MJFAS MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCES
PRINT ISSN: 2289-5981 I ONLINE ISSN: 2289-599X

RESEARCH ARTICLE

Catalytic pyrolysis of Asbuton into liquid fuel with Zeolite as catalyst

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Article history
Received 3 April 2019
Revised 12 May 2019
Accepted 18 September 2019
Published Online 15 April 2020

Abstract

Natural Buton Asphalt (Asbuton) is a naturally occurring asphalt that is contained in rock deposit located in Buton Island, Indonesia. Asbuton is mostly used as a mixture of bitumen since it has the potential to be cracked into hydrocarbon and produced as a liquid fuel for energy consumption. The present study aims to investigate the effect of pyrolysis temperature and the mass ratio of the Asbuton with catalyst on the Asbuton conversion. The pyrolysis process is carried out on a batch using vacuum reactor with various temperatures and mass ratios of catalyst to Asbuton. The gas coming out of the process is passed through the condenser, where the condensed gas (liquid product) is collected in the flask, whereas the uncondensed gas (gas product) is collected in a gas holder and the yield is analyzed upon the pyrolysis process completion. The respond parameter of the catalytic pyrolysis are oil flammability, yield, and oil density. The synthesized ZSM-5 catalyst is more effective for the Asbuton bitumen cracking process as opposed to the Natural Zeolite. Furthermore, it is investigated that the most optimum operating condition throughout this experiment was 70.07% and obtained at 350 °C with 9% ZSM-5 catalyst. In terms of product characterization, the liquid product can be ignited during the flame test. From the S.G. and API gravity values, it is suggested that the products belong to crude oil range, and thus, confirming that Asbuton has great potentials to be developed into alternative fuel.

Keywords: Asbuton, catalytic pyrolysis, zeolite

INTRODUCTION

The decreasing amount of light oil and the increasing global demand for energy and environmental concerns, such as global warming has made research into alternative sources of fuel promising. Various alternative sources of fuel are starting to be discovered, such as biodiesel (Marco *et al*., 2016), biofuel (Stepachevaa *et al*., 2016), biogas (Tan *et al*.,2018), and biochar (Huang *et al*, 2015; Mohammad Aziz *et al*, 2018; Abdullah *et al*, 2017). In Buton Island, there are natural asphalt reserves contained in rock deposits that reach up to 650 million tons, which makes Indonesia among one of the largest natural asphalt-producing countries in the world. Due to its abundance, Asbuton has a great potential to be developed into an alternative fuel source. Furthermore, the asphalt bitumen content in Asbuton varies between 10–40% (Indonesian Ministry of Public Works, 2006), which is quite large compared to that of other countries such as United States (12–15%) and France (6–10%). In terms of quality, Asbuton is relatively comparable with oil asphalt. So far, Asbuton has only been used as an asphalt mixture for road construction in the form of fine Asbuton, Asbuton micro, Asbuton grains, and partially extracted Asbuton. In contrast, the use of bitumen as a liquid fuel is still not widely known.

A number of methods for energy recovery from sand oil has been studied, such as extraction of hot air (hot water process), cold air extraction (cold water process), supercritical extraction, solvent extraction (Ma and Li, 2012), and pyrolysis (Jia, *et al*., 2015; Shin, *et al*.,2017; Xiaolong, *et al*., 2017). Hot water processes are used in Canada for commercial bitumen recovery from sand oil. However, since the properties of Asbuton are not compatible with the nature of Canadian oil sand, the extraction of hot water is not suitable for Asbuton. The Buton oil sand is an oil-wet type of oil sand and its mechanical strength and rigidity are higher than those of oil sands in other areas (Ma and Li, 2012).

Furthermore, Asbuton has also been studied via extraction with organic solvent. Specific solvent is to be used for the extraction, however, there is no solvent extraction process commercialized available at the moment because of the difficult solvent recovery process, and thus, making the overall process economically unfeasible. In addition to extraction, pyrolysis process has also been used to extract the bitumen from Asbuton. Previous study by Ma and Li (2012) suggests that the bitumen content extracted from the Asbuton using this method reached up to 30%. The experiments were carried out with the final temperature of 600 °C pyrolysis. This study reports that pyrolysis with low activation energies focuses mainly on changes in adsorbed hydrocarbons and the breakdown of weak chemical bonds, such as C-O bonds and C-S bonds. Simulated distillation on liquid products shows a fraction with a boiling point below 180 °C is a gasoline fraction, diesel fractions range from 180 °C to 360 °C, and a fraction higher than 360 °C belongs to heavy oil. The results of this study illustrate that the main fraction in liquid products of pyrolysis is diesel, about 52%, and the yield of gas products from pyrolysis is less than 5%.

Liu et al. (2014) studied the thermo gravimetric properties of Asbuton pyrolysis kinetics. The results of this study indicate that pyrolysis is an effective method used to separate and recover bitumen from mineral rock (sandstone host). The final temperature of pyrolysis was reported at 550 °C in vacuum condition. In addition, Zhao and Yu (2011) conducted a study of kinetics of asphaltene thermal hydrocracking and catalytic hydrocracking reactions. It was reported that bituminous residues contain a large number of macromolecules such as asphaltenes. High concentrations of hetero-atoms, metals, carbon, and polar compounds in asphaltenes interfere with upgrading and bitumen conversion in the presence of processes such as coke formation and catalyst deactivation. This study compared the thermal hydrocracking and catalytic hydrocracking, as well as showing that the catalytic hydrocracking process promotes liquid production and inhibits coke formation effectively (Zhao and Yu, 2011).

Junaid et al. (2009), on the other hand, studied the bitumen Athabasca cracking by using Natural Zeolite catalysts of Chabazite and Clinoptilolite. The results of this study suggest that Natural Zeolites can break down heavy molecules, especially asphaltenes and light hydrocarbons such as pentane and hexane. In addition to utilizing Natural Zeolite as a catalyst, a Synthesized Zeolite (ZSM-5) was also used for the comparison of catalytic pyrolysis of Asbuton. The ZSM-5 is widely used in the petrochemical industry as a fluid catalytic cracking catalyst. The charge-balancing proton (H^+) species are in the OH– group of the Si(OH)Al species, which could act as Brönsted centers in the catalytic processes. The Brönsted sites of Zeolites are responsible for the cracking of hydrocarbon molecules. They are also considered the active sites for the isomerization and alkylation reactions. HZSM-5 with SiO_2 to Al_2O_3 molar ratio of 25 and 30 demonstrates the largest number of Brönsted acid sites, which are responsible for the cracking of hydrocarbon molecules (He *et al.,* 2017).

Based on the presented studies, it is suggested that the Zeolite catalyst can be effectively utilized for the process of bitumen cracking in the Asbuton. In this research, Asbuton pyrolysis is carried out in various operating temperatures with or without the use of Zeolite catalyst. A Natural Zeolite (Zeolite Clinoptilolite) and Synthetic Zeolite (ZSM-5) are used in this experiment. The catalytic pyrolysis performance is evaluated by flame test, comparing the yield of the product, as well as characterization of oils by density.

EXPERIMENTAL

Materials

The materials used were Asbuton derived from Lawele, Southeast Sulawesi, Indonesia; Trichloroethylene (TCE, Asahi Glass, 99%) for the initial bitumen analysis; Zeolite Clinoptilolite (Natural Zeolite); Commercial Zeolite Socony Mobil-5 (ZSM-5 (mesh 270) Pingxiang Naike Chemical Industry, SiO_2: Al_2O_3 = 25:1).

Raw material preparation

The purpose of the raw material preparation was to make the diameter of the Asbuton particle uniform by doing size reduction with a crusher or hammer with a sieve of 10 mesh and 20 mesh.

Catalyst activation

Catalyst activation was carried out by calcination in furnace under 550 °C for 4 h to create the protonated form of HZSM-5 prior to storing it in desiccator. The charge-balancing proton (H+) species are in the OH– group of the Si(OH)Al species, which could act as Brönsted centers in the catalytic processes. The Brönsted sites of Zeolites are responsible for the cracking of hydrocarbon molecules (He *et al.,*2017).

Catalyst characterization

Catalyst characterization for ZSM-5 was performed using Scanning Electron Microscopy Analysis (SEM) for morphology (texture) and crystal structure of the catalyst, Brunauer-Emmett-Teller Analysis (BET) to get the value of the surface area and porosity of the catalyst. Next, X-Ray Fluorescence (XRF) was used to determine the composition of the catalysts that are capable of producing more cracked bitumen. lastly, X-ray powder diffraction (XRD) was applied to obtain the crystal size of catalyst.

Analysis of initial bitumen content

Bitumen content in rocks was analyzed by extraction method using Soxhlet according to SNI 03-3640-1994. The water content in the Asbuton rock was removed by heating in the oven at approximately 105 °C. The mass of initial Asbuton was weighed and extracted using a Soxhlet. Trichloroethylene (TCE) was used as a solvent due to its suitability for organic compounds (bitumen). The mass of the left-over mineral was calculated as follows:

$$Initial\ bitumen\ mass = \frac{mass\ of\ Asbuton - mass\ of\ mineral}{mass\ of\ Asbuton} \quad (1)$$

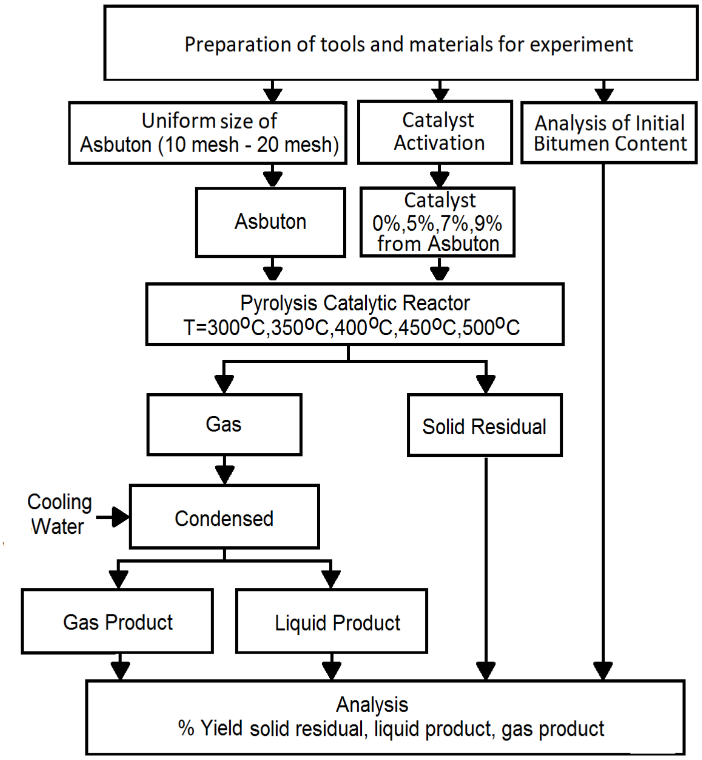

Fig. 1 Experimental flow diagram.

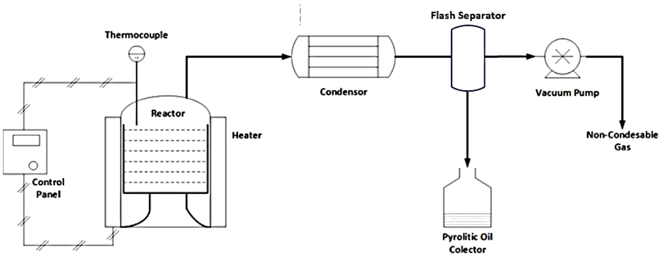

Fig. 2 Catalytic pyrolysis scheme.

Cracking bitumen in asbuton

Bitumen cracking process was carried out by pyrolysis process with and without catalyst under vacuum condition in the stainless-steel reactor. In this process, various mass compositions of Asbuton and catalyst were prepared before being inserted into stainless steel glass and stirred. The mass of Asbuton was a fixed variable while the mass of catalyst was varied depending on variable (mass%) as shown in Fig. 1. The catalytic pyrolysis scheme is presented on Fig. 2. The stainless-steel glass is then put inside the reactor. Vacuum pump and heater are utilized to obtain the desired vacuum pressure and temperature, respectively as shown in Fig. 1. A spiral condenser was used to condense the gas stream so that liquid product can be drained in an Erlenmeyer flask. The uncondensed gas was securely trapped in gas bags. The pyrolysis process was kept for 2 h until the liquid product stopped dripping. The yield analysis for each product was carried out accordingly.

Analysis of product

% Yield of product

$$\% \, Yield \, of \, product = \frac{mass \, of \, product}{mass \, of \, bitumen \, in \, Asbuton} \, x \, 100\% \qquad (2)$$

Characterization of oils by density

The liquid product was measured by DMA 35 Portable Density Meter, followed by the calculation of the specific gravity according to ASTM D 1298 standard at 60 °F using Eq. (3) and (4) (Chemstations, 2004):

$$API_{60°\mathrm{F}} = \frac{141.5}{s.g_{60°\mathrm{F}}} - 131.5 \qquad (3)$$

$$S.G._{60°\mathrm{F}} = \frac{\rho_{i60\ °\mathrm{F}}}{\rho_{H2O60\ °\mathrm{F}}} \qquad (4)$$

where $API_{60°\mathrm{F}}$ is API gravity, $S.G._{60°\mathrm{F}}$ is Specific gravity, $\rho_{i60\ °\mathrm{F}}$ is density of component i at 60 °F, and $\rho_{H2O60\ °\mathrm{F}}$ is density of water at 60 °F (Chemstations, 2004).

RESULTS AND DISCUSSION

Catalyst characterization

The ZSM-5 catalysts were characterized by Scanning Electron Microscopy (SEM), X-Ray Fluorescence (XRF), Brunauer-Emmett-Teller (BET), and X-ray powder diffraction (XRD).

SEM was conducted on the ZSM-5 catalyst to determine whether the desired morphological structure of the catalyst has been achieved. As can be seen from Fig. 3, the ZSM-5 has a microcrystalline and molecular porous structure. This morphology is considered to be able to make the bitumen cracked. The porous catalyst with very small pores contains small molecules but prevents large molecules from entering.

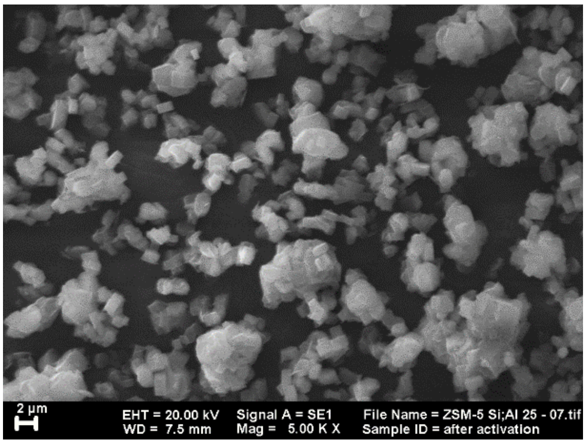

Fig. 3 Morphology of ZSM-5 Zeolite catalyst with 5000x magnification.

XRF method is a non-destructive analysis technique used to identify and determine the elemental composition of solids, powders, or liquid samples. It is used to determine the composition of the catalysts that are able to produce more cracked bitumen.

Table 1 The concentration of elements in the ZSM-5

Chemical Element	Concentration (%)	Chemical Compound	Concentration (%)
Si	97.8	SiO_2	99.2
Ca	0.95	CaO	0.36
Ti	0.08	TiO_2	0.04
Mn	0.058	MnO	0.02
Fe	0.49	Fe_2O_3	0.185
Cu	0.16	CuO	0.05
Zr	0.08	ZrO_2	0.027
Ba	0.33	BaO	0.07

It can be seen from the XRF test results in Table 1 that Si is the most abundant component of the ZSM-5 used in this experiment, thus, the ZSM-5 can be classified as a High-Si Synthetic Zeolite catalyst. Zeolites with high Si content have hydrophilic properties and can act as acid catalysts for hydrocarbons. Metal oxides found in ZSM-5 can act as a buffer in the pores of the ZSM-5, so that the active side of the catalyst remains open. Thus, making it effective to help the bitumen cracking process. Commercial synthetic catalysts are generally composed of 85 to 90% silica (SiO2). Cracking catalysts are insulator catalysts possessing strong acidic properties by altering the cracking process mechanisms through an alternative mechanism involving chemisorption by proton donation and desorption, which result in cracked oil. (Speight, 2011)

BET is a test method to determine the physical adsorption process of gas molecules on the surface of a solid as a basis for analyzing the surface area of a particular material. The BET test is used to determine the surface area and total pore volume of ZSM-5.

XRD relies on the dual wave/particle nature of X-rays to obtain information about the structure of crystalline materials and the crystal size of catalyst. The results of the BET and XRD test are shown in Table 2.

Table 2 Catalyst ZSM-5 properties.

Catalyst ZSM-5	Surface Area (m2/g)	Total Pore Volume (cc/g)	Crystal Size (nm)
Before Activation	224.9	0.4299	101.25
After Activation	353.7	0.8672	99.45

Table 2 shows that after activation, surface area and total pore volume of catalyst increase, causing more active sites to be exposed on the external surface. The ability of the ZSM-5 to crack bitumen lies in its pores so that the long chains of bitumen can be cut into molecules with shorter chains.

The catalyst also has smaller crystal size than before activation as shown in Table 2.The reduction of crystal size leads to higher levels of activity, conversion, and stability (Mohammadparast *et al.,* 2015).

Flame test

Flame test analysis aims to show that the liquid product can produce flame as one of the properties of liquid fuel, as shown in Fig. 4. As can be seen, the liquid product can be ignited, thus, confirming the properties of liquid fuels. This suggests that Asbuton has a great potential to be developed into an alternative fuel.

The effect of catalysts addition to the yield of bitumen in liquid products

Based on the bitumen extraction using Soxhlet with chloroform solvent, the initial bitumen value of 20% of mass was obtained from Asbuton. This value was used as a reference for the next analysis stage. It is expected that a large amount of liquid product can be produced from the bitumen cracking process. The effect of pyrolysis temperature and the amount of Zeolite used on the product of bitumen cracking is presented in Fig. 5. In general, it can be shown that higher amount of catalysts used resulted in the higher yield of liquid product. As indicated from Fig. 5 and Table 2, the highest yield of the liquid product is 61.53% and achieved at 350 °C with a 9% catalyst and decreases as the pyrolysis temperature is raised above 350 °C. The yield of the liquid product increases to the optimum temperature to achieve the highest yield of liquid product and decreases when the pyrolysis temperature is increased.

The effect of ZSM-5 Zeolite on Asbuton with catalytic pyrolysis process is shown on Fig. 6. As shown in Fig. 6 and Table 3, the highest liquid product yield is 70.07% and achieved at 350 °C with a 9% catalyst from Asbuton.

Fig. 4 Flame Test on the liquid product of the catalytic pyrolysis process

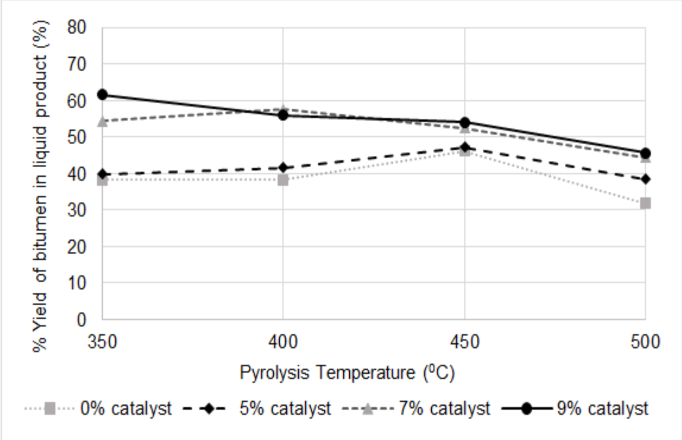

Fig. 5 The effect of catalyst addition at each temperature on yield % of product with Natural Zeolite Clinoptilolite as catalyst.

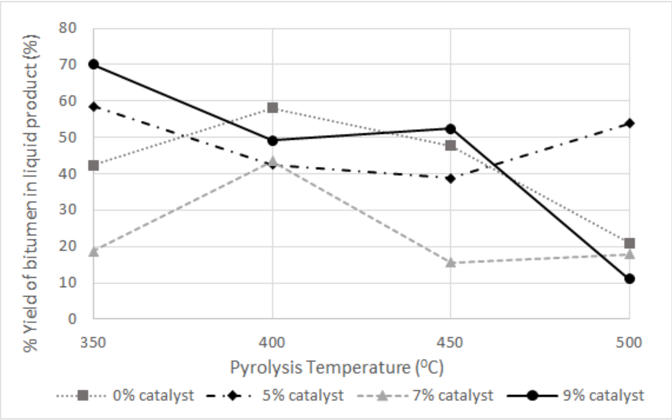

Fig. 6 The effect of catalyst addition at each temperature on yield of product with Synthetic Zeolite (ZSM-5) as catalyst.

The role of the catalyst is made more pronounced by the pyrolysis temperature, as mentioned in a study by Liu et al (2014) in which the bitumen begins to decompose at 200 °C. The pyrolysis process makes the bitumen contained in Asbuton to crack into liquid fuel products.

As can be seen from Fig. 5 and Fig. 6, in general, lower yields of bitumen in the liquid products were obtained with the increasing temperature, with the highest yield being recorded at 350 °C for both natural and ZSM-5 Zeolites-catalyzed processes. Particularly for the ZSM-5-catalyzed processes, some fluctuating yields were investigated as the temperature increases. As shown in Fig. 6, 0% and 7% catalysts have an optimal yield at 400 °C and show reduction in yield when the temperature is increased. Meanwhile, the 9% catalyst has an optimum yield at lower temperature of 350 °C. The addition of catalysts and higher temperatures improve the cracking process and produce smaller chains, resulting in more gas products will be formed than liquid products.

Vamvuka (2011) explained in their work that bitumen pyrolysis process with temperature reaching 600 °C can produce heat resistant material (gas phase or short chain hydrocarbons). Due to the addition of the catalyst, lower pyrolysis temperature would be required to crack the bitumen into short, non-condensable short-chain hydrocarbons. Labadidi et al. (2014) mentioned that asphalt, the main constituent of bitumen, experiences cracking at temperature 380 °C. However, with the addition of a 9% Zeolite catalyst asphalt, bitumen was able to crack at 350 °C. This might be caused by the presence of a catalyst that leads to the decrease of the required energy to crack the bitumen. A study conducted by Ma and Li (2014) revealed that bitumen cracking process produces the most abundant liquid product at a temperature range of 380 °C–480 °C. From Fig. 5 and 6, it can be seen that the highest yield of each catalyst addition lies in the range between 350 °C and 480 °C.

Furthermore, this study particularly points out that the best operating condition for the Asbuton bitumen pyrolysis in order to get the highest yield of liquid product was at 350 °C with 9% natural Zeolite Clinoptilolite catalyst loading, which gave 61.53% yield. In the case of Zeolite ZSM-5 catalyzed pyrolysis, as high as 70.07% yield was obtained with the same operating conditions, i.e. at 350 °C with 9% catalyst loading. The ZSM-5 shows better result than Natural Zeolite because it has better formation and composition, the ratio of Si and Al can be that can be specified for better selectivity cracking, and less impurity detected.

Characterization of oils by density

The density of the liquid product for each catalyst used is shown in Table 4. A specific gravity was measured according to ASTMD 1298 standard at 60 °F (Chemstations, 2004). The specification standard based on the specific gravity or API of Handbook of Petroleum Product Analysis by Speight (2002) is shown in Table 5. The obtained results confirm that the liquid products have property specification as crude oil (the API value < 10) that need further fractionation process to produce specific fuels.

Table 3 Optimal condition of product in each catalyst percentage.

Catalyst Percentage	Natural Zeolite		Synthesize Zeolite	
	Optimal Pyrolysis Temp.	Yield of Product	Optimal Pyrolysis Temp.	Yield of Product
0%	450 ^{0}C	46.15%	400 ^{0}C	58.05%
5%	450 ^{0}C	47.13%	350 ^{0}C	58.63%
7%	400 ^{0}C	57.57%	400 ^{0}C	43.53%
9%	350 ^{0}C	61.53%	350 ^{0}C	70.07%

Table 4 Density, Specific Gravity, and API results of liquid products.

Catalyst Percentage	Specification	Temperature (^{0}C)			
		350	400	450	500
0%	Density (g/cm^3)	1.0047	1.0037	1.0038	1.0147
	S.G.	1.0057	1.0047	1.0048	1.0157
	API	9.1944	9.3346	9.3205	7.8078
5%	Density (g/cm^3)	0.9998	1.0024	1.0026	1.0011
	S.G.	1.0008	1.0034	1.0036	1.0021
	API	9.8840	9.517	9.4891	9.7003
7%	Density (g/cm^3)	1.0007	1.009	1.014	1.012
	S.G.	1.0017	1.010	1.0150	1.0130
	API	9.7568	8.5948	7.9040	8.1795
9%	Density (g/cm^3)	1.016	1.019	1.025	1.018
	S.G.	1.0170	1.0200	1.0260	1.0190
	API	7.6296	7.2199	6.4080	7.3562

Table 5 Specific gravity and API gravity of crude oil and selected products (Speight, 2002).

Material	S.G 60 ◦F/60 ◦F	API gravity Deg.
Crude	0.65–1.06	87–2
Casinghead liquid	0.62–0.70	97–70
Gasoline	0.70–0.77	70–52
Kerosene	0.77–0.82	52–40
Lubricating oil	0.88–0.98	29–13

CONCLUSION

In this study, lower yield of bitumen cracking products was produced in the presence of the Natural Zeolite catalysts compared to those catalyzed by Synthesized Zeolite. Thus, it can be concluded that the synthesized ZSM-5 catalyst is more effective for the Asbuton bitumen cracking process as opposed to the Natural Zeolite. Furthermore, it is investigated that the most optimum operating condition throughout this experiment was 70.07% and obtained at 350 °C with 9% ZSM-5 catalyst. In term of product characterization, the liquid product can be ignited during the flame test. From the S.G. and API gravity values, it is suggested that the products belong to crude oil range, and thus, confirming that Asbuton has great potentials to be developed into an alternative fuel. However, further characterization is required to determine the composition of the liquid products and to determine whether the properties of the liquid products meet the specific criteria of typical liquid fuels.

ACKNOWLEDGEMENT

The authors would like to thank the Department of Research and Society Service (LPPM), Institute Technology Sepuluh Nopember for the financial support provided through Graduate Research Program.

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