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Synthesis of Cr/Al₂O₃- Bentonite Nanocomposite as the Hydrocracking Catalyst of Castor Oil

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Synthesis and characterization of Cr/Al_2O_3 -bentonite nanocomposite as a hydrocracking catalyst of castor oil have been conducted. The catalyst was prepared according to the following method. At the first step, bentonite was activated using H_2SO_4 to obtain H-bentonite, after that the activated clay was pillarized by Al_2O_3 , Next, a salt solution of Cr (NO₃)₃·9H₂O was impregnated in the pillared bentonite sample, followed by calcination and reduction to obtain the final catalyst, i.e. Cr/Al_2O_3 bentonite. The catalysts including unpillared bentonite were characterized using infrared spectrometer (FT-IR), X-Ray diffractometer (XRD), X-ray Fluorescence

spectrometer (XRF), Transmission Electron Microscope (TEM) and gas sorption analyzer. GC-MS analysis was conducted to characterize the hydrocracking product. The research results showed that pillarization of bentonite caused an increasing of the basal spacing of bentonite in an amount of 1.01 nm. Although Chrom was unevenly dispersed on the bentonite and it probably blocked the bentonite framework resulted in the decrease of catalyst specific surface area, Cr/Al₂O₃-bentonite catalyst gave the best conversion of 64.03%. The GC-MS analysis data showed that the hydrocracking products contained various kinds of organic compounds such as acetone, acetic acid, methyl benzene, octane, heptanal, 2-octene, 1-undecanoic acid, 9-octadecenal and 10-undecenoic acid.

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Synthesis of Cr/Al₂O₃-Bentonite Nanocomposite as the Hydrocracking Catalyst of Castor Oil

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Keywords: Cr/Al₂O₃-bentonite, nanocomposite, hydrocracking, castor oil

Abstract. Synthesis and characterization of Cr/Al₂O₃-bentonite nanocomposite as a hydrocracking catalyst of castor oil have been conducted. The catalyst was prepared according to the following method. At the first step, bentonite was activated using H₂SO₄ to obtain H-bentonite, after that the activated clay was pillarized by Al₂O₃, Next, a salt solution of Cr(NO₃)₃·9H₂O was impregnated in the pillared bentonite sample, followed by calcination and reduction to obtain the final catalyst, i.e. Cr/Al₂O₃-bentonite. The catalysts including unpillared bentonite were characterized using infrared spectrometer (FT-IR), X-Ray diffractometer (XRD), X-ray Fluorescence spectrometer (XRF), Transmission Electron Microscope (TEM) and gas sorption analyzer. GC-MS analysis was conducted to characterize the hydrocracking product. The research results showed that pillarization of bentonite caused an increasing of the basal spacing of bentonite in an amount of 1.01 nm. Although Chrom was unevenly dispersed on the bentonite and it probably blocked the bentonite framework resulted in the decrease of catalyst specific surface area, Cr/Al₂O₃-bentonite catalyst gave the best conversion of 64.03%. The GC-MS analysis data showed that the hydrocracking products contained various kinds of organic compounds such as acetone, acetic acid, methyl benzene, octane, heptanal, 2-octene, 1-undecanoic acid, 9-octadecenal and 10-undecenoic acid.

1. Introduction

Petroleum is the most widely used energy source in the world, both macro and micro sectors require petroleum mainly for industrial, transport and agricultural machinery. Petroleum or fossil fuel is limited and unrenewable so it could deplete if consumed continuosly. According to population growth and fuel usage data, fuel depletion will occur significantly in 2050 [1].

Biogasoline is used as an alternative energy source to replace petroleum, one of which is castor oil. Castor oil is a non-edible oil from *Ricinus communis L*. plant that does not compete with food resource. Jatropha resistance in a long dry season and grow on marginal lands so it can be planted in a larger area with lower production costs [2]. Hydrocracking reaction of castor oil needs bifunctional catalyst (metal-bearers catalyst) for cracking and hydrogen activation. Bentonite is abundant but undeveloped so its economic value is low. Bentonite has disadvantages such as damaged layer structure or disappeared porosity due to heating at high temperature [3]. It needs modifications such as pillarization and active metal impregnation in bentonite interlayer. Chrom (Cr) is used as the active metal because it has unfilled d-orbital. The d-orbital is capable of providing space for the reactions taking place.

The usage of Cr as metal on a bearer catalyst had been done by Trisunaryanti and Jujarama. Trisunaryanti used natural zeolite as the bearer catalyst and Cr as the metal. The result showed that the conversion of gasoline fraction when using catalyst at the cracking process was much greater than the cracking without any catalyst [4]. Meanwhile Jujarama had the same result, it show that Cr on the bearer catalyst quite effective in the hydrocracking process to biogasoline. The catalyst

Cr-activated natural zeolites had higher surface area than the natural zeolite. The hydrocracking product was also more similiar to gasoline by the number of carbon atoms between C_6-C_{12} [5].

Bentonite is a clay where mostly consisting of montmorillonite. Bentonite was greatly affected from acid activation, ion exchange, heating, hydrothermal treatments and some other physicochemcial processes [6]. Single cell unit of montmorillonite concists of two sheets of tetrahedral silica where the middle layer is the octahedral sheet. Montmorillonite lattice has a negative charge due to isomorphic subtitution of ions in the structure. This negative charge is balanced by cations on the surface of the sheet. Cations that most commonly found in nature are sodium and calcium [7].

Wijaya, et al. had conducted hydrocracking of used cooking oil by nickel-bentonite catalyst but the acidity value was lower and the surface area was smaller. This led to the hydrocracking conversion value that approximately only 8.33-23.33% [8].

Based on the descriptions, the research of castor oil hydrocracking using Cr/Al_2O_3 -bentonite catalyst had been conducted. Bentonite was activated using sulfuric acid and was pillared using Keggin ion of Al_2O_3 . The purpose of this research was to converse castor oil to biogasoline and learnt the influence of Al_2O_3 pillaring and Cr impregnating in bentonite catalyst.

2. Experimental

Materials used in the research were castor oil, H_2SO_4 1,2 M, HF 1%, natural bentonite, chromium nitrate nonahydrate $Cr(NO_3)_3 \cdot 9H_2O$ as Cr source, $AlCl_3 \cdot 6H_2O$ as Keggin ion source, hydrogen gas, nitrogen gas and aquadest. This research used several laboratorium tools such as laboratory glassware, fixed-bed cracking reactor, a set of reflux tool, magnetic stirer, furnace, oven, centrifudges, digital weight, 250 mesh filter, hot-plate stirer and porcelain mortar. The catalyst were analyzed by using X-ray diffractometer (XRD-6000 Shimadzu), Infrared spectrometer (FTIR-8201 PC Shimadzu), gas chromatography-mass spectrometer (GC-MS QP-2010S Shimadzu), Transmission Electron Microscope (TEM) (Jeol JEM-1400), gas sorption analyzer (Quantachrome NovaWin version 11.0) (BET method) and X-ray fluorescence spectrometer (XRF) (PANalytical Minipal 4).

Bentonite preparation: bentonite was washed using aquadest by dispersing it for 24 h in 1:2 ratio (w/v). The upper layer of bentonite was separated then dried at 90-110 °C. The dry bentonite was grinded and filtered using 250 mesh filter. Bentonite was washed again using HF 1% to remove silica impurities. It was dispersed in hydrofluoric acid solution by 1:2 (w/v) ratio for 15 min. The suspension was neutralized using aquadest until pH 6. It was dried at 90-110 °C, grinded and filtered using 250 mesh. HF-bentonite was activated using H₂SO₄ 1,2 M with 1:4 (w/v) ratio and was stirred for 1 h. It was labeled as activated natural bentonite (H-bentonite).

Pillarization: the Keggin ion (Al_{13}) solution was prepared by slow titration of $ACl_3 \cdot 6H_2O$ solution with NaOH solution under vigorously stirring until the ratio of OH⁻/Al = 2,2 was reached then was refluxed for 3 h at 80-90 °C. Intercalation process was conducted by dropping the warm solution into a bentonite dispersion, stirred for 24 h and calcined at 500 °C for 2 h. The obtained aluminum pillared bentonite was denoted as Al_2O_3 -bentonite.

Chrome impregnation: the impregnation was conducted by submerging Al_2O_3 -bentonite in $Cr(NO_3)_3 \cdot 9H_2O$ solution (Cr 1% (w/w)) then refluxed for 5 h at 80-90 °C. The suspension was heated using oven then calcined by N_2 gas flow on 500 °C. Gas flow rate on calcination process was 20 mL/min. The formed catalyst was reducted by using H_2 gas at 15 mL/min flow rate and 400 °C for 2 h. The catalyst was labeled as Cr/Al_2O_3 -bentonite.

Acidity test of the catalyst: The acidity of catalyst was analyzed using gravimetric method of ammonia gas absorption. Dry crucible porcelain was weighed. The samples was inserted and weighed again. Crucible porcelain contained samples was put in oven for 2 h then put it in the desiccator and vacuumed it. Ammonia gas was flowed into the desiccator then stopped after there was no longer bubbles. The dessicator was closed and let it for 24 h. The sample acidity was calculated by the following formula:

weight adsorbed = $\frac{(W_2 - W_1)}{(W_1)MW}$ mol/g

Castor oil catalytic cracking: castor oil was set in fixed-bed reactor's feed column with catalyst:castor oil ratio was 1:5 (w/v) at 500 °C for 1 h and 20 mL/min H₂ gas flow. The feed column being put in a furnace which associated with an ice cooler and flask pear shape as product reservoir. Liquid product (biogasoline) was analyzed by using GC-MS. According Twaiq *et al.* the hydrocracking conversion value of biogasoline product could be determine using following formula [9-11]:

 $Conversion = \frac{product \ volume}{feed \ volume} x100\%$

3. Result and Discussion

Minerals composition in bentonite: compounds composition in bentonite instrumentally determined using X-ray fluorescence spectrometer (XRF). Highest mineral of metal oxides were SiO₂, Fe₂O₃, Al₂O₃ dan CaO (Table 1). The increasing of SiO₂ composition indicated that Si/Al ratio was getting higher. The more Si atom in bentonite, the more Si/Al ratio would increase. Alumina increased due to intercalation of polioksocation aluminum $[Al_{13}O_4(OH)_{24}(H_2O)_{12}]^{7+}$ on the interlayer silicate bentonite that made the basal spacing value (d₀₀₁) increased to 1.97 nm and Δd_{001} to 1,01 nm (Table 2). Fe₂O₃ and CaO decreased due to a decline of Fe and Ca cations ammount in the interlayer space since they exchanged with Al₂O₃ and Cr. In Cr/Al₂O₃-bentonite catalyst, Cr₂O₃ increased to 0.56%. This showed that Cr had been succesfully impreganted in the interlayer bentonite.

	Concentration			
Oxides	H-bentonite (%)	Al_2O_3 -bentonite (%)	Cr-Al ₂ O ₃ -bentonite (%)	
SiO ₂	56.90	61.30	63.30	
Fe ₂ O ₃	17.50	16.80	13.90	
Al ₂ O ₃	11.00	17.00	18.00	
CaO	6.88	1.53	1.36	
Cr ₂ O ₃	0.05	0.05	0.56	

Table 1. Bentonite minerals composition

Crystalinity test for catalyst: Crystalinity test for H-bentonite, Al₂O₃-bentonite and Cr/Al₂O₃-bentonite used X-ray Diffraction spectrometer (XRD). The analysis result could be seen on Fig. 1. Na-bentonite diffractogram showed the montmorillonite peak at $2\theta = 5.92^{\circ}$ but shifted to $2\theta = 5.5^{\circ}$ on H-bentonite with higher intensity. This was caused by acidification treatment by sulfuric acid where some quartz mineral dissolved in acid liquid so the montmorillonite levels increased. The pillared bentonite diffractogram showed that there was a shift to $2\theta = 4.44^{\circ}$ which made the basal spacing increased from Na-bentonite 1.49 nm ($\Delta d_{001} = 1.49-0.96 = 0.53$ nm) to 1.97 nm ($\Delta d_{001} = 1.97-0.96=1.01$ nm). [Al₁₃O₄(OH)₂₄(H₂O)₁₂]⁷⁺ which has larger size than 1.01 nm (from range ±0.9-1.1) was intercalated in bentonite's layer resulting the basal spacing value increased. Silica layers on bentonite could open more broadly when current cations replaced by intercalating bigger pillaring species. The interlayer cations may exchange all inorganic and organic cations, and also hydrated hydroxy-polynuclearmetal cations. In Fig. 1 (d) and Fig. 1 (e) were seen peak shift at $2\theta = 1^{\circ}$ to 5°. Chrom was oxidized into Cr₂O₃ in the interlayer of bentonite, it proved in Table 2 that Δd_{001} value increased to 1.94 nm with Cr₂O₃ value increased to 0.56% (Tabel 1).

Table 2. Basal spacing value of the catalysts.					
Catalyst	Pore diameter (nm)	$\Delta d_{001} (nm)$			
Na-bentonite	1.49	0,53			
H-bentonite	1.62	0.66			
Al ₂ O ₃ -bentonite	1.97	1.01			
Cr-Al ₂ O ₃ /bentonite	2.9	1.94			



Fig. 1. Diffractogram of catalysts (a) Na-bentonite, (b) H-bentonite, (c) Al₂O₃-bentonite, (d) calcined bentonite and (e) Cr- Al₂O₃/bentonite.

Functional group characterization: Infrared Spectrometer (FTIR) can be used to identify functional group of molecules in a particular material including bentonite. Bentonite can be observed in wavenumber range 400-4000 cm⁻¹. Infrared spectra results of Na-bentonite, Al_2O_3 -bentonite and Cr/Al_2O_3 -bentonite were presented in Fig. 2. The wavenumber of 470 cm⁻¹ was bending vibration of Si-O-Si in the tetrahedral layer. Absorbtion of 794 cm⁻¹ was symmetric stretching vibration while stretching vibration of Si-O-Si was 1041 cm⁻¹. The identification of H-O-H bending vibration of water molecules was 1635 cm⁻¹. Absorption peak at wavenumber region 3425 cm⁻¹ indicated the vibration of water molecules in the solid surface while at 3626 cm⁻¹ was stretching vibration of silanol (Si-O-H) and aluminol (Al-O-H).



Fig. 2. Infrared spectra of bentonite and its modifications (a) H bentonite, (b) Al₂O₃ bentonite and (c) Cr/Al₂O₃-bentonite.

The dealumination process was shown in the wavenumber of 918 and 1041 cm⁻¹. Transmittance peak of Al-O-Al strain vibration at 918 cm⁻¹ was more diminished toward Cr/Al₂O₃bentonite. The cations (Al) in the tetrahedral layer decreased, making the Si/Al ratio increased. This was reinforced by the appearance of asymmetric stretching vibration of Si-O-Si at 1041 cm⁻¹ who shifted toward larger wavenumber by 1049 cm⁻¹. From energy equation or $E = h \frac{c}{\lambda}$ meant that the larger the wave number, the larger the energy is. This clearly indicates that the more the non-polar character, the energy to vibrate the molecules is getting bigger. This meant Si/Al ratio (non-polar) was getting bigger since it shifted to the larger wavenumber.

Morphology analyzes of bentonite: Transmission Electron Microsope (TEM) analysis was done because it could show morphology changing between bentonite layers. In Figure 3 (a) and (b) which is Al_2O_3 pillared bentonite, dark and thick lines showed the alumino silicate layers while the brighter colour indicated the larger pore. The distance between the dark lines showed qualitatively the intercalation. Meanwhile in Figure 3 (c), the dark-coloured granules were Cr metals. This showed that Cr metals were succesfully dispersed into the bentonite's cavity. At a magnification of 20 nm (d), the darker and brighter colour indicated that Cr metals were not dispersed unevenly on the surface of bentonite but both structures Al_2O_3 -bentonite and Cr/Al_2O_3 -bentonite did not damage, the layers was still remaining.



Fig. 3. Morphology of the catalyst (a) Al₂O₃-bentonite resolution of 100 nm, (b) Al₂O₃-bentonite resolution of 20 nm, (c) Cr/Al₂O₃-bentonite resolution of 100 nm and (d) Cr/Al₂O₃-bentonite resolution of 20 nm.

Surface area, average pore radius and total pore volume: The results of gas sorption analyzer using BET method were shown on Table 3. Bentonite which treated with H_2SO_4 1.2 M labeled as H-bentonite had the highest surface area. Intercalation during pillarization led to surface area decreasing which also shown by the reduced value of the basal spacing. This was occured because blocking of bigger molecules such as Cr and Keggin ions (Al₂O₃) in the bentonite pore. The another reason is because Cr did not dispersed equally so it was clotting on the surface of bentonite (sintering).



Fig. 4. Adsorption-desorption isotherms patterns of bentonite.

Catalyst	Surface area (m^2/g)	Average pore radius (Å)	Total pore volume (cc/g)
H-bentonite	109.18	71.1	0.19
Al ₂ O ₃ -bentonite	108.76	57.8	0.16
Cr-Al ₂ O ₃ /bentonite	84.05	67.7	0.14

Table 3. Pore characters of bentonite.

The adsorption-desorption isotherms patterns towards N_2 gas can be used to determine the type of solid isoterm. In Fig 4, All the tree showed type IV isotherm, indicated that the formation was mesoporous material. The knee on the graph sowed the pores of the material which was disorganized porous materials. In Cr-Al₂O₃ showed the sharper graph, indicated that the material was more porous so it had larger cation exchange space.

Acidity test of the catalyst: Acidity is determined by gravimetric methods of ammonia (NH₃) absorption. Ammonia which was alkaline absorbed by bentonite which had acid sites in its framework. Ammonia size is relatively smaller than bentonite's cavity so ammonia was able to insert the acid site in the cavity. Ammonia did not only interact with the acid sites on the surface but also with the acid site in the cavity. Table 4 shows that the surface acidity increased from H-bentonite to Cr/Al₂O₃-bentonite. This was reinforced in Fig. 2 where each of the spectras indicated the presence of N-H vibration of NH₃-Al at 1450 cm⁻¹ and N-H vibration of NH₄⁺ at 1635 cm⁻¹. Brønsted and Lewis acid sites on their surfaces are proton donors and electron pair acceptor respectively [12]. N-H vibration of NH₄⁺ was Brønsted acid sites because there was exchange of electron pairs as N-H vibration of NH₄⁺ was Brønsted acid sites because there was proton donor. From Figure 5, Lewis acid sites' transmittance was higher than the Brønsted's. It showed that Lewis acid sites more dominated the bentonite's surface. If transmittance is getting close to zero, it meant there were more compounds so that they could absorb the signal and the transmitted signal getting less. This was also applied to pillared and impregnated bentonite because both had dominated Lewis acid sites.



Fig. 5. Infrared spectra after acidity test (a) H-bentonite, (b) Al₂O₃ bentonite and (c) Cr/Al₂O₃-bentonite.

Catalytic activity test: castor oil conversion into biogasoline was done by using hydrocracking process with H-bentonite, Al_2O_3 -bentonite and Cr/Al_2O_3 -bentonite. Castor oil hydrocracking with Cr/Al_2O_3 -bentonite produced the greatest conversion (Table 5) since this catalyst had the highest catalytic activity. The porous structure of bentonite caused larger surface area so the catalytic activity increased.

Table 5. Liquid product conversion of castor oil in hydrocracking at temperature 500 °C.

Catalyst	Conversion (%)	
H-bentonite	59.48	
Al ₂ O ₃ -bentonite	57.93	
Cr/Al ₂ O ₃ -bentonite	64.03	

Table 6. Ten highest peak of GC-MS analysis of castor oil hydrocracking product using Cr/Al₂O₃-bentonite catalysts.

Prediction compound	Molecular	Retention time	Percentage of area
	formula	(min)	(%)
Aceton	C_3H_6O	2.21	1.97
Acetic acid	$C_2H_4O_2$	2.46	1.83
Methyl benzene	C_7H_8	3.79	0.29
Octane	C_8H_{18}	4.25	1.01
Heptanal	$C_7H_{14}O$	7.14	30.16
2-Octanone	$C_8H_{16}O$	9.95	2.47
Acetamide	$C_{13}H_{22}N_2O_2$	22.98	1.15
1-Undecanoic acid	$C_{11}H_{20}O_2$	26.93	35.50
9-Oktadecenal	$C_{18}H_{34}O$	27.50	1.16
10-undecenoic acid	$C_{19}H_{36}O_2$	36.46	1.39

Hydrocracking products were analyzed by using gas chromatography-mass spectrometry (GC-MS) to determine the character product qualitatively. Table 6 presented the list of 10 highest peaks hydrocracking products of Cr/Al_2O_3 -bentonite catalyst. The C_5 - C_{10} compounds were acetone, acetic acid, methyl benzene, octane, heptanal and 2-octanone with retention time of 2.21-9.95 minutes. Biogasoline fraction precentage which was obtained from C_5 - C_{10} carbon was 37.73%. This showed that the hydrocracking product contained mixtures of aliphatic, cyclic, aldehydes, ketones and carboxylic acid. Hydrocracking product also contained large hydrocarbon fraction such as 1-undecanoic acid, acetamide, 9-octadecenal and 10-undecenoic acid.

Conclusion

Based on the data, we can conclude that pillarization of bentonite using Al_2O_3 increased the basal spacing by 1.96 nm with Δd_{001} 1.01 nm while impregnation process gives the highest surface acidity by 8.26 mmol ammonia/g. Chrom/ Al_2O_3 -bentonite could be used as a catalyst for hydrocracking process of castor oil since it gave the highest conversion by 64.03% where the main product was light hydrocarbon fractions of C₅-C₁₀.

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