The improved diesel-like fuel from upgraded tire pyrolytic oil

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ADDITIONAL CONSIDERATIONS OF THE PYROLYSIS PROCESS

Temperature. The effect of temperature on pyrolysis is an important factor which researchers focus on. The researchers have determined that the oil yield increased until the optimum pyrolysis temperature, then the oil yield decreased as the temperature increased, but did not change much after a maximum pyrolysis temperature. The amount of uncondensed gas during pyrolysis increases with temperature until the whole of the polymer becomes pyrolyzed. It is difficult to state a specific optimum temperature for the pyrolysis of tires, as the optimum pyrolysis temperature varies depending on the reactor type and other factors such as tire size. According to the researchers, maximum oil yield is reached in the range of 450–500 °C, the amount of oil decreases up to 600 °C, and there is not much change in the amount of oil above 600 °C. In the pyrolysis experiments carried out at temperatures below the optimum temperature, it was observed that the retention and pyrolysis times were prolonged. Coking gas and tar formation increases with repolymerization reactions, the fuel quality of the oil decreases. Although the pyrolytic oil yield at high temperature is lower, the physical properties of the obtained pyrolytic oil are closer to diesel fuel. Therefore, the pyrolysis temperature is controlled depending on whether the pyrolytic oil is to be used as a diesel-like fuel or for heating purposes.

Williams *et al.*[1] determined the maximum pyrolytic oil yield of 55 % at 450 °C between 450–600 °C in a fixed-bed reactor. They found the highest pyrolytic oil yield of 45.1 % at 500 °C in the rotary kiln between 450–600 °C. Dai *et al.*[2] found the maximum pyrolytic oil yield of 52 % at 450 °C in tire pyrolysis experiments between 360–810 °C in the fluidized bed reactor. As can be seen from the studies, the pyrolytic oil yield in the fixed-bed reactor is higher than that of

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the rotary kiln (fast pyrolysis) and fluidized bed reactor. Although pyrolysis is slow in fixed-bed reactors, secondary reactions are less due to the shorter residence time in the vapor phase. Therefore, the pyrolytic oil yield is higher. Polymer phase and vapor phase catalytic reactions, residence time, temperature control is easier in fixed-bed reactors. Consequently, more selective products can be obtained.

Cepic *et al.*[3] in the pyrolysis experiments of tires in the 400–750 °C range, showed that the oil yield increased from 27 to 43.6 % by increasing the temperature from 400 to 500 °C, and decreased to 26.6 % at >500 °C. It was observed that the amount of pyrolytic gas increased regularly from 14.3 to 33.5 % in the range of 400–720 °C. Since there is no remarkable change in the amount of carbon black after 500 °C, it is understood that the pyrolysis of the polymer is completed at 500 °C. Above this temperature, the pyrolytic oil yield first increases and then decreases, and the gas amount continues to increase, indicating that secondary reactions continue in the vapor phase. Alsaleh *et al.*[4] stated that the volatile components increase as the retention time in the gas phase is prolonged at high temperatures, therefore the pyrolytic oil yield decreases. Dai *et al.*[2] obtained the maximum oil yield of 52 % at 450 °C, in their pyrolysis experiments performed in a fixed bed reactor between 360 °C - 810 °C.

Heating rate. The heating rate is another important factor affecting the thermal degradation rate of the polymer. The heating rate also affects the residence time of thermally cracked components in the polymer phase and gas phase, which is one of the most important factors regarding the yield, chemical and physical properties of the pyrolytic oil. In high temperature pyrolysis processes, the residence time in the vapour phase is longer than in the polymer phase and secondary reactions occur in the vapour phase. In low temperature pyrolysis processes, the retention time in the polymer phase is longer than in the vapour phase and secondary reactions occur in the polymer phase. Secondary reactions in the vapour phase break molecules down into smaller molecules. Therefore, the amount of non-condensable gas at high temperature is higher and pyrolytic oil is lower. The longer retention time in the polymer phase causes the conversion of small molecules to large molecules by the repolymerization reaction, and higher is the pyrolytic oil yield at low temperature. Depending on the heating rate, pyrolysis is known as slow and fast pyrolysis. In slow pyrolysis, the pyrolysis temperature, thermal conduction, and thermal decomposition rate is low, the retention time can be up to several hours, and the feed size is relatively large. The best example of slow pyrolysis reactor is fixed-bed reactor. The main product of slow pyrolysis is carbon black. In fast pyrolysis, the thermal conduction rate is high, the residence time is short, and the thermal decomposition rate is high. Examples of fast pyrolysis reactors are rotary reactors and fluidized bed reactors. The main

product of the fast pyrolysis process is pyrolytic oil, which is much more valuable than carbon black.

Many researchers have obtained different results in the pyrolysis experiments depending on the heating rate and pyrolysis temperature, in the amounts of pyrolytic oil and non-condensable gas. Uyumaz *et al.*[5] found the highest pyrolytic oil yield at 450 °C as 53.3 and 42 wt. %, respectively, in a nitrogen atmosphere of 1000 ml min^{-1}, at a heating rate of 10 and 20 °C min^{-1} in a fixed-bed reactor. At the same temperature, the amount of pyrolytic gas was determined as 10 and 20.4 wt. %, respectively. Chouya *et al.*[6] obtained 40 wt. % pyrolytic oil at 550 °C with the highest efficiency in the experiment performed with a heating rate of 20 °C min^{-1} in a nitrogen atmosphere between 300–600 °C. Aziz *et al.*[7] obtained 40 wt. % pyrolytic oil and 11 wt. % gas at 400 °C from the pyrolysis of bus tires at a heating rate of 20 °C min^{-1} in the range of 300–500 °C. Banar *et al.*[8] obtained 38.8 wt. % pyrolytic oil and 27.2 wt. % pyrolytic gas at 400 °C, at a heating rate of 5 °C min^{-1}, in the range of 350–600 °C, from the pyrolysis of wire-removed tires. As it can be seen in the experiments of the researchers, the amount of pyrolytic products obtained at the same heating rate or pyrolysis temperature may vary in different studies. From this, it can be understood that not only the heating rate and pyrolysis temperature are effective on pyrolysis, but also parameters such as tire size, catalyst and reactor type.

Catalyst. Catalysts play a critical role in the thermochemical processing of hydrocarbons in terms of promoting targeted reactions, reducing reaction temperature, and improving process system efficiency. Porous heterogeneous catalysts such as alumina, silica and aluminosilicate are widely used in the catalytic cracking of heavy hydrocarbons because it is more economical and easy to find. Zeolites are the most important catalysts used in petrochemical reactions such as cracking, alkylation, isomerization, oligomerisation, cyclisation, aromatisation, hydrogenation and hydrodesulfurization, dehydroaromatization, conversion of methanol to olefins and hydrocarbons. The pore size and acidity of zeolite-type catalysts are important factors in the catalytic cracking of hydrocarbons. In general, the catalytic activity of zeolites increases with increasing surface area, the number of acidic sites, and acid strength. Catalysts with large surface area and high acid strength increase the rate of degradation of polyolefins. As the acid strength and pore diameter of the catalyst increase, the isomerization rate along with the fuel quality also increases. However, the strong acidity and high pore size cause rapid deactivation of the catalyst. Therefore, slightly acidic and long-lived catalysts are preferred in the pyrolysis of polyolefins. Various studies have shown that the catalysts used during the pyrolysis process have a significant impact on the quality and quantity of pyrolytic products. Chang *et al.*[9] reported the effects of microporous zeolite (ZSM-5), hierarchical zeolite (HZSM-5) and mesoporous silica (MCM-41) catalysts on the

cracking of palm oil. Catalytic cracking of vegetable oil over HZSM-5 was found to produce liquid fuel rich in gasoline fraction. They showed that MCM-41 is more selective for C5+ olefin products compared to the ZSM-5. The selectivity of MCM-41 towards particular liquid hydrocarbons such as gasoline, kerosene or diesel was found to be strongly dependent on the pore size and the surface area of the catalyst. Due to the higher accessibility of reactants in mesoporous MCM-41, as compared to zeolite, it is suitable for catalytic reactions dealing with large molecules. However, the cracking activity of MCM-41 is lower than that of ZSM-5 zeolite due to the lower acidity and shape selectivity of the mesoporous materials. Almeida *et al.*[10] described the pore size effect of catalytic on the cracking of polymers. Catalysts containing strong acid sites, and higher porosity, are more effective in cracking polyolefins. However, the strong acidity and high pore size cause rapid deactivation of the catalyst. They observed that the ZSM-5 catalyst allows the cracking of large molecules due to its three-dimensional pore size. Zhu *et al.*[11] stated that mass transfers of bulk molecules are insufficient in cracking reactions due to the weak hydrothermal stability and acidity of zeolites such as mesoporous MCM-41. They stated that the smaller ZSM-5 crystals increase the rate of dimeric and oligomeric cracking reactions due to their easier diffusion into the linear and branched polymer matrix, thus ZSM-5 catalysts show a good catalytic activity. Eze *et al.*[12] reported that when they used a 1:1 ratio of MCM-41 and ZSM-5 catalyst mixture, the degradation rate of plastics was the highest and a gasoline-like fuel rich in aromatic compounds was obtained. Ayanoglu *et al.*[13] distilled pyrolytic oil using lime and natural zeolite as catalysts and obtained the best results when 10 % lime was used in the experiments. They were obtained 18 % by weight light fraction (such as gasoline), 70 % by weight heavy fraction (such as diesel) and 12 % by weight precipitate phase in experiments. Li *et al.*[13] determined the desulfurization ability of mesoporous molecular sieves (MCM-41), mesoporous aluminosilicate (MAS) and Y-type zeolite (NaY) catalysts by using hydrotreated naphtha diesel and DPT model compounds. The pore diameter of MCM-41 (3.51×10^{-9}) and MAS 3.27×10^{-9}) is larger than NaY (7.9×10^{-10} m). The acidic character of adsorbents is NaY> MAS> MCM-41. For DPT, which is a smaller molecule than hydrotreated diesel, the desulfurization ability of amorphous microporous NaY is higher than that of MAS and MCM-41. However, the desulphurization capacity of NaY is reduced because larger molecules with higher viscosity, such as hydrotreated diesel, block the microporous NaY. In the experiment with MAS, MCM-41 and NaY catalysts, the sulfur concentration of hydrotreated diesel decreased from 207 mg L^{-1} to 93, 98 and 135 mg L^{-1}, respectively. To improve the adsorptive desulfurization properties, stability and acidity of mesoporous molecules, the adsorbent is modified with transition metals. One of the best examples of modification of mesoporous structure is MAS. Li *et al.*[13] found that Cu(I) loaded *MAS* was more

effective than MAS, MCM-41 and NaY zeolite. In experiments with Cu(I)-MAS and MAS adsorbents, 315 mg L^{-1} sulfur value of diesel decreased to 54 mg L^{-1} and 109 mg L^{-1}, respectively. The authors stated that MAS is more effective than MCM-41 in the desulphurization process since it is more acidic than mesoporous MCM-41. Zhang et al.[14] reduced the sulfur value of crude pyrolytic oil and its distillates by 82 % and 84 %, respectively, by oxidative desulphurization using H_2O_2-formic acid as the oxidant and selective adsorption with Al_2O_3.

Fuel blends. The most suitable method to obtain diesel-like fuel is to blend pyrolytic oil and biodiesel obtained from animal or vegetable oils with a high cetane index and flash point. Since the commercial value of other vegetable oils and animal fats is higher than palm oil, biodiesel produced from palm oil is the most economical solution to obtaining a fuel blend. Patel et al.[15] determined that when 5 % ethanol is added to a mixture of 95 % pyrolytic oil and diesel (85 % pyrolytic oil), fuel consumption is reduced, it creates better brake thermal efficiency and can be used as an alternative fuel for diesel engine without any engine modification.

Quality of pyrolytic oils

Pyrolytic oils obtained from tires are not suitable for direct use as a diesel equivalent or diesel like fuel due to their low cetane index, low flash point, high density and high viscosity. The calorific value of pyrolytic oil is the diesel equivalent best feature. During the thermal or catalytic decomposition reactions of tires, physicochemical properties of pyrolytic oils change depending on parameters such as temperature, heating rate, inert carrier gas, and tire size and reactor type. Since aromatic, cyclic, mono- and dimer structures of different sizes are formed during thermal decomposition, pyrolytic oils do not have a certain standard chemical structure.

Physicochemical properties of pyrolytic oil

In the following, important parameters determining the properties of pyrolytic oils, upgraded pyrolytic oils and blended fuels are attempted to be explained separately.

Carbon residue. Carbon residue indicates the tendency of oil to deposit a carbonaceous solid residue on a hot surface, such as a burner or injection nozzle, when its vaporizable constituents evaporate. Fuel which deposits the minimum amount of carbon is preferable. As it can be seen in Tables II–IV, carbon residue was found to be between 0.13 and 0.20 % in UCPO fuels, between 1.30 and 1.98 % in CPO fuels, and 0.7 % in DF. Accordingly, more carbon residues are formed in the injection and combustion nozzles in CPO than in DF, in UCPO there is not as much residue in DF. This indicates that diesel fuels would form higher deposits. Fuels with high carbon residue content could cause increased fouling of

the gas ways; consequently, more frequent cleaning is necessary, especially of the turbocharger and exhaust gas boiler.

pH. As it can be seen in Table V, the pH value of UCPO is almost the same as the known diesel fuel, but very slightly lower. This may be due to components with acidic functional groups formed during the thermal degradation of rubbers. The pH of palm biodiesel and UCPO blends is slightly higher than that of diesel fuels but is not corrosive.

Density. Density is an important factor which determines the combustion quality and other physicochemical properties of the fuel. Since the density of alternative fuel is lower than diesel, mechanical failures occur due to the high pressure formed in the engine as a result of feeding more fuel to the engine, and also more fuel consumption occurs. The impurities such as water, carbon residues, and asphaltenes in the pyrolytic oil are the reasons for the high density. Presumably, the catalyst reduced the conversion of large molecules to smaller molecules and the amount of water. Table II shows the density of the pyrolytic oil obtained from thermal pyrolysis to be between 895.18 and 920.74 kg mol^{-1} in the temperature range of 450–600 °C. As it can be seen in Tables III and IV, in the same temperature range, the densities of the pyrolytic oils obtained from catalytic pyrolysis with 1 and 2 % catalyst were found to be between 832.52 and 856.29 kg mol^{-1} and between 842.11 and 858.71 kg mol^{-1}, respectively. The density values of CPO were significantly reduced in catalytic pyrolysis. In the pyrolysis experiments performed with 2 % catalyst, the density values were found to be slightly higher than 1 % catalyst. From this result, it is understood that as the amount of mesoporous MCM-41 increases, cracking reactions proceed in the direction of the formation of liquid molecules, and gasification decreases. As it can be seen in Table V, the flash point and cetane index of the fuel increased despite the decrease in density in the catalytic experiments. It is understood from the decrease in the density, the cracking of large molecules into smaller molecules causes an increase in the aliphatic structure of the fuel and a decrease in the aromatic structure, and the amount of water mixed into the pyrolytic oil also decreases. At the end of the distillation of the pyrolytic fuel with the packed column, the density value of the fuels obtained as a result of the desulfurization and filtration of the distillates from the bleaching earth is close to the conventional diesel fuels. As seen in Table V, the density value of the fuel obtained by blending up to 60 % of UCPO with PBD was found to be similar to diesel fuel.

Viscosity. Viscosity is one of the most important factors affecting the combustion quality of the fuel. Low viscosity fuels do not lubricate the pistons sufficiently, causing piston wear and leaks. Since high viscosity fuels increase droplet formation during injection, they make combustion difficult, affect fuel

economy, exhaust emissions and harm the environment. The most ideal viscosity range for the known diesel is 2–4 cSt.

The results from Table II show the viscosities of TPO and UTPO in the range of 450 to 600 °C. The viscosity values of TPO are considerably higher (4.36 to 5.97 cSt) than conventional diesel fuel, and the viscosity values decreased as the pyrolysis temperature increased. The high viscosity values of TPO show that it will cause problems even if it is blended with diesel fuels in limited proportions. The viscosity values of UTPO in the range of 450–600 °C are lower (2.84–3.87 cSt) than TPO and are equivalent to conventional diesel fuels. This shows that the upgraded thermal pyrolytic oil can be blended with other diesel-like fuels or diesel fuel in any ratio.

As it can be seen in Tables III and IV, the viscosity values of the upgraded pyrolytic oils obtained from catalytic pyrolysis were significantly improved. As can be seen in Table III, the viscosity values of UCPO obtained in range of 450 to 600 °C using 1 % catalyst were found between 2.18 and 3.42 cSt, and slightly better than the viscosity values of UTPO. These results show that the viscosity values of UCPO are equal to the conventional diesel fuels. As can be seen in Table IV, the viscosity value of the upgraded pyrolytic oils obtained from the pyrolysis experiments performed with 2 % catalyst was found to be slightly higher than the study performed with 1 % catalyst. The viscosities of the upgraded pyrolytic oils were found between 2.57 and 3.57 cSt (Table IV). As can be seen in Table V, fuel blends of UCPO with PBD in all mixing ratios can be used as diesel-like fuel.

From these results, it was understood that the viscosity of the pyrolytic oils decreased as the pyrolysis temperature increased, the viscosity of the pyrolytic oil increased slightly due to the secondary reactions triggering the formation of larger molecules as the amount of catalyst increased, and the viscosity of the pyrolytic oils obtained from the experiments without catalyst was higher. It has been observed that *UCPO* obtained from experiments with 1 % catalyst can be mixed with *PBD* or conventional diesel fuels in any ratio.

Flash point. The flash point of liquid fuel is the temperature at which the fuel reaches the necessary heat for combustion with air. There is a fire hazard in the storage and circulation of fuels with a low flash point. As it can be seen in Tables III and IV, flash points of upgraded pyrolytic oils are lower than the conventional diesel fuels. Although the flash point is expressed as a concept related to the fire hazard of the fuel during storage and transportation, volatile components are high in low flash point fuels. Volatile compounds consisting of small molecules cause a decrease in density and high-pressure during combustion. The flash point of diesel fuels is above 45 °C, both for ignition hazards during storage and transportation and for regular combustion in diesel engines.

The amount of volatile components is high in low flash point fuels. The volatility of the fuel has a significant effect on the delay time. During injection, the fuel droplets come into contact with the heated air in the combustion chamber. During evaporation, the fuel removes energy from the droplet, cooling the environment and increasing the ignition delay time. Isoparaffins and aromatic compounds have stable molecular structures and require higher temperatures and pressures than *n*-paraffins to initiate the oxidation process by forming free radicals. As it can be seen in Tables II–V, pyrolytic oils containing high concentrations of isoparaffin and aromatic compounds have long ignition delay times (low cetane index).

Therefore, using UTPO or UCPO directly as a diesel fuel equivalent will cause problems. For this reason, it is appropriate to mix pyrolytic oils with high flash point and high cetane index fuels such as palm oil biodiesel (PBD), animal oil biodiesel (TOBD) to increase the flash point and *CI*. While mixing with normal diesel only at low ratios forces them to remain within reasonable limits, higher mixing ratios with PBD or TOBD seem possible. As seen in Table V, flash point values of UTPO fuel blended with PBD are close to diesel fuel values.

*Gross calorific value (*GCV). As it can see in Tables III–V, the calorific value of pyrolytic oils is close to the calorific value of known diesel fuels. The calorific value of the fuel mixture obtained as a result of blending with palm oil biodiesel, which has a lower calorific value, increases as the pyrolytic oil content in the mixture increases. In order to obtain a diesel-like fuel, it is considered appropriate to add at least 50 % of UTPO to PBD.

Iodine number. The iodine value gives a measure of the average degree of unsaturation of fuel: the higher the iodine value, the greater the number of C=C double bonds. The iodine value is directly proportional to the degree of unsaturation and inversely proportional to the melting point of the fuel. A lower iodine value is better for a diesel engine as it has a higher carbon content. On the other hand, the high iodine value attenuates the hydrocarbon chain. As seen from Tables II–IV, the iodine value of pyrolytic oils is considerably higher than the iodine value of conventional diesel fuels. This means that pyrolytic oils have high unsaturated bonds, a lower cetane index than diesel fuel, and a lower flash point with a lower calorific value. For this reason, pyrolytic oils cannot be used alone as a diesel equivalent fuel. They may need to be upgraded, blended with other fuels, and added with special additives to improve fuel quality. Values such as moisture, nitrogen, sulphur in upgraded pyrolytic oils are within reasonable limits when compared to the conventional diesel fuel.

Cetane index. The cetane index (*CI*) is an empirical parameter related to the ignition delay time of diesel fuels. The ignition delay is the time interval between the start of fuel injection and the self-start of ignition. The ignition delay time of fuels with high *CI* value is very short. The ignition delay time of diesel cycle

engines is an essential parameter to effectively control the combustion process. Fuels containing high concentrations of *n*-paraffin generally have lower ignition delay times than isoparaffins and aromatic compounds. High viscosity fuels form larger droplet diameters, prevent evaporation and thus cause incomplete combustion, prevent the engine from starting in cold temperatures, and increase the emission of unburned hydrocarbons and particulate matter.. Distillation curves provide information about fuel quality to be correlated with engine performance. The temperature of 10 % of the distillation fractions reflects the ease of evaporation, while the temperature of 90 % of these fractions indicates the presence of high molecular weight compounds that will be difficult to evaporate completely. Therefore, distillation curves provide important information about particulate matter and unburned hydrocarbon emissions and residues in the engine. Fuels with low *CI* can also increase particulate matter emissions. Because combustion, in the last stage of the expansion cycle, causes a temperature drop in the combustion chamber, and the combustion rate decreases. This increases the concentration of unburned hydrocarbons condensed on the surface cause an increase in particulate matter mass. The density at 15 °C and the temperatures at which 10, 50 and 90 vol. % are recovered (distillation recovery temperatures) are determined by standard test methods and the cetane index is calculated from these test data using known correlations. *CI* was calculated empirically according to ISO 4264:2018 method, by substituting the experimental data in:

$$CI = 45.2 + 0.089x \times 2t_{10N} + (0.131 + 0.901B)t_{50N} +$$
$$+ (0.0523 - 0.42B)t_{90N} + 0.00049(t_{10N}^2 - t_{90N}^2) + 107B + 60B^2 \quad \text{(S-1)}$$

where $t_{10N} = t_{10} - 215$; $t_{50N} = t_{50} - 260$; $t_{90N} = t_{90} - 310$; t_{10} is the 10 % distillation recovery temperature, °C; t_{50} is the 50 % distillation recovery temperature, °C; t_{90} is the 90 % distillation recovery temperature, °C; $B = (e^{-0.0035D_N}) - 1$; $D_N = D - 850$, D is the density at 15 °C, kg m^{-3}.

Parameters for calculating *CI* according to the ISO 4264:2018 standard are given in Table S-1. As it can be seen in Table V, the cetane index of the blended fuel increased, and the problems caused by the low cetane index of the pyrolytic fuel were eliminated.

TABLE S-1. Parameters for calculating *CI* according to the ISO 4264:2018

Sample	Density at 15 °C	t_{10} - 215	t_{50} - 260	t_{90} - 310	D_N	*B*
PBD	879	120	82	50	-800	-0.0965
UCPO	852	-114	-40	40	2	-0.0070
UCPO10	876	107	63	49	26	-0.0870
UCPO20	874	3	52	26	24	-0.0806
UCPO30	871	68	56	39	21	-0.0709
UCPO40	868	22	28	46	18	-0.0611
UCPO50	865	31	23	43	15	-0.0511
UCPO60	861	29	30	44	11	-0.0378
DF	863	30	18	20	13	-0.0445

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