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Chemical Feedstock Recycling (4)

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Utilization of red mud as catalyst in conversion of waste oil and waste plastics to fuel

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Abstract The aim of this study was to investigate the possibilities of using a by-product (red mud) from alumina production as a catalyst for recovery of waste. The conversion of waste mineral oil (WMO) and waste mineral oil/municipal waste plastic (WMO/MWP) blends over red mud (RM), a commercial hydrocracking catalyst (silica–alumina), and a commercial hydrotreating catalyst (Ni–Mo/alumina) to fuel has been studied. The effect of the catalyst and the temperature on the product distribution (gas, liquid, and wax) and the properties of liquid products were investigated. In the case of hydrotreatment of WMO, the liquids obtained over RM at both 400° and 425°C had larger amounts of low-boiling hydrocarbons than that of thermal or catalytic treatment with hydrotreating catalyst. Gas chromatography and nuclear magnetic resonance analysis of the liquid products showed that RM had hydrogenation and cracking activity in hydrotreatment of WMO. In coprocessing of WMO with municipal waste plastics, temperature had an important effect as well as the amount of MWP in the blend and the catalyst type. The hydrocracking at 400°C produced no liquid product. In hydrocracking at 425°C, the product distribution varied with catalyst type and MWP amount. The commercial hydrocracking catalyst had more cracking ability in the conversion of WMO/MWP to liquid and gas fuel than RM. In the case of hydrocracking over RM, the largest amount of liquid having satisfactory quality was obtained only from the blend containing 20% MWP.

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Introduction

The disposal of waste such as plastics, tires, oil, and grease is an important environmental problem. At present most of them are disposed in sanitary landfill sites or by incineration. However, both landfilling and incineration are not an environment-friendly solution. These wastes were produced from petroleum and they can be considered as a hydrocarbon source. Therefore, conversion of waste plastics and oils to useful products may be promising as a solution to their growing environmental problem.

Various investigators have dealt with the conversion of plastics to fuels and chemicals. They have focused mainly on thermal degradation, dealing with refinery processes such as pyrolysis, gasification, hydrocracking, and catalytic cracking. In the case of waste oil, there are numerous patents related to re-refining and reprocessing. Different types of distillation processes have been developed for re-refining of waste oils. In reprocessing, waste oils are converted to heavy naphtha and diesel fuel by thermal degradation. However, the thermal cracking of used oils has serious problems with regard to product quality. The oil produced has a strong odor, it precipitates tar, and it darkens over time. It is generally known that thermal degradation produces olefin-rich oil which rapidly changes color and composition due to oxidation and polymerization reactions. Both re-refining and reprocessing need a hydrogenative treatment as a final step.[1,2] Pyrolysis of waste mineral oils has been extensively studied by Moliener and coworkers.[3,4,5] Pyrolysis of lube oil waste yielded important quantities of valuable products such as C_1-C_3 alkanes, C_2-C_4 olefins, and BTX as a function of the pyrolysis conditions.[3,4] However, the liquid pyrolysis products also contained heavy metals which were present in waste oil. Some sorbents have been used as metal traps in pyrolysis of mineral waste oils.[5] These studies showed that the performance of the sorbent depends on the type of metal. In the coprocessing of coal with an automobile crankcase, it was observed that coal acted as a trap for the metals.[6]

Another approach in coprocessing studies is the investigation of waste oils as a coal liquefaction solvent. Utilizing waste oil as coal liquefaction solvent is beneficial to liquefaction chemistry.[7] Orr et al.[6] liquefied different types of coal with different waste oils and they obtained reasonable conversions. Lazaro et al.[8] studied the copyrolysis of a lube oil waste and coal. The copyrolysis increased the quantity and quality (a higher proportion of light olefins and BTX) of the products obtained as compared with the products obtained in coal pyrolysis.

Although there have been numerous studies on coprocessing coal with waste only, a few studies relating to coprocessing waste plastics with waste oil are found in the literature. Coprocessing of waste plastics with waste oils provides another alternative to produce fuels and chemicals from wastes. Both waste oils and common plastics have a paraffinic base, and so a homogenous mixture can be obtained by dissolving waste plastic in waste oil. Yoon et al.[9] suggested that simultaneous processing of mixed waste plastics with waste oil can yield synergistic effects.

In this study, hydrotreating of waste mineral oil (WMO) and hydrocracking of municipal waste plastics (MWP)/WMO mixture was carried out with and without catalyst. Red mud (RM) and commercial catalysts were used in catalytic experiments. RM is a by-product of the alumina manufacture process. It mainly contains the oxides of iron, aluminum, silicon, and titanium. It has been used for the liquefaction of coal[10,11,12] and biomass[13] and in the hydrogenation of anthracene oil[14] as a catalyst. To our knowledge there are no literature reports on the hydrotreating of WMO and only two published reports on polymer degradation in the presence of RM. The goal of this research was the utilization of a waste material as a catalyst for the recovery of other wastes.

Material and methods

Waste mineral oil (WMO) used was provided by a petrol station. This oil was a mixture of waste automobile lubricating oil. MWP were obtained from a local waste manager in Izmir, in the form of fluff. Their compositions are shown in Table 1.

Table 1. Composition of municipal waste plastic (MWP) (wt%)

LDPE	HDPE	PP	PS	PVC	PET	Inert
12	21	56	2	2	1	6

LDPE, low density polyethylene; HDPE, high density polyethylene; PP, polypropylene; PS, polystyrene; PVC, polyvinyl chloride; PET, polyethylene terephthalate

RM was supplied as a sludge by Seydişehir Alumina Plant, Turkey. It was filtered and then dried at 110°C. It was activated as follows: 10 g of RM was added to 190 g of distilled water and stirred to form a slurry, to which 18 g of 31 wt% HCl was added. The resulting solution was boiled for 20 min and diluted with water to a total volume of 800 cm^3. A quantity of ammonia solution was slowly added until a pH of 8 was reached. The resulting precipitate was separated from the solution by filtration, washed three times with distilled water at 40°C, dried at 110°C, and finally calcined in air for 2 h at 500°C. The properties of RM are given in Table 2. The DHC-8 catalyst used is an amorphous hydrocracking catalyst consisting of non-noble hydrogenation metals on a silica–alumina base. It is a bifunctional catalyst incorporating both hydrotreating and hydrocracking functions. This catalyst is currently used for hydrocracking of vacuum gas oil in the Izmir refinery. The hydrotreating catalyst (HC) used is a commercial catalyst. It consists of oxides and sulphides of nickel (1%–10%) and molybdenum (5%–30%) on an alimuna support. This catalyst is currently used for hydrotreating of oil fractions in the Izmir refinery.

Table 2. Content of activated red mud (RM) (wt%)

Fe_2O_3	41.10
Al_2O_3	21.32
SiO_2	17.02
Na_2O	1.25
CaO	2.47
TiO_2	5.47

Hydrotreating and hydrocracking reactions with or without catalysts were carried out by using a 100-ml shaking type batch autoclave with a 25-g feed. Reactions were carried out at 4.0 MPa initial hydrogen pressure at different temperatures for the reaction time of 120 min. In catalytic experiments, 2.5 g of a catalyst was loaded in the reactor. At the end of the reaction time, the autoclave was cooled to room temperature using a fan. The gases were released. Reactor content was centrifuged to separate the liquid products. The remaining product in slurry form was extracted with tetrahydrofuran (THF) to remove wax compounds. The THF-insoluble fraction consisted of coke, catalyst, and undegraded waste plastics. The THF insolubles were extracted with xylene at 130°C to dissolve the undegraded waste plastics. The xylene insolubles consisted of coke formed during the reaction and inorganic matter. Details of the product analysis procedure are available in the literature.[15]

The liquid products from hydrocracking were analyzed by gas chromatography (GC) on a glass capillary column (50 m × 0.32 mm) coated with a stationary phase of cross-linked methyl siloxane. A flame ionization detector was used in analysis. The data obtained from GC was used to evaluate the simulated distillation curves.[16] The liquids were categorized into naphtha (boiling point <172°C), diesel (172°–232°C), and heavy oil (232%–500°C) fractions. The ^{1}H nuclear magnetic resonance (NMR) spectra of liquid products were recorded on a Bruker DPX400, and $CDCl_3$ was used as solvent. Metals in liquid products were analysed by atomic absorption spectroscopy AAS (Perkin Elmer, 2380). Liquid samples were dissolved in xylene (1/10,v/v) and were used in AAS analysis.

Results and discussion

Re-refining waste oil

Most of the waste oil re-refining technologies consist of water- and sludge-removing/ distillation/hydrotreating units or thermal cracking/distillation/hydrotreating units. In all these processes, catalytic hydrogenation is the final step. In this study, we aimed to find a simpler procedure. Without removing water and sludge-forming impurities, thermal and catalytic hydrotreating/hydrocracking were carried out. For this, waste automobile lubricating oil was subjected to heat treatment under a hydrogen atmosphere at temperatures between 350° and 425°C in the absence or presence of catalyst. Figure 1 shows the product distribution from catalytic and noncatalytic treatment. Under noncatalytic treatment, by increasing the temperature from 350° to 400°C, the liquid yield increased from a negligible amount to 80%. However at 425°C, the liquid yield drastically decreased to about 37% whereas the gas yield and the amount of waxy compounds increased sharply. These increased yields of light and heavy products shows that secondary reactions such as overcracking and repolymerization occurred above 400°C.

To accelerate hydrogenation reactions, a commercial hydrotreating catalyst (HC) was used. Although the HC catalyst is a hydrogenation catalyst, it also showed cracking activity. At 350°C, we obtained liquid products in ~59% yield with HC, whereas the thermal process produced waxy compounds instead of liquids at this temperature. In contrast to the thermal process, in the case of the HC catalyst overcracking reactions was not predominant at higher temperature, so there was no considerabe difference in the product yields at the temperatures of 400° and 425°C. The liquid product obtained at 350°C in the presence of HC was bright yellow and had a viscosity of 61.88 cst. Hydrotreatment with both thermal and HC at temperatures higher than 375°C produced naphtha and diesel instead of base lube oil. The simulated distillation curves of liquids obtained at 400° and 425°C are given in Figs. 2 and 3. At 400°C, the

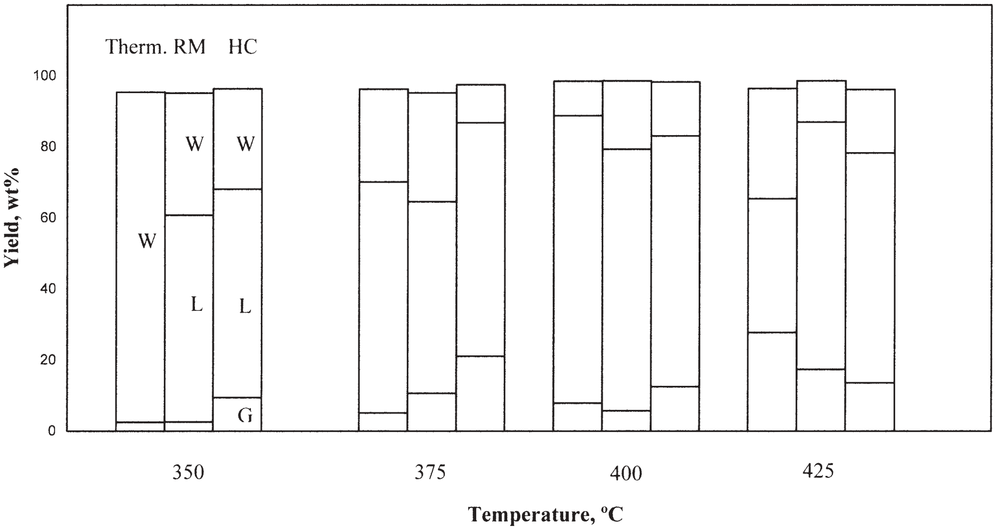

Fig. 1. Product distribution from hydrotreating of waste mineral oil (WMO) at different temperatures. *G*, Gas; *L*, liquid; *W*, wax

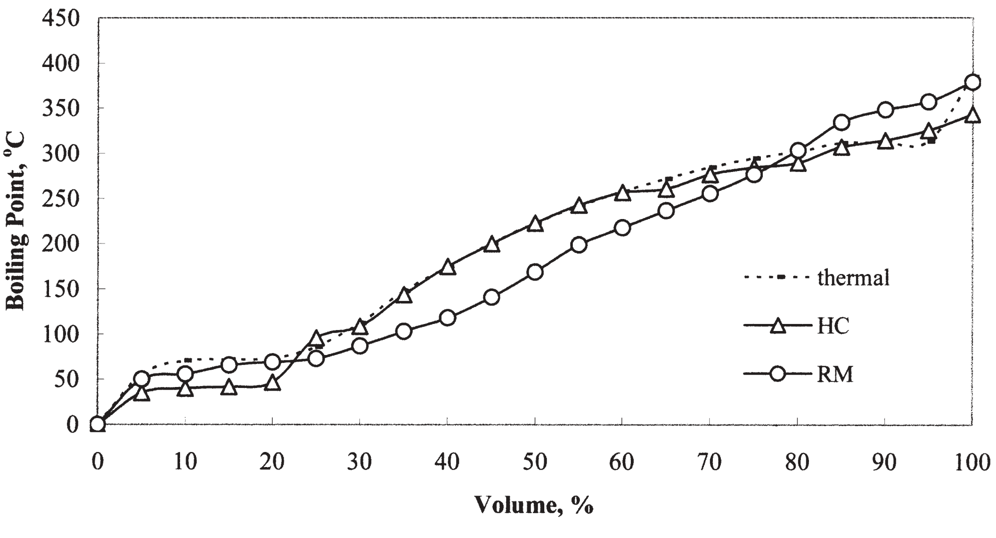

Fig. 2. Simulated distillation curves of liquids obtained by hydrotreating WMO at 400°C

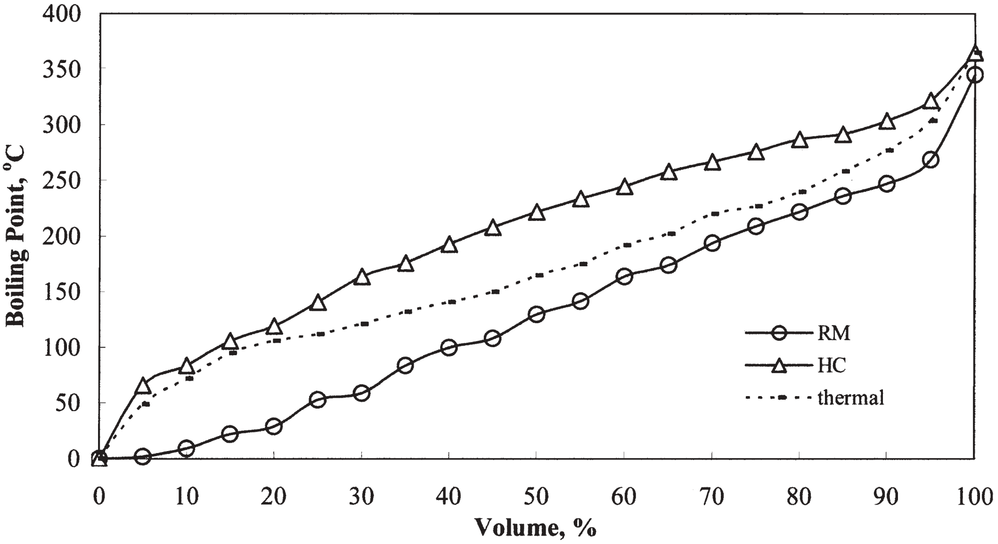

Fig. 3. Simulated distillation curves of liquids obtained by hydrotreating WMO at 425°C

use of the HC catalyst had no effect on the boiling point distribution of the liquid product. The liquid product obtained either in the absence or in the presence of HC consisted of 40% naphtha and 13% diesel. However at 425°C, a thermal run produced larger amounts of lower boiling hydrocarbons than that for a catalytic run due to overcracking. The liquid from the thermal run had a 55% naphtha fraction and a 23% diesel fraction whereas in the case of HC, 35% of the liquid was naphtha.

Because the water and impurities in waste oil are poisonous toward the catalyst, in conventional reprocessing of waste oil, feedstock is not directly subjected to the catalytic process. In this study, the water and impurities would also poison the catalyst, and the catalyst would be deactivated after a hydrotreating time of 1.5 h. We were not interested in the ageing rate of commercial HC catalyst. We used the HC catalyst only for comparison. The main aim of this study was to investigate the use of a disposable catalyst in hydrotreating of waste oil. RM can be considered cheap and disposable or a readily regenerable catalyst. RM showed a good cracking activity in our experiments. In catalytic treatment over RM, the maximum liquid yield (73.64%) was obtained at 400°C. As the temperature increased from 400° to 425°C, the gas yield increased from 5.72% to 17.37%, while the amount of waxy compounds decreased. The presence of RM prevented the repolymerization reactions at 425°C which led to the formation of waxy compounds and coke. The liquids obtained over RM at both 400° and 425°C had larger amounts of lower-boiling hydrocarbons than that of thermal or catalytic treatment with HC. In summary, the hydrotreatment of waste oils over RM at 400° and 425°C produced liquid fuels having 52% and 65% naphtha fractions, respectively.

In addition to boiling point distribution, we determined the chemical characteristics of the liquids. ^{1}H-NMR was employed to provide insight into the structural changes that occurred with variation of the catalyst. From the integration of NMR spectral regions given in Table 3, the volume percent of hydrocarbon types and the H/C atomic ratio were calculated according to correlations developed by Myers et al.[17] The hydrocarbon contents of liquids obtained from catalytic and noncatalytic treatment at 425°C are shown in Table 4. In the presence of RM, the percentage of aromatic and parafinic species in the liquid was similar to that of the thermal run. Using HC produces a liquid having more aromatic compounds. The liquid obtained over RM had no olefinic product. This showed that RM also has hydrogenation activity as well as cracking activity.

Table 3. Nuclear magnetic resonance spectral regions

Proton type	Chemical shift region (ppm)
Ring aromatics	6.6–8.0
Olefinic	4.5–6.0
α-Methyl	2.0–3.0
Methine (paraffins)	1.5–2.0
Methylene (paraffins)	1.0–1.5
Methyl (paraffins)	0.6–1.0

In this study, we also determined metal contents in the liquids, because the WMO contained metallic contaminants. Table 5 shows the amount of selected elements in the liquid product obtained from hydrotreatment of WMO. The results clearly show that the liquids from hydrotreatment have lower concentrations of Pb and Al than that of WMO. It may be concluded that Pb and Al were retained in coke or wax compounds. However, for the other elements it is difficult to reach conclusions. Their distribution in the hydrotreated products should be analyzed in greater detail. It is seen that the hydrotreatment conditions had no considerable effect on the metal content in the liquid. We can, however, mention that the condensation of Pb and Al in the liquid phase was avoided during the hydrotreatment process.

Table 4. The amount of hydrocarbon types in the liquid products from hydrocracking of waste mineral oil (WMO) and WMO/MWP (80:20) at 425°C

	Aromatics	Paraffins	Olefins	H/C	IP
Feed: WMO					
Thermal	3.15	96.24	0.61	2.11	0.57
With HC	6.26	93.74	0.00	2.24	0.92
With RM	3.46	96.54	0.00	2.18	0.80
With DHC-8	6.03	93.97	0.00	2.22	1.49
Feed: WMO/MWP (80/20)					
Thermal	2.86	96.91	0.71	2.09	0.64
With RM	1.01	98.70	0.29	2.04	0.15
With DHC-8	3.22	95.75	1.04	2.12	0.49

H/C, hydrogen/carbon ratio; IP, iso paraffin index

Table 5. Inorganic contents of liquids obtained from hydrotreatment of WMO (ppm) using different catalysts

Reaction temperature (°C)	350	375		400			425			WMO
	HC	–	HC	–	HC	RM	–	HC	RM	
Analyte Pb	10	13	12	18	9	15	22	25	18	118
Fe	13	27	18	19	14	17	20	15	21	23
Cr	1	2	1	3	1	1	2	1	2	2
Si	7	20	10	9	8	11	13	10	13	12
Al	2	2	4	2	2	3	3	2	4	7
Cu	2	5	3	4	3	3	3	3	6	4

Table 6. Product distribution from hydrocracking of WMO and WMO/MWP blends at 425°C

Reaction products (wt%)	No catalyst			DHC-8				RM			
	WMO	50/50[a]	80/20	WMO	80/20	50/50	20/80	WMO	80/20	50/50	20/80
Gas[b]	27.76		14.52	37.19	29.35	30.67	35.64	17.37	24.68	36.35	10.47
Liquid	37.60	wax	54.56	48.25	37.02	33.84	21.68	69.60	45.16	20.40	22.68
Wax	31.08		22.20	14.28	25.68	25.76	32.52	11.65	25.72	33.20	57.64
Coke	3.56		5.48	0.28	1.65	2.85	3.72	1.38	2.04	2.85	3.05
Undegraded polymer[c]			16.21		31.50	13.76	8.05		12.02	14.40	7.70

[a] Ratios of WMO/MWP
[b] Calculated from mass balance
[c] Based on MWP amount

Coprocessing of WMO with waste plastics

A series of thermal and catalytic coprocessing reactions was performed with different mixing ratios of MWP and WMO (WMO:MWP = 80:20, 10:10, and 20:80). The blends were hydrocracked at 400° and 425°C with commercial hydrocracking catalyst DHC-8, RM, and without catalyst.

For all mixing ratios, the hydrocracking at 400°C gave no liquid product even in the presence of catalyst. However, the hydrocracking of WMO alone at 400°C produced a large amount of liquid. It was noted that MWP contained high amounts of polypropylene (PP), 56% and we know from our previous study[18] that PP was easily degraded at 400°C with DHC-8 and without catalyst. This unexpected result shows that the antisynergic effect occurred because of the recombination reactions between the primary degradation products of WMO and waste plastics at a temperature as low as 400°C. When the hydrocracking temperature increased to 425°C we obtained liquid product as well as gas and waxy compounds. The product distributions from the hydrocracking experiments performed at 425°C are given in Table 6. The results of hydrocracking of WMO alone are also given. In the absence of catalyst, the blend containing 50% and 80% of MWP gave no liquid product. However, in hydrocracking of the blend containing 20% MWP, we observed the synergic effect with a larger amount of liquid product and smaller amounts of gas and waxy compounds. It is possible that the radicals formed from MWP degradation prevented the overcracking of WMO by radical quenching. However, when the amount of these radicals was increased, they hindered the degradation of WMO.

In contrast to thermal cracking, in catalytic experiments we observed the antisynergic effect for all mixing ratios. The blends gave less liquid and more waxy compounds than

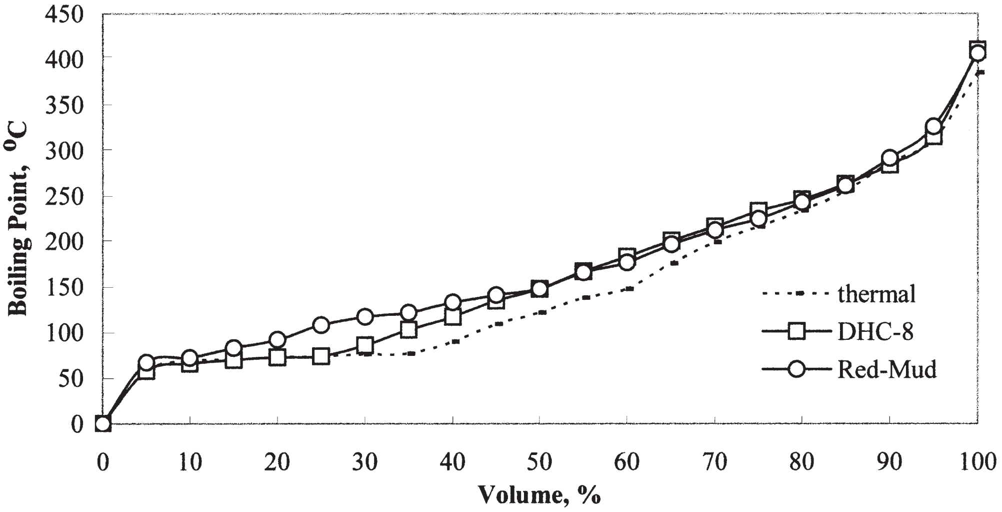

Fig. 4. Simulated distillation curves of liquids obtained from hydrocracking of WMO/municipal waste plastic (80:20) at 425°C

WMO alone. The changes in the product distribution with the amount of MWP in the blend varied with the catalyst type. In the case of DHC-8, for the blends containing 20% and 50% MWP, gas, liquid, and wax yields were almost the same, but when the amount of MWP increased to 80%, the liquid yield decreased, whereas the wax and gas yields increased. However, in the case of RM, increasing the amount of MWP in the blend to 50% increased the gas yield and decreased the liquid yield. When the amount of MWP reached 80%, gas yield dramatically decreased whereas the liquid yield was not considerably changed. Because the waste plastics are the dominant component in the 20/80 blend, it is clearly seen that RM can degrade the waste plastics to mainly waxy compounds. However, DHC-8 led to the formation of gaseous products as well as waxy compounds.

From the above results, it is reasonable to conclude that DHC-8 has more cracking ability in the conversion of MWP/WMO mixtures to liquid and gas fuel than RM. However, RM has the same activity as DHC-8 for the degradation of waste plastics. As shown in Table 6, the percentage of undegraded polymers in the hydrocracking with both DHC-8 and RM were almost the same for blends of 50:50 and 20:80, waste oil:waste plastics.

In this group of experiments, the boiling point ranges of liquid products obtained at 425°C were also determined. The effect of catalyst on the composition of the liquid product varied with the blend composition. In hydrocracking of the blend containing 20% waste plastics, the liquid from thermal hydrocracking had slightly lighter compounds than that from catalytic treatment. It contained 65% naphtha and 15% middle distillate. The boiling point distributions of liquids obtained over both DHC-8 and RM are close to each other (around 60% naphtha and 15% middle distillate). Figure 4 shows the ASTM curves of liquids obtained from the blend containing 20% MWP. When the amount of MWP was more than 20%, we obtained liquids with more heavy compounds. As mentioned above, thermal hydrocracking of a blend containing >20% waste plastic produced no liquid products. In hydrocracking of the 50/50 blend with RM, yields of naphtha and middle distillates in the liquid product were 45% and 20%, respectively. However, in the case of the 80/20 blend, the liquid was more viscous so we did not need to analyze them with GC. In hydrocracking with DHC-8, yields of naphtha and middle distillates in the liquid product were 55% and 16% (for 50/50 blend), and 12% and 50% (for 20/80 blend). These results also support the suggestion that the catalytic activity of DHC-8 was higher than that of RM. However, catalysts used in the hydrocracking of waste materials are subject to rapid deactivation by deposition of inorganic impurities and the formation of coke. Although the coke can be burnt from the catalyst, the inorganic deposits result in an irreversible loss of activity. For this reason, the use of RM has an advantage. This material is sufficiently cheap to be discarded after use. However, in hydrocracking with RM, to obtain large amounts of high quality liquid fuel, the ratio of waste plastics to waste oil must be limited. From our results we believe that the ratio of 80/20 is an ideal blend.

Table 7. Inorganic contents of liquids obtained from hydrocracking of WMO/MWP (80:20) at 425°C (ppm)

	Pb	Fe	Cr	Si	Al	Cu
Thermal	19	24	2	14	2	4
With RM	15	16	1	10	3	5
With DHC-8	14	17	1	10	2	3

In addition to the determination of the boiling point distribution, we determined metal contents and the chemical characteristic of liquids obtained from the 80/20 blend. The amounts of selected elements in the liquid products is given in Table 7. The liquids from hydrotreatment have a lower concentration of Pb and Al than that of raw WMO. By comparing Tables 5 and 7, we could not discuss the effect of the presence of waste plastics on metal contents in liquids. The metal contents in liquids obtained from the hydrocracking of a blend or MWO alone look similar.

The hydrocarbon types in liquid products from the hydrocracking of WMO/MWP (80:20) blend at 425°C are shown in Table 4. Similar to liquids obtained by hydrotreatment of WMO, the liquids from coprocessing contained large quantities of paraffinic hydrocarbons. Hydrocracking of the blend gave a liquid having less aromatics than that of WMO alone (especially for catalytic hydrocracking). In addition, the isoparaffin index of liquids obtained from the blend with catalytic hydrocracking was less than that of WMO alone. In the thermal case, the isoparaffin indexes of liquids from both blends and WMO alone were not considerably different to each other. The differences in the aromatic content and the isoparaffin index of liquids from the blends and MWO shows that waste plastics affected the catalyst activity, and that the commercial catalyst was more affected than RM. Surprisingly, all liquids contained olefin. It is noted that the liquid from RM contained less olefin than that from DHC-8.

Conclusions

The main objectives of this research were to study the application of RM as a catalyst in the conversion of waste oil and waste oil/waste plastic blends to fuel. In the first group of experiments, WMO was hydrotreated at temperatures between 375° and 425°C with and without catalyst. RM and a commercial HC were used:

- The maximum liquid yield (73.64%) was obtained at 400°C over RM and the liquid obtained had a 52% naphtha fraction.
- The liquids obtained over RM contained a higher amount of hydrocarbons with low boiling points than that obtained over HC.
- RM showed good hydrogenation activity as well as cracking activity.

In the second group of experiments, coprocessing of WMO with MWP was carried out with RM and a commercial hydrocracking catalyst (DHC-8) at the temperatures of 400° and 425°C:

- An antisynergic effect was observed. The blends gave less liquid and more waxy compounds than WMO alone.
- The presence of MWP affected the catalyst activity.
- The effect of catalyst varied with the blend composition.
- RM showed good activity for blends only containing 20% MWP.

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