REACTIONS OF USED MOTOR OIL AND RESIDUAL OIL USING MESOPOROUS Mo/Al$_2$O$_3$ CATALYST

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ABSTRACT

In the present study, mesoporous Mo-based catalysts of different pore size ranges were synthesized and investigated for its hydrocracking activity. Catalyst pore size has been considered a property affecting the mass transfer during reaction especially when dealing with large molecules such as residual oil. The effects of catalyst pore size on catalytic activities were evaluated via reaction conversion and liquid selectivity. Catalytic hydrocracking activity was also compared among the hydrocracking of used motor oil and residual oil. It was observed that the conversion is mainly related to the catalyst acid capacity, whilst the liquid products distribution in used motor oil is affected by catalyst pore size. Catalyst acid capacity promotes the cracking activity.

Keywords: Hydrocracking, Mesoporous Catalyst, Residual Oil, Used Motor Oil

1.0 INTRODUCTION

Declining conventional light crude reserves with the increasing world energy demand leads to the higher amount of heavy crude oil or residual oil being upgraded into lighter distillates. Residual oil contains low H/C atomic ratio, high concentration of asphaltenes, and heteroatoms that need more refining than conventional crude oil. The presence of these bulky and heavy molecules makes the catalytic processing of heavy oil more complex and need further attention. Among the heavy crude oil upgrading processes available, hydrocracking is the most promising route to produce excellent qualities of middle distillates with low aromatics, sulfur and nitrogen contents [1].

In hydrocracking process, a bifunctional catalyst with cracking and hydrogenation-dehydrogenation functions is required. However, the critical problem in hydrocracking process is deactivation of the catalyst, especially when dealing with residual oil. The pore mouths of catalyst are usually blocked by metal and coke concentrated near the outer surface of the catalyst [2; 3]. The activity of micropores catalyst is limited to small molecules and the micropore may not accommodate large molecules in heavy oil or residual oil. This limitation normally leads to pore mouth blockage. On the other hand, macroporous catalyst possesses small surface area and it is expected to have low dispersion of active sites [4]. Hence, the existence of mesopores in catalyst not only provides the proper paths for the reactants and products, but also expose the acid sites easily accessible for large molecule reactants [5]. Mesopores catalyst could be a potential catalyst to improve the hydrocracking activity. Recently, several mesoporous materials were reported

to be able to enhance the performance of catalysts in the hydrocracking activity [5-10]. However, it is also important to consider that the preferable pore size range may vary with the properties of feedstock due to the different chemical nature of the feedstock during the hydrocracking reactions. When heavy feeds are involved in the hydroprocessing activity, the pore size of a catalyst is much more important than the surface area [11, 12]. Though there are limited studies, the significant effect of pore structure on the hydrocracking of residual oil have been well reported. Alumina-supported catalysts with mesopore diameters of 7-20 nm showed higher activity in the hydrocracking of Athabasca oil sand bitumen than catalysts with pore sizes of 3-7 nm [13]; Ying *et al.* [14] demonstrated that catalysts with pore sizes of 25.2 nm and pore volumes of 0.87 cm^3/g achieved relatively higher conversions (63 wt%) of atmospheric residue into lighter distillates (b.p. <215°C) compared to catalysts with smaller pore size. Mesoporous NiMo/γ-Al$_2$O$_3$ catalyst with a pore size distribution of mesorange 10-20 nm presented the highest activity for the conversion of Kuwait vacuum residue to light distillates [15]. Nevertheless, due to the complex mixture of hydrocarbons in petroleum, the relations between catalyst pore size and its activity performance in hydrocracking reactions remains unclear. The catalyst deactivation remains an issue in petroleum hydrocracking.

The objective of the present study is to study the effects of catalyst pore size and related characteristic on hydrocracking reaction in different feedstock: used motor oil with lower boiling range hydrocarbons source and residual oil with higher boiling range hydrocarbons source. The conversion of used motor oil

into valuable diesel fuel products is favorable as this not only reduces the environmental problem due to waste oil pollution but also prolong the lifetime of oil resources owing to recycling of the oil.

2.0 MATERIALS AND METHODS

2.1 Catalyst Preparation

Mesoporous alumina of different textural properties were prepared via sol-gel method according to May *et al.* [16]. The molar composition of the mesoporous aluminas prepared were as follows: 1 aluminum alkoxide: 0.2 surfactant: 10 solvent: 4 water by varying the parameters during the preparation of alumina, which were aluminum precursor with corresponding solvent and surfactant. The alumina precursor and solvent used were: aluminum secbutoxide (Acros, 97%) with sec-butanol (Acros, 99%), aluminium iso-propoxide (Acros, ≥98%) with propan-2-ol (Merck, ≥99.9%), or aluminum tri-ethylate (Merck, 97%) with ethanol (Systerm, 95v/v%); while surfactant used was stearic acid (Acros, 97%). The detail procedure of preparation has been reported by Looi *et al.* [17]. Mesoporous alumina of small (3.3 nm), medium (9.0 nm) and large (18.9 nm) pore size were selected and prepared. Sample ID of synthesized mesoporous aluminas is based on the pore size of alumina, which is 3.3 nm (S), 9.0 nm (M), 18.9 nm (L) and 19.1 nm (L-E).

The alumina synthesized was then loaded with 18 wt% of Mo by incipient wetness impregnation method with an appropriate amount of aqueous solution of ammonium molybdate (VI) tetrahydrate (Acros). The impregnated MoO_3/Al_2O_3 catalyst was dried at 120°C for 16 h and followed by calcination at 450°C for 3 h.

2.2 Catalyst Characterizations

Brunauer-Emmett-Teller (BET) specific surface area (SA) of samples were measured by nitrogen adsorption/desorption analysis using Micromeritics ASAP 2020 at 77 K. Barret-Joyner-Hallender (BJH) model on desorption branch was employed to calculate the pore size distributions (PS) and pore volume (PV). The micropore surface area of aluminas was obtained from *t*-plot.

Transmission electron microscopy (TEM) images were obtained by using Phillips electron microscope (CM12) which operated at 120 kV. The powder samples were then dispersed in ethanol solution and dropped on a carbon film supported on a 400 mesh copper grid.

Temperature-programmed desorption of ammonia (NH_3-TPD) was used to measure the acid capacity of catalyst. It was performed by using an AutoChem II 2920. The sample was degassed at 120°C for 1 h in flowing helium to remove water vapour and cooled down to 50°C at a rate of 5°C/min. The sample was then exposed to 15% NH_3 in He for adsorption for 1 h until saturation. Finally, TPD was recorded from 50°C to 500°C at a ramping rate of 10°C/min.

2.3 Catalyst Activity Evaluation

Used motor oil or residual oil, used as the feedstock for hydrocracking reaction, was collected from a local workshop and refinery atmospheric distillation bottom. The mesoporous Mo/Al_2O_3 catalyst activity test was carried out in a high pressure batch reactor (Parr 4570). First, 70 g of feedstock was loaded into the reactor with 0.50 wt% of catalysts. The batch reactor was

then pressurized with H_2 gas to 500 psig at ambient temperature. This was followed by heating up the reactor to 400°C. After 1 h of hydrocracking reaction, the reactor was cooled down to room temperature. The reactor was depressurized before unloading. The liquid product was then recovered. Coke which is defined as dichloromethane-insoluble may be formed during the reaction was filtered using a membrane filter. The weight of gas produced during reaction was determined by the yield mass balance before and after a reaction. The liquid products were analyzed for their product distribution according to ASTM D86 to determine the yield of the lighter products after hydrocracking reaction. The conversion and the liquid products fraction are defined as in Eq. (1) and Eq. (2):

$$\frac{\text{wt of fraction}_{>340°C \text{ before reaction}} - \text{wt of fraction}_{>340°C \text{ after reaction}}}{\text{wt of fraction}_{>340°C \text{ before reaction}}} \times 100\% \quad (1)$$

$$\text{Liquid products fraction (vol\%)} = \frac{v_D}{v_{CO}} \times 100\% \quad (2)$$

where v_D is the volume of distillate fraction, while v_{CO} is the total volume of converted oil. The liquid product fractions were classified into gasoline (b.p. < 150°C), kerosene (b.p. 150-250°C), diesel (b.p. 250-340°C) and residue (b.p. > 340°C) fractions.

The experiments were repeated several times and the experimental errors associated with the reported data were estimated. The errors associated with the residual oil conversion was ±0.5 %, while for products yield and liquid oil fractions data were ±1.5 %.

3.0 RESULTS AND DISCUSSION

3.1 Physical Properties of Mesoporous Alumina

Aluminas of different textural properties in the present study were prepared by tuning certain parameters during preparation of alumina, such as aluminum precursor and surfactant [18; 19]. All the aluminas chosen consist primarily of mesoporosity and different textural properties. These are given in Table 1. These mesoporous aluminas possessed large surface area (300-380 m^2/g) generally. Al-S and Al-L exhibited some micropore surface area (38.3 and 29.0 m^2/g, respectively), whilst Al-M and Al-L-E possessed 1.9 and 0.71 m^2/g small micropore surface area. The pore volume of alumina increased with its pore size as expected. Generally, alumina with smaller pore size (3 nm) exhibited smaller pore volume (0.54 cm^3/g), and alumina with larger pore size possessed correspondingly larger pore volume (2.12 cm^3/g).

Table 1: Textural properties of prepared mesoporous alumina

Sample ID	BET surface area (m^2/g)	Micropore surface area[a] (m^2/g)	BJH pore size (nm)	BJH pore volume (cm^3/g)
Al-S	383	38.3	3.3	0.54
Al-M	301	1.9	9.0	0.90
Al-L	313	29.0	18.9	2.12
Al-L-E	338	0.71	19.1	2.09

[a] Obtained from *t*-plot

Type IV was observed in all isotherms, agreeing that all aluminas prepared were mesoporous materials. The hysteresis loops for aluminas Al-M, Al-L and Al-L-E can be categorized to H2 type [20]. It is usually referred to the presence of 'ink-bottle' type pores in the mesopore structure. On the other hand, the hysteresis loop for alumina Al-S prepared in the presence of surfactant showed H1 type. This indicates that Al-S exhibits a mixture of cylinder type of pores [20]. Pore size distribution of mesoporous aluminas are given in Figure 1(b). Al-S alumina showed narrow pore size distribution. Al-M alumina displayed the medium pore size distribution, and broader pore size distributions were observed for aluminas Al-L and Al-L-E, recognized by the presence of large pore size. Al-L-E gave a trimodal distribution with the major one at 18.9 nm and the other two at 47 nm and 63 nm.
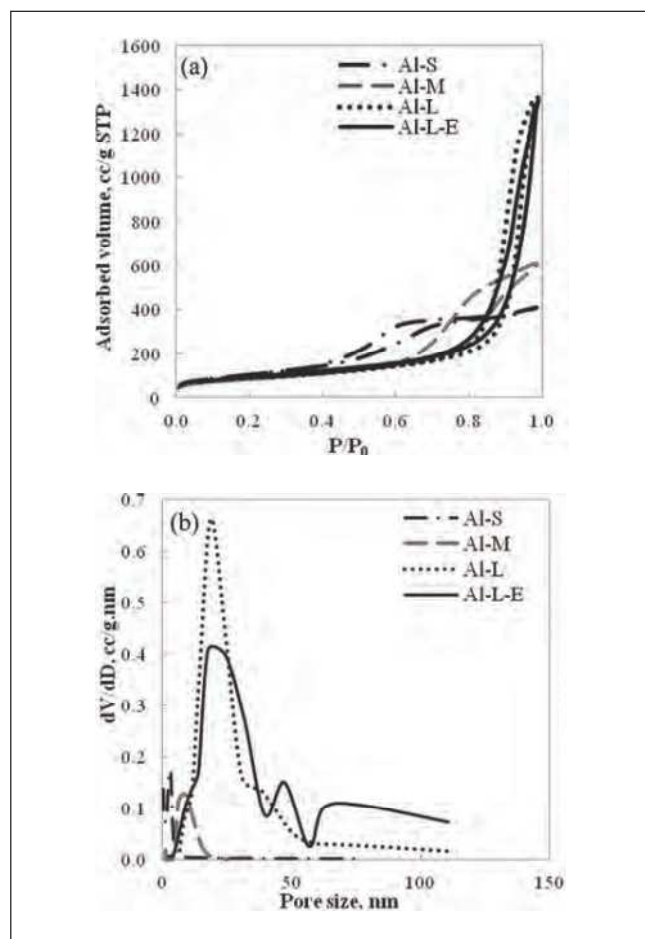

Figure 1: (a) Nitrogen adsorption-desorption isotherms and (b) corresponding BJH pore size distributions for mesoporous alumina

3.2 Acid Capacity of MoO₃/Al₂O₃ Catalyst

NH₃-TPD measurements were conducted in order to investigate the acid capacity of MoO₃/Al₂O₃ catalysts of different textural properties. The NH₃-TPD profile of MoO₃/Al₂O₃ catalysts has been reported earlier [17]. Basically, broad desorption peaks appeared at low temperature range (>600°C) showing weak acid sites [21], as expected for alumina. The total acid capacity of MoO₃/Al₂O₃ catalysts was calculated from the area under the curve of NH₃-TPD. The total acid capacity of MoO₃/Al₂O₃ catalyst increased in the order: MoAl-L-E ≈ MoAl-M<

MoAl-L < MoAl-S. The existence of micropore surface area in catalyst support is believed to enhance the acid capacity of the catalyst support. According to Table 1, MoAl-S with the largest micropore surface area was found to possess the highest total acid capacity (0.038 mmol/g), followed by MoAl-L (0.017 mmol/g). For MoAl-M (0.010 mmol/g) and MoAl-L-E (0.011 mmol/g), both catalysts exhibited smaller micropore surface area and showed lower acid capacity.

3.3 Hydrocracking Reaction

The characteristics of used motor oil and residual oil employed in this work are listed in Table 2. The used motor oil is lighter than the residual oil. It has lower density and lighter in terms of boiling range distribution which contains 43.68 % of residue while the residual oil has 81.47 % of residue. The sulfur content for both feedstocks are similar and lower than 1 wt%. The oxide catalyst after calcination was used directly in the hydrocracking reaction. The metal oxide would most probably react with the sulfur compound in the oil during reaction and be converted to metal sulfide which has different activity. This would not affect the results since direct quantitative comparison is made among the same feedstock and catalyst.

Table 2: Characteristics of used motor oil and residual oil

Properties	Amount	
	Used Motor Oil	Residual Oil
Density[a], g/ml	0.8703	0.8812
Asphaltenes, wt%	7.75	10.22
Sulfur, wt%	0.97	0.47
Distillation analysis, vol %:		
Gasoline (<150°C)	0	0
Kerosene (150-250°C)	9.29	0
Diesel (250-340°C)	47.03	18.53
Residue (>340°C)	43.68	81.47

[a] Measured at room temperature

The performances of mesoporous Mo/Al₂O₃ catalysts of different pore sizes in hydrocracking reaction were investigated. Table 3 shows the hydrocracking results of different feedstocks achieved by mesoporous Mo/Al₂O₃ catalysts of different textural properties. At the reaction temperature of 400°C, not much coke is expected to be formed. Small amount of coke was found in hydrocracking of used motor oil and negligible coke was obtained in hydrocracking of residual oil. In this study, the conversion of used motor oil achieved 62.43-67.86 wt%, whilst the conversion of residual oil is between 43.85-53.25 wt%. This observation is in agreement with the report that coke formation is more serious when the conversion of residues exceeds 60 % [22]. Although higher conversion was obtained in the hydrocracking of used motor oil compared to that in residual oil, more 340−°C residue was actually converted in the hydrocracking reaction of residual oil as more 340+°C residue was present in the feedstock initially compared to used motor oil.

For hydrocracking reactions with used motor oil as feedstock, the lowest conversion (62.43 wt%) and liquid products yield (84.02 wt%) were observed for hydrocracking reaction with 9.0 nm catalyst pore size (MoAl-M). On the other hand, catalyst MoAl-M also gave the highest gas products yield (15.91 wt%). The highest liquid products yield (86.77 wt%) with the lowest

Table 3: Hydrocracking results achieved by MoO_3/Al_2O_3 catalysts

	Catalyst						
	MoAl-S	MoAl-M	MoAl-L	–	MoAl-S	MoAl-M	MoAl-L
	Used Motor Oil			Residual Oil			
Conversion (wt %)	67.86	62.43	67.65	14.08	53.25	43.85	51.22
Products yield (wt %)							
Liquid	86.77	84.02	85.82	98.44	96.62	95.74	97.23
Gas	13.17	15.91	14.15	1.56	3.38	4.26	2.77
Coke	0.06	0.07	0.03	-	-	-	-
Liquid product fractions (vol %)							
Gasoline	17.19	15.80	16.60	-	-	-	-
Kerosene	28.66	27.64	33.20	7.53	13.44[a]	15.27[a]	12.16[a]
Diesel	35.34	32.58	33.20	35.12	47.04	33.60	41.05
Residue	18.81	23.98	17.00	57.35	39.52	51.13	46.79

[a] Gasoline was found in relatively low volume

gas products yield (13.17 wt%) were obtained by using 3.3 nm pore size MoAl-S catalyst in the hydrocracking of used motor oil. Meanwhile, MoAl-S catalyst also generated the highest gasoline yield in the reaction, whereas reaction with 18.9 nm MoAl-L catalyst gave the highest kerosene yield. MoAl-M gave the lowest yield of gasoline, kerosene and diesel. It is clear that the trend of conversions in reaction with residual oil and used motor oil achieved by catalysts with 3.3, 9.0 and 18.9 nm pore size are similar. For reactions with residual oil as feedstock, the highest conversion (53.25 wt%) was achieved by using 3.3 nm MoAl-S catalyst followed with MoAl-L and MoAl-M. However, MoAl-L catalyst with larger pore size, 18.9 nm gave the highest liquid products yield and the lowest gas products yield in the hydrocracking of residual oil. On the other hand, MoAl-M catalyst with 9.0 nm pore size generated the highest kerosene yield (15.27 vol%) in residual oil hydrocracking. The highest diesel yield was observed by using MoAl-S catalyst in residual oil hydrocracking.

The conversions' trend observed in this work is in agreement with the micropore surface area and total acid capacity of catalyst. MoAl-S catalyst with 3.3 nm provided good conversion in both used motor oil and residual oil hydrocracking reactions is most probably due to its largest micropore surface area (38.30 m^2/g) which has more concentration of acid sites over the support surface [23] and contributed the highest total acid capacity (0.038 mmol/g). Therefore, MoAl-S catalyst provides good cracking activity. This is followed by MoAl-L with 28.99 m^2/g micropore surface area and 0.017 mmol/g of total acid capacity, which also gave relatively good conversion and cracking activity. The catalyst with large micropore surface area assists in the increment of acid capacity of catalyst and thus enhances the cracking reaction. Therefore, MoAl-M catalyst gave the lowest conversions for both used motor oil and residual oil because it possessed the smallest micropore surface area (1.9 m^2/g) and the lowest acid capacity (0.010 mmol/g).

The different trend observed in liquid products yield with different feedstocks could be due to the effect of catalyst acid capacity and pore size. In the hydrocracking of used motor oil, the highest liquid products yield was obtained by using the smallest pore size (3.3 nm) with the highest acid capacity (0.038 mmol/g) MoAl-S catalyst with good cracking activity.

Meanwhile, for the hydrocracking of residual oil, the highest liquid yield was obtained by reaction using MoAl-L. Basically, on average smaller hydrocarbon molecules were involved in used motor oil, whereas residual oil mostly involved larger hydrocarbon molecules, thus a catalyst with larger pore size facilitates the accessibility of larger molecules to the catalyst active sites to crack into smaller hydrocarbon molecules. Apart from higher acid capacity, MoAl-L catalyst also exhibits large pore size (18.9 m) and large pore volume (2.12 cm^3/g), hence it can perform efficiently for converting residual oil into lighter oil due to less diffusional restrictions encountered in the catalyst [14; 24].

As presented in Figure 2, reaction with 18.9 nm pore size MoAl-L produced more primary products (gasoline and kerosene) in hydrocracking of used motor oil. It shows that the existence of bigger mesopores seems to promote cracking of diesel into primary products during hydrocracking of used motor oil. MoAl-S was good in promoting production of diesel via cracking of residue in both hydrocracking of used motor oil and residual oil. This could be due to its high acid capacity (0.038 mmol/g) that accelerated the cracking reaction and thus converted the residue fractions in used motor oil and residual oil into lighter molecules (diesel). MoAl-L catalyst gave moderate cracking function with its moderate acid capacity (0.017 mmol/g). The higher production of primary products via secondary cracking (cracking of diesel) in hydrocracking of used motor oil seems to be preferred by using catalyst with larger pore size. When looking at the volume ratio of primary products (gasoline and kerosene) to diesel, the ratio was found to increase from 1.30 to 1.33 and 1.50 for MoAl-S, MoAl-M and MoAl-L respectively showing that pore size range studied has the effects on product distribution of diesel to lighter oil.

However, this phenomena was not observed in hydrocracking of residual oil. Most motor oil are paraffinic base while for the case in residual oil is more complicated with aromatics. More than 80 % of the residual used is 340+°C residue. Cracking of the residue to lighter oil would be the major reactions occurring during hydrocracking reactions. There was only minute production of gasoline in current hydrocracking of residual oil. Temperature is still the controlling factor in cracking of C-C bond in hydrocarbons. Higher temperature is needed for cracking

of 340+°C residue in order to obtain more lighter liquid product. Catalyst acid capacity did play an important role during reaction. The trend of unconverted portion of residue left in the product and the diesel yield followed the catalyst acid capacity. The effect of the catalyst pore size was not observed in the present system of residual oil hydrocracking though. It could be due to the complicated molecular structure in the system as well as the pore size range studied is not suitable for the feedstock.
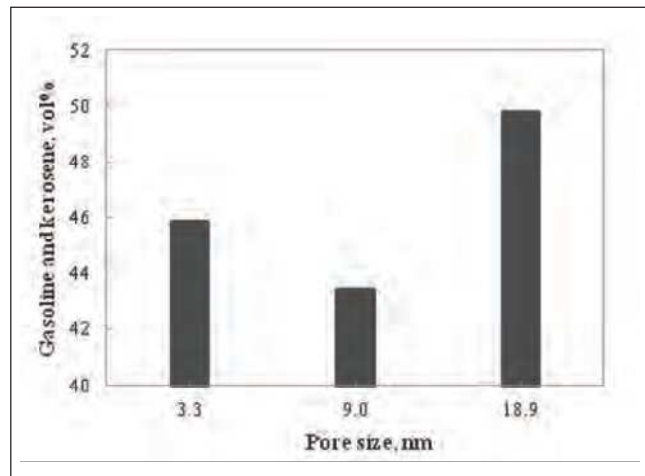

Figure 2: Effect of pore size of catalyst support on gasoline and kerosene yield in hydrocracking of used motor oil

To have a better understanding of effect of acid capacity in the hydrocracking reaction, the performances of two catalysts, i.e. MoAl-L-E and MoAl-L which exhibited similar pore size (~19 nm) but different acid capacity was compared. As shown in Figure 3, MoAl-L catalyst with higher acid capacity showed higher conversion and generated more lighter liquid yield than MoAl-L-E with lower acid capacity achieved. It showed that the lower the acid capacity of catalyst the lower the diesel selectivity.

4.0 CONCLUSION

Mo/Al$_2$O$_3$ catalysts with different support pore size i.e. 3.3, 9.0 and 18.9 nm were prepared and used to study the hydrocracking of used motor oil and residual oil. Each prepared alumina has
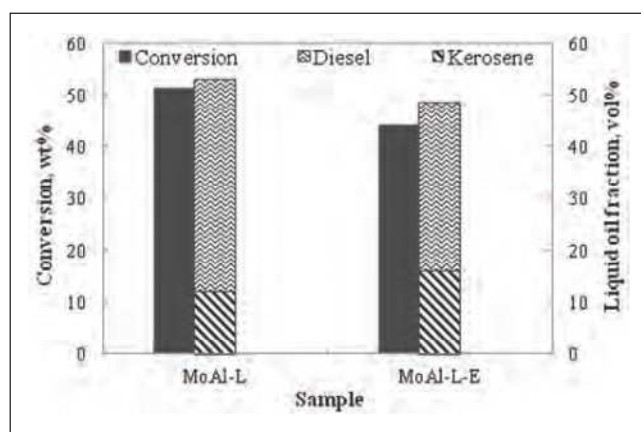

Figure 3: Comparison hydrocracking results between lower and higher acidity but with similar pore size of catalysts

different physical (surface area; pore size; pore volume) and chemical (acid capacity) properties due to the different method used during the alumina preparations. It is difficult to study the effect of catalyst pore size on hydrocracking reactions directly since each catalyst has different properties. Among prepared Mo/Al$_2$O$_3$ catalysts, catalyst with large micropore surface area and high acid capacity exhibited better hydrocracking activity since it showed high conversion in used motor oil and residual oil, as well as produced more liquid products yield in the present study. Basically, the acid capacity of the catalyst is consistent with the conversion activity of catalysts in hydrocracking. Meanwhile, it was also observed that the ratio of gasoline and kerosene to diesel increase with the catalyst pore size. It is believed that suitable catalyst pore size would affect the liquid product distribution. Further study on the effect of pore size in hydrocracking reactions using specific feedstock or model compound may help to elucidate the case.

5.0 ACKNOWLEDGEMENT

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PROFILES

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