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The Effect of Combined H-USY and ZSM-5 Catalyst in Catalytic Cracking of Waste Cooking Oil to Produce Biofuel

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Abstract. The catalytic cracking of waste cooking oil was investigated at various operating temperatures and reaction time in this paper. The purpose of this research was to study the characteristics of biofuel produced through the catalytic cracking process using the combination of catalyst H-USY and ZSM-5 with mass ratio 0.5:0.5 (w/w). The effect of combined catalyst with variation in operating temperatures (400 °C, 450 °C, 500 °C and 550 °C) and reaction times (30, 45 and 60 min) were studied on waste cooking oil after pretreatment. The ratio by mass of waste cooking oil to the amount of catalyst used is 40:1 (w/w). The process was carried out in a flow reactor under the atmospheric pressure (1 atm) and an air flow rate of 100 cc/min. The result showed that the combined catalyst is most effective in the catalytic cracking of waste cooking oil at 450 °C reaction temperature and 60 min reaction time. Under these optimal conditions, the percentage yield by mass of liquid biofuel product was 49.35 % with a product composition of 15.71 % gasoline (C₆-C₁₂), 11.97 % diesel products (C₁₇-C₂₀), and 1.09 % Heavy oil (C₂₀ >).

Keywords: Biofuel, catalytic cracking, H-USY, waste cooking oil, ZSM-5

INTRODUCTION

The stock of world fossil energy will run out in the near future because the production of oil continues to increase but its availability is not renewable. Therefore, in anticipation of future fuel oil crises, renewable energy resources such as biofuel must be developed and utilized [1-8].

Biofuels are one such renewable energy source. Biofuels provide many advantages, namely sustainability, reduction of greenhouse gas emissions, and security of supply [9]. Biodiesel has advantages compared to petroleum-derived oil, it can reduce harmful emissions such as CO and CO₂ in the burning of this fuel didn't produce sulfur emissions (SO₂) it has high cetane number that makes its ignition properties to be exceptionally well [10].

Waste cooking oil (WCO), generated from cooking oil derivation, namely palm oil [11] sunflower oil [12], soybean oil [13], coconut oil [14] etc. has become the worldwide interest [15]. Biofuel production is among the optimum processes to utilize the oil waste in terms of efficiency and economically effective [7]. Waste cooking oil contains fatty acids, mostly palmitic acid (21.47 %) and oleic acid (28.64 %) [16].

The biofuel term refers to the gaseous or liquid fuels widely applied in the transportation which mainly generated from biomass [17]. Biodiesel may be produced by various methods, including trans-esterification and catalytic cracking. Trans-esterification is currently the most extensively applied process, in regards to its simple technical process as well as efficient [18]. The drawback of this method is the high content of glycerol that would be

generated throughout the trans-esterification process [19], which must be removed through a costly extraction process [15]. In contrast to trans-esterification, which can only be applied to create biodiesel, the catalytic cracking can generate gasoline and kerosene [20]. In comparison to trans-esterification, the catalytic cracking of WCO to create bio-oil has been an interesting procedure to manage the waste, in terms of its economic cost, rate of conversion which is relatively high, as well as both energetic and chemical value [21].

Catalytic cracking refers to process of conversion which might be used in a varied feed stocks from gas oil to oil and residuum [22-26]. The catalytic cracking idea is particularly similar to thermal cracking, however, it is different in regards to the application of a catalyst, which theoretically is not consumed in the process. It is one of several practical applications applied in a refinery that employ a catalyst to enhance the efficiency process and product slate. In terms of process parameters, catalytic cracking is typically performed at temperatures ranging from 485 to 540 °C (900–1000 °F) and pressures up to 100 psi [27].

Zeolites are extensively applied in the chemicals and oil refining production in the process of catalytic cracking in the petrochemical industry. The compounds carry a noticeably large surface areas and high adsorption capacity with molecular dimension of the pores [28]. Between the zeolites, H-ZSM-5, H-Beta and H-USY are the most broadly applied for catalytic upgrading processes [29]. Zeolite capacity as cracking catalyst is based on acidity and stability, which is depended on Si/Al ratio. High acidity can increase the catalytic activity, thermal stability and acid resistance. The active core of zeolite which functions in cracking reactions is part of a Bronsted-acid which is centered on Al atoms [30-31].

The purpose of this study was to determine the optimum concentration of combined ZSM-5 and USY catalysts to generate biofuel from waste cooking oil. Y-zeolites have high temperature stability [32]. ZSM-5 showed a moderate selectivity towards hydrocarbons [33] and a medium pore ZSM-5 catalyst has the ability to convert n-alkanes to aromatics which selectively cracks low octane components into higher octane components [34]. The goal of employing a combined catalyst is to utilize the unique advantages of each zeolite to their best effect. The process was conducted in a fixed-bed flow reactor at a constant pressure of 1 atm with variation of reaction time and temperature. The effect of the catalyst on biofuel and the physical properties of hydrocarbons products such as density, dynamic viscosity, calorific value will be discussed in this paper.

Several studies for the production of biofuels by catalytic cracking process using various types of catalysts have been carried out, but the use of combined H-USY with ZSM-5 catalysts for catalytic cracking of waste cooking oil has never been thoroughly investigated. Therefore, the combined catalyst activity in biofuel production and the quality of products produced with waste cooking oil as raw materials need to be further studied.

MATERIALS AND METHOD

Materials

The material used in this research were waste cooking oil, tetraethyl orthosilicate ($\text{Si}[\text{OET}]_4$), tetra propyl ammonium hydroxide (TPAOH), aluminum isopropoxide ($\text{Al}[\text{OPr}]_3$), NaOH, USY catalyst, filter paper, litmus paper and distilled water.

Method

Preparation of Samples

Waste cooking oil was filtered using charcoal from kernel palm shells placed in 2 inch diameter PVC pipe, with a wire filter at the bottom. The filters holds the charcoal, which traps waste, odor, and residue that may be carried along from the frying process and clean waste cooking oil is accommodated in the container bottom. This procedure was according to the method investigated by Nazarudin et al. [35].

Catalyst Synthesis Procedure

The method of ZSM-5 catalyst synthesis was referred to Nazarudin [36] with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 20$ ratio with the following materials weights 25.39 g of $\text{Si}[\text{OEt}]_4$ ($\text{SiC}_8\text{H}_{20}\text{O}_4$), 12.3935 g of TPAOH ($(\text{CH}_3\text{CH}_2\text{CH}_2)_4\text{N}(\text{OH})$), 0.55375 g of $\text{Al}[\text{OPr}]_3$ ($\text{C}_9\text{H}_{21}\text{AlO}_3$), 16.71 g of H_2O and 0.01725 g of NaOH for each ZSM-5 synthesis process. After weighing it down, all the ingredients were mixed in a small Teflon container and stirred using a magnetic stirrer at room temperature ($30\text{ }^\circ\text{C}$) for 24 h until clear solution was formed. Then, the clear solution was put into a reactor for the hydrothermal treatment in an oven at $165\text{ }^\circ\text{C}$ temperature for 5 days. After the ZSM-5 crystal were formed, the crystals were washed with distilled water with the help of a centrifugal pump until it reached $\text{pH} = 7$. Then, it was followed by the drying process at $105\text{ }^\circ\text{C}$ in an oven for 24 h. The catalyst was then calcined for 5 h at $550\text{ }^\circ\text{C}$ temperature to burn the TPAOH template molecule to CO_2 in a stable and heat-resistant catalyst. USY catalyst was bought from Aldrich chemistry Singapore and then calcined at $550\text{ }^\circ\text{C}$ for 5 h to obtain H-USY catalyst, according to USY calcination process in Nazarudin [36].

Catalytic Cracking Process

Equal amounts by mass (0.5 gram) of H-USY and ZSM-5 were mixed in a small container then put in a pyrolysis reactor's bed and then the temperature was set using the computer system and then it can be controlled and monitored through the control panel. The preheating reactor and the pyrolysis reactor filled with the combined catalyst were heated to the target temperatures which were $400\text{ }^\circ\text{C}$, $450\text{ }^\circ\text{C}$, $500\text{ }^\circ\text{C}$ and $550\text{ }^\circ\text{C}$. After it reached the desired temperature, then the waste cooking oil at a ratio of 40:1 (w/w) with the catalyst was injected through the injection port to preheat the reactor to change the phase of waste cooking oil liquid phase into gaseous phase and nitrogen to help to push the waste cooking oil gaseous phase from nitrogen tank flowed through and controlled with flow meter then the waste cooking oil gaseous phase enter the pyrolysis reactor where the catalytic cracking reaction took place and reacted for 30, 45 or 60 min. The condensable gas product from catalytic cracking was condensed into the liquid product in the cooling system product reservoir and the incondensable gas was accommodated in the gas sampling bag. After the experiment, the residual left in the reactor and condensed liquid biofuel products were weighed, and the gas conversion was calculated by subtracting the initial weight with the residue and liquid formed in the final product.

RESULTS AND DISCUSSION

Catalysts Characteristics

X-ray Diffraction (XRD) Analysis

XRD analysis was carried out to identify and determine quantitatively the shape of the ZSM-5 crystalline phase and the crystallinity and types of minerals contained in the zeolite. The characterization of ZSM-5 catalyst was carried out using XRD with 5.0084 initial position of 2° theta and the final position is 59.9864 with continuous scan type, 10 mm specimen length extension and temperature measurement is $-273.15\text{ }^\circ\text{C}$ with Cu as anode material and the diffractometer type is XPert MPD.

Figure 1 shows the diffraction peaks at $2\theta = 8.1085^\circ$, 9.0164° , 23.2667° , 23.4996° and 24.1308° . The types of components of the ZSM-5 catalyst are characterized by the 2θ peak appearance in certain regions of the crystallinity level of the component structure. The peaks in the ZSM-5 diffractogram showed a pattern similar to the standard ZSM-5 diffractogram in Fig. 2 which is 2θ at $7-9^\circ$ and at 23.05° and 23.8° . The result pattern also shows the identical characteristic peaks of ZSM-5 that have been synthesized by Liu et al. [37], Song et al. [38], Treacy et al. [39] and Pan et al. [40]. It can be concluded that the synthesis catalyst is the type of MFI structure. This MFI structure is obtained from the use of TPA+ as a template. The X-ray diffraction pattern showed that the synthesis catalyst consists of MFI phase with high crystallinity and there is no other crystalline phase in the synthesis of ZSM-5 and has the similar structure as the ZSM-5. The Crystallization of ZSM-5 synthesis is fairly good because the ZSM-5 diffractogram shows sharp peaks and good peak separation.

ZSM-5 catalyst has a hexagonal crystal morphology. The results of the analysis with 30000x magnification, as shown in Fig. 3a, show that the ZSM-5 form groups (clusters) with polymorphous (not homogeneous) structures. The results of the analysis show that the crystallization formed is not well aggregated. In the results of the study by Song et al. [38], the particles of ZSM-5 formed groups (clusters) and there was an aggregation between ZSM5 particles. The EDX spectra in Fig. 3b shows the elemental composition of the synthesized ZSM-5 by percent mass. The result of 96.43 %, 3.57 % Al and the trace of Na derived from sodium hydroxide used in the synthesis process of the catalyst with 0 % indicate.

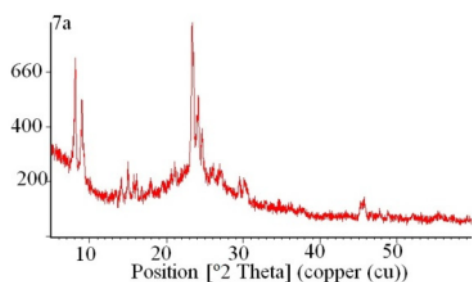


FIGURE 1. XRD Patterns of ZSM-5 catalyst synthesis.

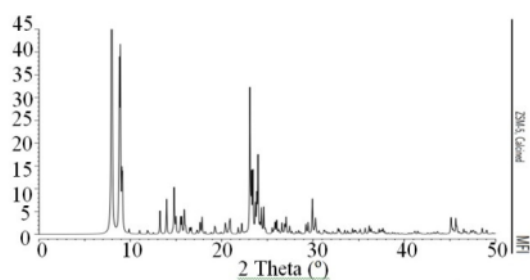
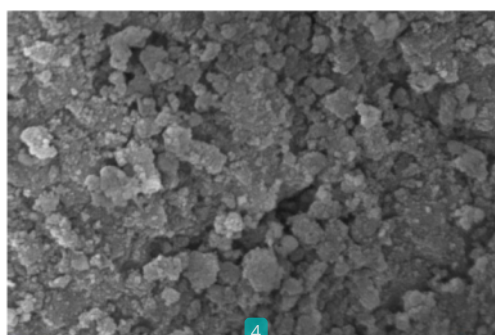
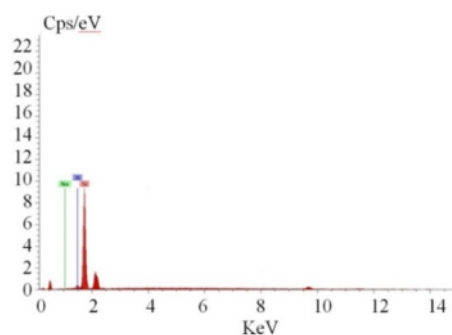


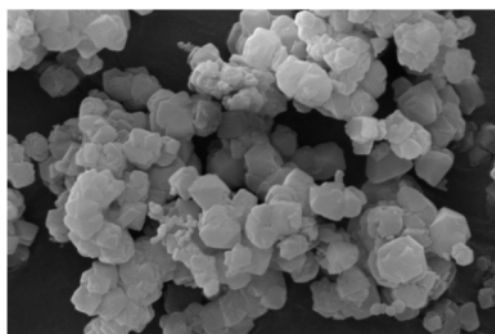
FIGURE 2. ZSM-5 Diffractogram Standard by Treacy et al.



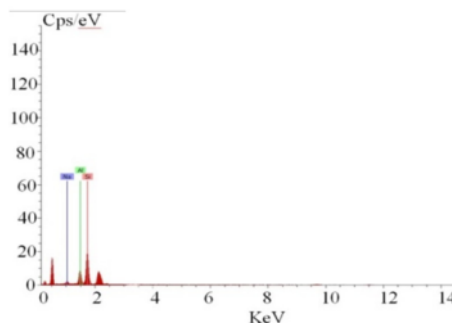
(a)



(b)



(c)



(d)

FIGURE 3. (a) SEM image of ZSM5 catalyst, (b) EDX spectra of ZSM-5 catalyst with the elemental content of the ZSM-5 catalyst, (c) SEM image of H-USY catalyst, (d) EDX spectrum and composition of synthesized ZSM-5 catalysts.

SEM analysis of H-USY catalyst with a magnification of 20000x in Fig. 3c showed the regular crystal structure formed a tabular layer and had a hollow space between the crystal structure because USY catalyst is a zeolite that has a large pore size and faujasite framework type (FAU). This FAU type zeolite has a channel size bigger than ZSM-5 (MFI) or β -Zeolite (BEA). The EDX analysis on H-USY catalyst was carried out to determine the chemical composition contained in the sample. The results of EDX analysis are presented in Fig. 3d and it is shown that the micro-structure of the H-USY sample contains the composition of elements Si, Al, and Na with a relative mass percentage of Si elements of 71.47 %, Al elements of 23.18 % and 5.35 % in the catalyst.

The Effect of Reaction Temperature and Reaction Time in the Catalytic Cracking Process

The catalytic cracking process is carried out in a fixed-bed flow reactor by inserting the combined catalyst H-USY and ZSM-5 with 1:1 ratio (w/w) into the catalyst bed in a vertical reactor and waste cooking oil is inserted through the horizontal reactor after being heated to the target temperature. The products obtained were analyzed using gravimetric analysis, with the temperature variables used are 400 °C, 450 °C, 500 °C and 550 °C with ratio catalyst to raw material is 1:40 (w/w).

The effect of reaction time on biofuel yield in the catalytic cracking process of waste cooking oil using combined H-USY and ZSM-5 catalyst can be seen in Table 1 that a reaction time of 60 min results in the highest yield of liquid biofuel products (49.35 %) at 450 °C reaction temperature and the pattern showed that at 30 min reaction time produce the smallest amount of yield liquid product at 550 °C temperature reaction for the overall observation while for the other different temperature reaction and time reaction for every testing is varied [41].

Density Analysis

Density is one of the physical properties of biofuels that shows the weight ratio of biofuels in a certain volume, the density expresses in g/cm^3 . The longer the chain of fatty acids, the higher the density. The level of saturation also affects the density which is the value of the density of a product will decrease with the increase number of double bonds in the product. The density of liquid biofuel of catalytic cracking waste cooking oil at 450 °C reaction temperature and 60 minutes reaction time with a combined H-USY and ZSM-5 catalyst is 0.93384 g/cm^3 . The initial density of waste cooking oil is 0.9772 g/cm^3 , it can be concluded that there is a decrease in the density after the catalytic cracking process due to the decreasing chain of fatty acids because it has undergone a cracking process into the biofuel product with hydrocarbon fuel chains such as C_6 - $_{12}$ (gasoline) and C_{13} - C_{20} (diesel).

Calorific Value Analysis

The Calorific value analysis process uses an Automatic Calorimeter PARR 6400 to determine the calorific value in units of cal/g by the isoperibol or adiabatic method. This instrument combines the closed loop sub-cooling system into the calorimeter. This sub-system uses a thermo-electric cooling assembly that is installed directly into a one liter water tank that supplies cooling water to the calorimeter. An externally pressurized nitrogen tank is used to supply rinse water to the calorimeter. The calorific value indicates the energy contained in the fuel per unit mass of fuel. This calorific value is important to measure the energy content of each fuel mass so that consumption to produce certain energy can be calculated correctly. The calorific value of the liquid biofuel product is 38.8850 (Mj/Kg).

Viscosity Analysis

The viscosity was measured by using a KV-6 viscometer bath from GRHA Pertamina Indralaya laboratory. Kinematic viscosity was determined by measuring the flow time of the sample through a calibrated capillary viscometer at 40 °C based on the law of gravitation. The viscosity of the liquid biofuel produced by catalytic cracking waste cooking oil at 450 °C reaction temperature and 60 min reaction time with a combined H-USY and ZSM-5 catalyst is 5.567 cSt.

5 Gas Chromatography–Mass Spectrometry (GC-MS) Analysis.

From all data, then, one sample with the best liquid yield biofuel which is catalytic cracking at a reaction temperature of 450 °C and reaction time of 60 min was then tested for GCMS to determine the content and the components in the biofuel. The results obtained are presented in the Fig. 4, it can be seen that there are 100 peak areas. The retention time data show the type of compound contained in the sample tested by comparing it with the standard data contained in the chromatogram. These 100 peak areas are then grouped based on the amount of carbon atoms it has. The retention time of highest peak area and the other peak area data are combined together based on the same amount of carbon atoms in the compounds.

GC-MS data shows that 12.57 retention time is 1-undecene (C₁₁H₂₂) compound with the highest peak area of 2.57 % as the representation of the hydrocarbon's contents with 11 atom carbons in the product with the total of 11.01 peak area for hydrocarbon with 11 carbon atoms. The data then for each compound was grouped based on the length of the carbon chain. The results of the analysis shows that the product contains 31.31 % area of gasoline (C₅-C₁₂), 24.26 % area of diesel (C₁₃-C₂₀), 2.21 % area of heavy oil (C > 20), 25.49 % acidic compounds and the rest is other fraction with 16.72 % area.

TABLE 1. The Result of Catalytic Cracking with different reaction temperature and reaction time using combined H-USY and ZSM-5 catalyst.

Temperature (°C)	Time (minutes)	Waste Cooking Oil (gram)	Liquid Yield (%)	Residue (%)	Gas Yield (%)
400	30	40.67	41.53	8.21	50.26
450		40.95	40.93	22.56	36.51
500		38.41	21.92	16.43	61.65
550		40.29	21.77	13.25	64.98
400	45	39.74	46.45	15.68	37.87
450		41.46	32.18	22.65	45.18
500		40.98	48.05	2.76	49.19
550		40.05	21.05	16.70	62.25
400	60	41.23	39.07	13.24	47.68
450		40.08	49.35	6.16	44.49
500		40.13	24.99	13.28	61.72
550		40.63	27.69	12.21	60.10

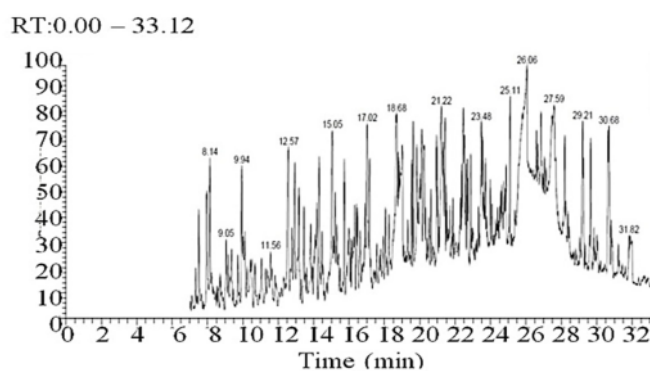


FIGURE 4. GC-MS analysis data of catalytic cracking WCO at 450 °C reaction temperature and 60 minutes reaction time.

CONCLUSION

Reaction time and temperature have significant effects on the production of biofuel from waste cooking oil by catalytic cracking. The highest yield liquid was obtained by catalytic cracking using combined H-USY and ZS-5 is 49.35 %. The hydrocarbon contents in the product of catalytic cracking process were diesel oil (C₁₃ – C₂₀) with 15.37 % area, C₆-C₁₂ (gasoline) with 2.21 % Area of heavy oil (C >20). The biofuel product has a calorific value of 40.0906 MJ/Kg, density of 9.069 g/cm³ and kinematic viscosity of 9.2441 cSt.

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