and the catalyst was dried with oven for 24 hours at 100 °C. The impregnated dried metal catalyst was calcined using furnace at 500 °C for 3 hours. The calcined catalyst was reduced with H_2 gas at 400 °C for 2 hours using furnacetermoline. The synthesized Cr-Zeolite catalyst was then analyzed using FTIR, XRD, SAA, AAS, BET/Isothermal and TEM instrumentsm [10,11,12].

Catalyst acidity test

The number of acid sites wasdetermined by the gravimetric method by using ammonia gas. Empty porcelain crafts were put in the oven at 100 ^oC for 1 hour then weighed as W1. Then each zeolite (Natural Zeolite,H-Zeolite, and Cr-Zeolite) was given as much as 0.025 grams into a porcelain container and weighed as W2. Zeolite samples that were in porcelain containers were inserted into desiccators and then the desiccators were vacuumed. After being vacuumed, ammonia gas was flowed into the desiccators until saturated. The desiccators were then closed for 24 hours. After 24 hours, the desiccators were opened and the zeolite sampleswere taken and weighed as W3. The acidity of sample was determined by the equation:

$$Acidity = \frac{W_3 - W_2}{(W_2 - W_1)M} \times 1000 \text{ mmol/gram}$$

With M is MW of ammonia. The catalyst that had been acidified was gravimetrically tested qualitatively with FTIR instruments to see the uptake of Bronsted acid and Lewis acid sites [8,13].

Hydrocracked oil of plastic pyrolysis without catalyst

The process of hydrocracking the oil of plastic pyrolysis resultswas preceded by the treatment of hydrocracking without catalyst (thermal cracking) with a plastic oil mass of 30 grams. Hydrocracking was carried out at 300 °C for 15 minutes and 30 minutes with a 20 mL / minute hydrogen gas flow rate to obtain optimum time. The formed results were accommodated and analyzed by GC-MS instrument [14,6].

Catalyst activity and selectivity test

The hydrocracking process further utilized the Cr-Zeolite catalyst to determine the activity and selectivity of the catalyst. Hydrocracking was performed by varying the catalyst:feed ratio 0.5; 1.0; and 1.5 (% w/w). The hydrocracking process was carried out using hydrocracking reactor at temperature of 300 °C with hydrogen gas flow rate with obtained optimum time. The formed results were analyzed by GC-MS. The % (w/w) conversion of hydrocracked products (liquid, solid, and gas) can be calculated by the following equation:

$$\%\ (w/w)\ \text{liquid product} = \frac{\text{liquid product weight (g)}}{\text{feed weight (g)}} \times 100\%$$

$$\%\ (w/w)\ \text{coke} = \frac{\text{coke weight (g)}}{\text{feed weight (g)}} \times 100\%$$

$$\%\ (w/w)\ \text{residue} = \frac{\text{residue weight (g)}}{\text{feed weight (g)}} \times 100\%$$

$$\%\ (w/w)\ \text{gas} = \frac{\text{gas weight (g)}}{\text{feed weight (g)}} \times 100\%$$

III.RESEARCH RESULTS AND DISCUSSION

Catalyst Synthesis

The synthesis of Cr-Zeolite catalyst began with the preparation of natural zeolite. The used natural zeolite came from Yogyakarta. Activation of zeolite with H_2SO_4 (sulfuric acid) 1 M had the main purpose of activating the active side of zeolite so that zeolite had high acidity properties. Acidified zeolite was washed

with distilled water to remove SO_4^{2-}(sulfate ion) ions left in the surface and external zeolite [8,9].

The zeolite that was free from sulfate ions then impregnated with Cr (chromium) metal which amounted to 1% of the weight of the zeolite by the hydrothermal method. Walujodjati [15] disclosed that hydrothermal catalyst synthesis can increase the formation of a solid material with a high degree of crystallinity or may even reach a single crystal material structure. The autoclave app is a closed system making it extremely improbable for steam originating from internal heating closed system to exit [16]. The calcination process was carried out to produce an ordered structure of chromium impregnated zeolite with reaction equation [8]:

$$Cr(NO_3)_3\ (s) \text{ ---------} \blacktriangleright CrO\ (s) + N_2O_6\ (g) + NO_2\ (g)$$

$$N_2O_6\ (g) \text{ --------} \blacktriangleright 2NO_2\ (g)$$

The Cr-Zeolite catalyst after calcination was reduced by high temperature hydrogen gas to remove its metal oxide form by reaction [8,9]:

$$CrO\text{-}Zeolit_{(s)} + H_{2\,(g)} \xrightarrow[kalor]{} Cr\text{-}Zeolit_{(s)} + H_2O_{(g)}$$

Catalyst Characterization

Cr-Zeolite characterized by FTIR produced spectra in the figure 1. There are three important peaks: 3448, 1635, and 1033 cm^{-1}. The wave number 3448 cm^{-1} is the vibration -OH [17], the wave number 1635 cm^{-1} is the OH bending vibration of water, and the 1033 cm^{-1} wave number is the stretching vibration of Si-O- Si. In the H-Zeolite spectra there is a shift of wave to 1072 cm^{-1} [9,18]. This indicates that the success of acid activation is marked by the occurrence of dealumination in zeolite due to the shift of the waves to the larger ones. The% T drops in this wave number shows that on the zeolite surface to Cr-Zeolite, the pores are more open and the zeolite is cleaner than the existing impurities [19].

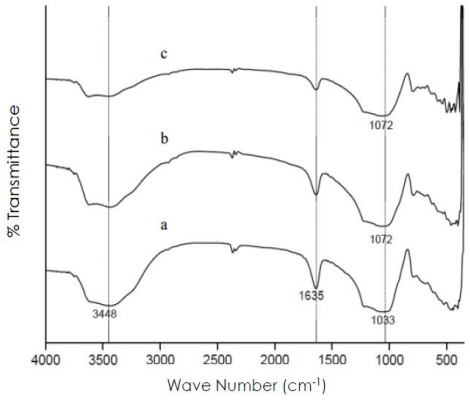


Figure 1. FTIR spectra on (a) natural zeolite, (b) H-Zeolite and (c) Cr-Zeolite.

Natural zeolite, H-Zeolite, and Cr-Zeolite are characterized by XRD by producing difractogram in the following figure:

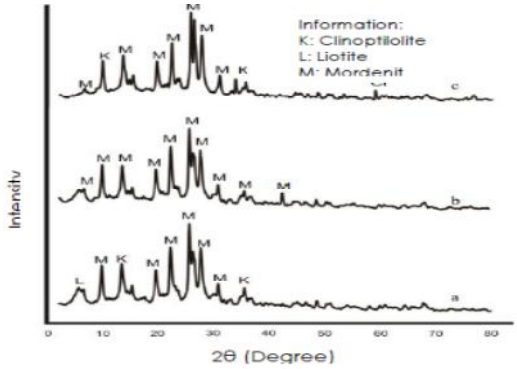


Figure 2. Difractogram pattern on (a) natural zeolite, (b) H-Zeolite and (c) Cr-Zeolite.

In the figure above it can be seen that the dominant mineral of zeolite structure is Mordenit. According to Morris et al. (1981) that at 2θ = 59.15 ° with the d = 1,5597 Å value is the typical diffraction pattern of chromium metal so that the chromium metal expansion in zeolite succeeded is shown by chromium metal diffraction pattern also appear at 2θ = 58,71 with d = 1,559 Å. Thus, it can be concluded that the chromium metal expansion into the zeolite pores had been successfully performed. The number of acid sites was observed by the ammonia gas adsorption method. The counted number of acid sites was the total number of Lewis acid sites and Brønsted acid [21,22,23]. The table 1 below show the number of acid sites of the catalyst.The rising number of acid sites from zeolites to H-Zeolites was regulated activation of acids that increased acid sites, while the increase of acid sites from H-Zeolite to Cr-Zeolite was due to impregnation of chromium metal which resulted in an increase in the number of acid sites as well [12].

Table 1. The number of acid sites

Sample	Number of acid sites (mmol/g)
Zeolite	0.23
H-Zeolite	1.571
Cr-Zeolite	2.353

Chromium metal can be known to exist by AAS method. The amount of chromium metal contained in the zeolite is shown in Table 2:

Table 2. Chromium metal content

Measurement	Measurement Results (ppm)
1	5159
2	5193
3	5125
Average	5159

The amount of impregnated chromium metal into the zeolite was theoretically 1% or 10000 ppm. In the above table, the amount of impregnated chromium

metal is only 5159 ppm or 0.52%. This might be due to impregnation of less than optimal metals.

Zeolite was characterized by TEM to look at its morphological structure. The result of characterization of catalyst with TEM instrument is presented in the following figure 3.

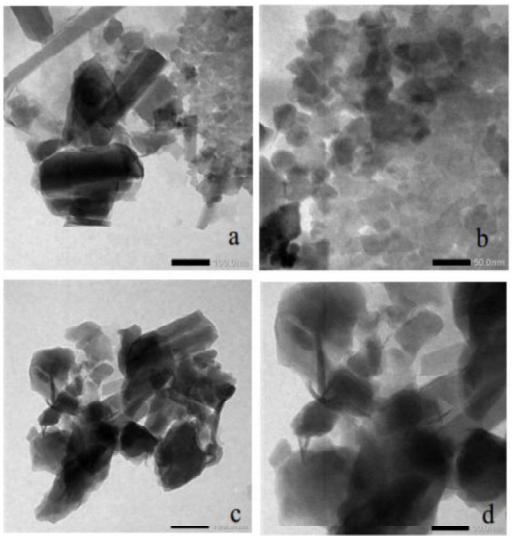


Figure 3. The morphology of the catalyst (a) 100 nm magnification zeolite, (b) 200 nm magnification zeolite, (c) Cr-Zeolite magnification 100 nm, and (d) Cr-Zeolite magnification 200 nm.

In the figure3 there are grayandthick black areas. Dark black areas indicate part that cannot be penetrated by light emitted by TEM. That part is something that has large mass in this case was the metal itself. Fig. 3 (a) and (b) show that the area of black is concentrated less when compared with Fig. 3 (c) and (d). This is because Fig. 3 (c) and (d) are zeolites that have been impregnated with chromium metals so that the black-colored portions are more numerous than Figures (a) and (b).

Table 3. BET/Isothermal analysis result

Sample	Specific Surface Area (m^2/g)	Pore Volume (cc/g)	Average pore diameter (nm)
H-Zeolite	23,93	0,10	8,361
Cr-Zeolite	23,72	0,13	10,77

Bet/isothermal characterization results show that except average pore diameter, there were no significance changes on specific surface area and pore volume of the H-Zeolite after treating by chromium.

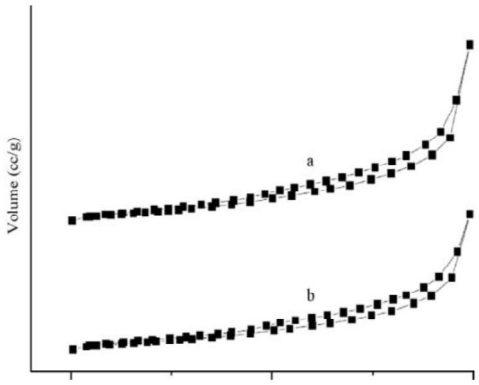


Figure 4. Adsorption-Desorption curve of (a) Cr-Zeolite and (b) H-Zeolite

Adsorption-desorption curve indicate that both materials, H-Zeolite and Cr-Zeolite have mesoporous structures

Converting Plastic Waste into Oil

The pyrolysis process was carried out with pyrolysis reactor at 350-400 ^oC for 5 hours. The successful obtained liquid product was 40.13% while 59.87% was a combination of solid and gaseous fraction and density of 0.791 g/mL. Plastic oil was analyzed by GC-MS instrument to see its fractions. Plastic oil chromatograms are presented in Figure 5. Light fractions (C1-C4), gasoline (C5-C12), and diesel (> C13) contained in a plastic oil of 0.54% respectively; 75.58%; and 23.88%. The resulting plastic oil was then used for thermal cracking without catalyst with time variations of 15 and 30 minutes at a temperature of 300 °C to determine the best time for the cracking process. The mild fractions (C1-C4), gasoline (C5-C12), and diesel (> C13) contained in the thermal cracking for 15 minutes respectively were 0.21%,; 76.48%; and 23.31%. While mild fractions (C1-C4), gasoline (C5-C12), and diesel (> C13) contained in thermal cracking for 30 minutes

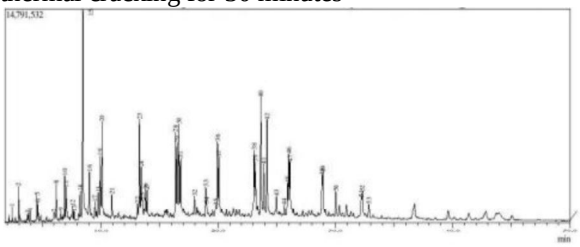


Figure 5. Plastic pyrolysis oil chromatogram

respectively were 1.9%; 81.69%; and 16.41%. The largest fraction of gasoline was produced on thermal cracking for 30 minutes. Thus, that time becomes the reference for the next hydrocracking process.

Catalytic Activity Test

The obtained catalytic activity from the LDPE plastic hydrocracking results was determined by conversion percent of each product in the form of liquid, coke, residue and gas products. The amount of obtained liquid product indicates how much catalyst ability to

convert feed (liquid fraction) to product (solid, liquid, and gas). The hydrocracking reaction was carried out with various catalyst ratios of 0.25; 0.5; and 1.0% (% w/w of catalyst: feed) at 300 °C for 30 minutes. The resulting products were liquid, coke, residue and gas products listed in Table 3.

Table 4. Percentage of obtained products from the use of each catalyst

Variation	Conversion			
	Liquid	Coke	Residue	Gas
Thermal Cracking	44.32	8.01	19.04	28.63
Cr-Zeolite 0.25%	44.6	0.07	20.96	34.37
Cr-Zeolite 0.5%	52.15	0.09	16.48	31.28
Cr-Zeolite 1%	53.2	0.4	13.32	33.2

From Table 3 it can be seen that the greater the% w/w catalyst:feed ratio will increase the liquid and coke products and decrease the residue. The more acid sites the more liquid products that are able to be converted than the fewer acid sites. The catalyst:feed ratio which produced more liquid product was Cr-Zeolite 1% which also had the highest number of acid sites. Munir, et al. [24] have converted a mixture of waste plastic into fuel with a variety of micro-mesoporous zeolite catalysts. The results were 11% plastic waste that was successfully converted into liquid product with thermal cracking technique and 22% with the aid of catalyst. This shows the percentage of liquid product that's successfully converted using a larger catalyst than normal thermal cracking. From the above research, it can be adjusted with the results of research that hydrocracked liquid products with catalysts are formed more than ordinary thermal cracking techniques. From these data it can be concluded that 1% Cr-Zeolite catalyst produces a large amount of product and can minimize the amount of residue left behind. To see the fraction contained in each oil of hydrocracking result is characterized using GCMS.

Catalytic Selectivity Test

The catalyst selectivity test is performed by hydrocracking process using a catalyst to see the quality of the oil produced. Each catalyst has its own selectivity to the product it binds to. The selectivity of the product can be determined by summing each of the light fractions (C1-C4), the gasoline fraction (C5-C12), and the diesel fraction (> C13) shown on the chromatogram. Each peak must be interpreted first to know what compound is represented on each peak. Peaks with SI values below 90 tend to be ignored because of the incompatibility of target compounds with very large references. The resulting fractions for each catalyst, thermal cracking, and pertamax are presented in Table 4.

Table 4. Fraction contained in hydrocracking oil and gasoline (pertamax)

Variation	Fraction (%)		
	Light Fraction (C1-C4)	Gasoline Fraction (C5-C12)	Diesel Fraction (>C13)
Thermal Cracking	1.9	81.69	16.41
Cr-Zeolite 0.25%	1.47	85.5	13.02
Cr-Zeolite 0.5%	0	87.4	12.6
Cr-Zeolite 1%	0	93.22	6.78
Pertamax	0	99.48	0.51

From the above table it can be seen that hydrocracking oil with 1% Cr-Zeolite catalyst produces a high selectivity to the gasoline fraction. Overall, good results are obtained from the selectivity of the gasoline fraction by each catalyst is above 85%. This value is quite satisfactory because the selectivity that's shown for each catalyst is very large. When compared with thermal cracking, each catalyst also produces a greater selectivity value than thermal cracking because in addition to the catalyst there will be increased selectivity to the gasoline fraction. % content. Wahyudi, et al. [25] say that the greater the catalyst ratio will produce the highest liquid product with high selectivity as well. The results of this study shows that Cr-Zeolite catalyst ratio is the largest, ie 1% has produced the highest liquid product (53.2%) and with high selectivity of the gasoline fraction (93.22%). When compared with Pertamax, the selectivity of hydrocracking oil with 1% Cr-Zeolite catalyst is very much different ie 99.69%.

CONCLUSION

Based on the results and discussion in this study it can be concluded:

The use of Cr-Zeolite catalysts in the hydrocracking of pyrolysis oils effectively improves the liquid product when compared to ordinary thermal cracking.

The catalyst:feedratio 0.25% increased liquid product by 0.28%,ratio of 0.5% increased liquid product by 7.83%, and1% ratio increased liquid product by 8.88%.

Cr-Zeolite catalysts increase the percent ofgasoline fraction when compared with thermal cracking. Without the use of the catalyst, the resulting gasoline fraction was 81.69%. In the catalyst:feedratio, 0.25% produced 85.5%; 0.5% produced 87.4%;1% produced 93.22% of gasoline.

The largest liquid product and gasoline fraction were produced from the use of catalyst:feed ratio of 1% (% w/w).

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