Research Article

Excess Molar Viscosities and Excess Molar Gibbs Energies of The Mixtures of Tire Pyrolytic Oil + Diesel Fuel at 293.15 K and 303.15 K

*U. S. Vural

[1]Selcuk University, Faculty of Science, Department of Chemistry (Retired)
E-mail: *usvural@gmail.com

Abstract

Diesel-like fuel mixtures are obtained by blending the pyrolytic oil obtained from the pyrolysis of tires and diesel fuel. The excess thermodynamic properties of blended fuel mixtures give a preliminary idea about the transport, storage and combustion properties of the fuel mixture. In this study, pyrolytic oil and diesel fuel were mixed in different proportions at temperatures of 293.15 K and 303.15 K and their excess molar properties were determined. A positive deviation was observed in the excess molar volume and excess molar Gibbs energy values of the two-component mixture, and a negative deviation was observed in the excess molar viscosity values. Volumetric expansion and flow rate of the mixture were found to be higher at 303.15 K. It has been observed that at low pyrolytic oil concentrations, dispersive and physical forces are dominant between molecules, while at high pyrolytic oil concentrations, π-π interactions are more dominant for the molecules.

Keywords: Excess molar; Gibbs energy; viscosity; molar volume; tire derived fuel; diesel.

1. Introduction

In parallel with the increase in tire production worldwide, the amount of scrap tires is also increasing. Scrap tires are a type of waste that threatens biological life and pollutes the atmosphere as a result of fire [1]. Pyrolysis technique has recently started to gain importance among the methods of reducing scrap tires [2-3]. Thermal cracking of hydrocarbons under oxygen-free conditions is called pyrolysis, and as a result of pyrolysis of tires, 35-50% pyrolytic oil (TPO), 30-40% pyrolytic carbon black (CBp) and 10-15% non-condensed gases are obtained [4,5]. Pyrolytic oil is a type of fuel rich in aromatic compounds, somewhat similar to diesel, with a high calorific value [5]. Pyrolytic oils obtained from the pyrolysis of tires or mixtures of TPO and known fuels, which meet the energy requirements of transportation vehicles and industry, are generally called tire derived fuel (TDF) [6-7]. Remarkable research has been carried out on the use of fuel obtained by blending a certain amount of pyrolytic oil and diesel fuel (DF) in diesel vehicles. Hamzah et al. blended TPO and diesel fuel at a ratio of 10%-50% and tested it in a single-cylinder diesel engine operating at 1200-2400 rpm [6]. They obtained the highest performance values in the 10% TPO mixture, and observed that as the TPO ratio increased, the exhaust gas temperature, engine power and torque, and gas combustion pressure decreased. Mohan et al. purified crude pyrolytic oil with petroleum ether and silica gel and stated that a mixture of 50% diesel, 40% upgraded TPO and 10% ethyl levulinate showed the best performance and emission values in single-cylinder diesel engines [7]. Mia et al. fractionally distilled TPO and subjected it to desulfurization and color removal processes [8]. They found that the fraction between 40°C and 120°C was similar to kerosene, while other fractions differed from diesel fuel.

Vural et al. succeeded in obtaining diesel-like fuel by using advanced purification techniques in catalytic pyrolysis experiments [5]. From these studies, it is understood that crude pyrolytic oil can be mixed with diesel fuels in certain proportions with simple purification techniques.

The excess molecular properties of fuel mixtures are very useful for understanding the conformational stability and unfolding behavior of molecules [9]. Determining the volumetric and some thermodynamic properties of the fuel mixture is important in terms of giving an idea about the transportation and ignition and combustion of the fuel mixture. Excess properties can be defined as the difference between the ideal volumetric properties of the mixture and the observed volumetric properties as a result of the molecular interactions between the components that make up the mixture and in the solution [9,10]. The excess volumes (V^E) are defined as volume of real solutions - volume of pure components (ideal solution). Excess thermodynamic properties, which can also be understood as deviations from ideal thermodynamic behaviors, provide a better understanding of the behavior of components in chemical and physicochemical events, and their roles in production and application processes. In general, the excess properties result from three types of interactions between the constituent molecules of liquid mixtures [9,11,12].

- *Positive effects:* Physical interactions consisting of dispersion forces or weak dipole-dipole interaction,
- *Negative effects:* chemical or specific interactions, including charge transfer, H-bonds, and other complex formation interactions, and
- *Structural effects:* These are the structural contributions arising from the differences in the size and shape of the

component molecules in the mixture depending on the structure of their molecules.

In this study, some excess molar properties of the mixture of upgraded pyrolytic oil (*UTPO*) and diesel fuel (*DF*) were investigated at 293.15 K and 303.15 K. It was observed that unstable, weak physical interactions occurred in the mixture due to the increase in temperature, and no chemical interactions occurred between the molecules.

2. Material and Methods

Scrap tire crumbs, 1-2 cm in size, were obtained from the local recycling company. Diesel fuel was obtained from local Lukoil fuel sales point. Some physicochemical properties of the components are given in Table 1. In previous pyrolysis experiments, researchers stated that thermal cracking reactions were largely completed at 450°C, and that the thermal cracking rate decreased between 450°C and 550°C [13,14]. In this study, asphaltenes, organic residues, carbon and water were removed from the pyrolytic oil by the adsorption method without using advanced purification techniques such as distillation, desulfurization and chemical refining.

2.1. Tire Pyrolysis

300 g of tire crumbs were put into a 1000 mL balloon connected to a condenser and thermometer. It was heated to 450ºC with a jacketed heater. Pyrolytic gases were passed through the condenser and 120 mL of pyrolytic oil was obtained.

2.2. Upgraded Pyrolytic Oil (UTPO)

10 g of acid-active bentonite was added to the pyrolytic oil, stirred at 50ºC for half an hour and filtered. 10 g of silica gel was added, stirred at 50ºC for half an hour and filtered again. Thus, 100 mL of upgraded pyrolytic oil (*UTPO*) was obtained by removing water, carbon residues and polymeric impurities (Figure 1).
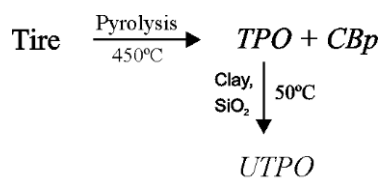
$$Tire \xrightarrow[450ºC]{Pyrolysis} TPO + CBp \xrightarrow[\substack{Clay, \\ SiO_2}]{50ºC} UTPO$$

Figure 1. Tire pyrolysis and upgrade of TPO.

2.3. Fuel Blends

Fuel mixtures were prepared as $v_{UTPO}+(10-v_{UTPO})$ by volume, increasing 1 mL of *UTPO*, making a total of ten pieces of 10 mL each. The sample tubes were mixed with a magnetic stirrer for 5 min at room temperature by closing the mouth with a Teflon cap. Then, the supernatant in the mixtures was separated by filtration. The filtered solutions were kept in a thermostatic water bath at 293.15±0.1 K and 303.15±0.1 K.

2.4. Density and Viscosities of Fuel Blends

The densities of fuel blends were measured by Anton Paar DMA 35N Denismeter. Calibration of the density meter was performed in four replicates with triple distilled water. It was measured as 1.9980 g/cm3 at 20ºC and is compatible with the literature [15]. Kinematic viscosity measurements were made with an Ostwald viscometer. The viscometer was calibrated with toluene. The viscosity of spectroscopically pure toluene was measured as 0.5858 mPa·s at 20°C, in accordance with the literature [16]. Four replicate flow times were measured for each sample and the arithmetic mean was used for calculations. The repeatability of viscosity measurements was 0.005 mPa·s. The samples were placed in the viscometer and the viscometer was immersed in the thermostatic bath kept constant at a temperature of 293.15 K and 308.15 K, respectively. The viscometer was left for 15 min for its content to reach thermal equilibrium. Flow time was measured with a stopwatch to an accuracy of 10^{-2} seconds. Dynamic viscosity values were calculated from kinematic viscosity using measured density values (υ/d).

Table 1. Some physical parameters of DF, UTPO and TPO.

Properties	TPO	UTPO	DF
Density, g/cm^3	0.9123	0.9056	0.8256
Viscosity, mPa·s	4.7842	4.3319	3.1819
Molar mass, g/mol	430[a]	-	280[b]

[a][17], [b][18]

2.5. Calculation of Excess Molar Properties

Excess molar properties of a liquid solution are defined by the following equation [19]:

$$Y^E = Y - \sum_{i=0} x_i Y_i^0 \tag{1}$$

where Y^E is excess molar properties of the solution, *y* is the molar properties of the solution, x_i is the mole fractions of components, and Y_i is the molar properties of pure components, respectively. Eq 1 is written as Eq .2 for excess molar volume (V^E).

$$V^E = \sum_{i=1} x_i M_i \left(\frac{1}{\rho} - \frac{1}{\rho_i}\right) \tag{2}$$

in which M_i is the molar masses of components, ρ is the density of the mixture, and ρ_i is represent the densities of pure components, respectively. The excess molar properties (Y^E) can be correlated using the Redlich–Kister equation [19]:

$$Y^E = x_i(1 - x_i) \sum_{i=0} A_i (2x_i - 1)^i \tag{3}$$

The values of the coefficient A_i were calculated by method of least squares along with the standard deviation $\sigma(Y^E)$. The coefficients A_i are adjustable parameters for a better fit of the excess functions. The standard deviations, $\sigma(Y^E)$, calculated by using Eq. (4):

$$\sigma(Y^E) = \left[\frac{\sum_{i=1}^{i=n}(Y_{cal,i}^E - Y_{exp,i}^E)^2}{n-p}\right]^{1/2} \tag{4}$$

where n is the number of experimental data points, p is the number of parameters, Y_{exp} and Y_{cal} are the experimental and calculated parameters, respectively. In this study, the density and excess molar properties of the mixture of $xUTPO + (1-x)DF$ at 293.15 K and 303.15 K were investigated. The densities of the mixtures over the entire range of compositions at temperatures of 293.15 K and 303.15 K are listed in Table 2. Matlab 2021b was used to calculate the coefficients of polynomial equations and draw graphs.

3. Result and Discussions

Diesel fuel consists of 72% branched alkanes, 10% saturated cycloalkanes, 8-9% alkylbenzenes, 5% alkylated *PAHs* and low amounts of *PAHs*, aromatic acids and

alkanoic acids [20]. Most of diesel fuel has a stable linear alkane structure. *TPO* structure contains 49.54% aliphatic compounds and 16.65% aromatic compounds [21,22]. The largest fraction of aromatic compounds in *TPO* are limonene and monoaromatic compounds. Monoaromatic components are more reactive and mobile than polyaromatic components. Limonene also has aromatic π-electrons and an unsaturated –C=C- bond. Since *UTPO* contains monoaromatic components that make intramolecular bonds with π-electrons, the electronegativity of *DF*, which contains a larger amount of aliphatic and polynuclear aromatic components, is higher than *UTPO*.

The excess molar volumes of the binary mixtures of *xUTPO* + (1-*x*)*DF* were calculated from Eq. (2) over the entire composition range and at T=293.15 K and 303.15 K, and given in Table 2. The density values of the binary mixture increases depending on the increase in *UTPO* concentration. Since both components contain highly stable alkane molecules, there was no significant increase in the volumetric expansion of the mixture with a 10°C increase in temperature, and a slight decrease in density values was observed. As seen in Figure 2, a positive deviation was observed in the V^E values of the binary mixture. The backbone of *DF* has a more stable structure than *UTPO* due to its cycloalkane and alkane structures containing strong C-C bonds. *UTPO*, which contains more π-electrons such as monoaromatic components and limonene, has an electron donor character. Additionally, strong intramolecular π- π bonds (charge-transfer bonds) occur between π-electrons of monoaromatic molecules and limonen in the *UTPO*. As seen in Figure 3, limonene also contains an alkene molecule (-C=C-) in addition to aromatic π-electrons. As seen in Figure 3, electrons on the aromatic structure can be easily dispersed, and π-electrons in parallel, eclipsed or T-shaped edge to face aromatic planes form strong dipole interactions [23]. The reason for the positive deviation in V^E values at low *UTPO* concentrations is the dipole interactions between *UTPO*, which consists of smaller molecules, and *DF*, which has polyaromatic compounds. When *UTPO* concentration increases, due to the pi-electrons localized on *UTPO* molecules, strong intramolecular bonds are formed between the monoaromatic components in *UTPO*. In the *UTPO+DF* mixture, when the *UTPO* concentration is more than 50%, the positive deviation in V^E values decreases because the intramolecular chemical interactions of *UTPO* become stronger than the intermolecular physical interactions between *UTPO* and *DF*. No significant volumetric expansion of the mixture was observed at a temperature increase of 10°C due to the intramolecular bonds of *UTPO*, which are stronger than the dipole interaction between *DF* and *UTPO* molecules. However, it is possible to say that at higher temperatures, volumetric expansion will become more pronounced as intramolecular π-π interactions will weaken. Figure 2 show the excess molar volumes calculated from experimental data and Redlich–Kister equation. Redlich–Kister equation coincided with the experimental results in great agreement.

Dynamic viscosities (η) of the mixtures of *UTPO* and *DF* at 293.15 and 303.15 K were calculated by the measured densities and kinematic viscosities. The viscosity deviation was calculated [19] by Eq. (5) are given in Table 3.

$$\Delta \eta = \eta - \Sigma x_i \eta_I \qquad (5)$$

where η is the viscosities of the mixture and x_i and η_i are the mole fraction and the viscosities of pure components, respectively. The Redlich–Kister equation given in Eq. (3) is written in terms of viscosity as in Eq. (6). The Redlich–Kister coefficients in Eq. (6) were calculated by the least squares method and are given in Table 4.

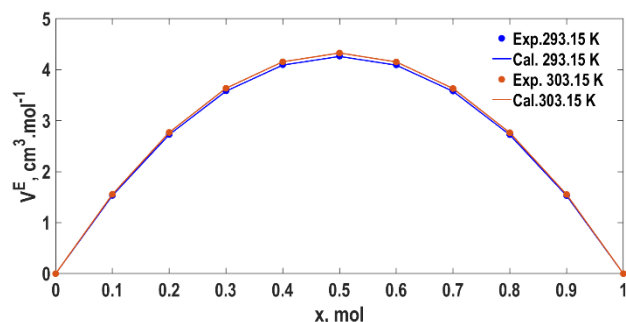$$\eta^E = x_i(1-x_i) \sum_{i=1} A_i (2x_i - 1)^i \qquad (6)$$

Figure 2. The experimental and calculated excess molar volume of the mixture of xUTPO+(1-x)DF at 293.15 K and 303.15 K.
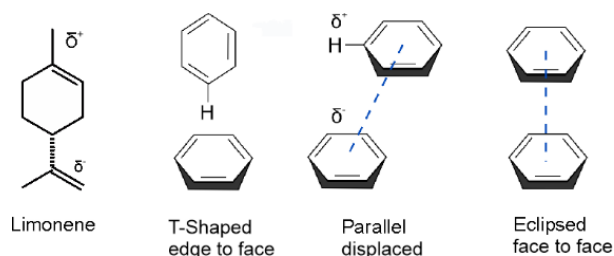

Figure 3. π-electons interactions according to the geometry of the aromatic structure.

Table 2. Densities, viscosities, excess molar volume, of the mixture of xUTPO+(1-x)DF at 293.15 K and 303.15 K.

	ρ, g/cm^3	η, mPa.s	V^E, cm^3/mol	ρ, g/cm^3	η, mPa.s	V^E, cm^3/mol
X	293.15 K			303.15 K		
0.0	0.8256	3.1819	0.0000	0.8189	2.6621	0.0000
0.1	0.8327	3.2699	1.5358	0.8260	2.6961	1.5578
0.2	0.8400	3.3639	2.7319	0.8333	2.7381	2.7710
0.3	0.8475	3.4639	3.5869	0.8408	2.7881	3.6379
0.4	0.8552	3.5699	4.0998	0.8485	2.8461	4.1578
0.5	0.8631	3.6819	4.2701	0.8564	2.9121	4.3302
0.6	0.8712	3.7999	4.0979	0.8645	2.9861	4.1552
0.7	0.8795	3.9239	3.5837	0.8728	3.0681	3.6335
0.8	0.8880	4.0539	2.7283	0.8813	3.1581	2.7660
0.9	0.8967	4.1899	1.5331	0.8900	3.2561	1.5542
1.0	0.9056	4.3319	0.0000	0.8989	3.3621	0.0000

The reason for viscosity deviation depends on two factors [19]. (1) The difference in size and shape of the molecules and the loss of dipolar coupling cause the viscosity to decrease. (2) Specific interactions between components, such as hydrogen bonding and charge-transfer complexes, cause the viscosity of the mixture to increase.

As seen in Figure 4, the viscosity decreases as the *UTPO* concentration increases up to 50%. *UTPO* at low concentrations, consisting of smaller molecules, is easily dispersed in *DF*. As the *UTPO* concentration increases, the dispersion of *UTPO* in diesel becomes difficult and the viscosity begins to increase, since the π-electron density and intramolecular π-π bonds in *UTPO* increase. It has been understood that dispersive and physical forces (dipole interactions) are effective in decreasing the visc"osity of the

mixture up to 50% *UTPO* concentration, and intramolecular charge-transfer bonds (chemical interactions) are effective in increasing the viscosity at higher *UTPO* concentrations. As the temperature increases, the density of localized π-electrons on the aromatic structure in *UTPO* decreases, and the freer *UTPO* molecules are more easily dispersed within the *DF*.

Table 3. The excess molar viscosities and excess molar Gibbs energies of the binary mixture of xUTPO+(1-x)DF at 293.15 K and 303.15 K.

	$\Delta\eta^E$, mPa.s	ΔG^{*E}, J/mol	$\Delta\eta^E$, mPa.s	ΔG^{*E}, J/mol
x	293.15 K		303.15 K	
0.0	0.0000	0.0000	0.0000	0.0000
0.1	-0.0270	0.0061	-0.0360	0.0010
0.2	-0.0480	0.0106	-0.0640	0.0019
0.3	-0.0630	0.0136	-0.0840	0.0026
0.4	-0.0720	0.0152	-0.0960	0.0030
0.5	-0.0750	0.0156	-0.1000	0.0031
0.6	-0.0720	0.0147	-0.0960	0.0029
0.7	-0.0630	0.0127	-0.0840	0.0024
0.8	-0.0480	0.0096	-0.0640	0.0017
0.9	-0.0270	0.0053	-0.0360	0.0009
1.0	0.0000	0.0000	0.0000	0.0000

Table 4. A coefficients and $\sigma(Y^E)$ values in the Redlich and Kister equation.

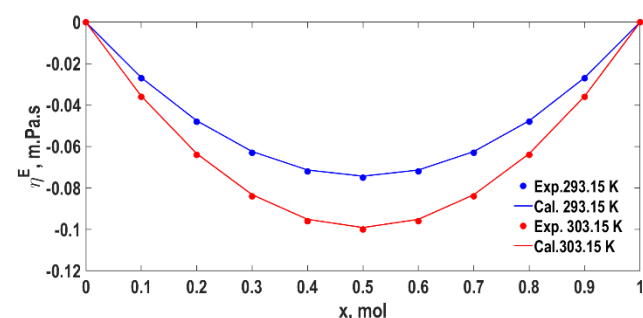| Parameter	V^E, cm³/mol	$\Delta\eta^E$, mPa.s	ΔG^{*E}, J/mol
	293.15 K		
A0	1.71E+01	-0.2971	0.06298
A1	-1.93E-02	-1.28E-09	-5.22E-03
A2	-4.83E-02	2.09E-08	2.65E-03
A3	1.17E-03	8.51E-09	-4.20E-04
$\sigma(Y^E)$	1.90E-02	1.81E-03	5.13E-04
	303.15 K		
A0	1.73E+01	-3.96E-01	0.01282
A1	-2.69E-02	2.32E-08	-1.28E-03
A2	-4.94E-02	7.64E-08	-3.48E-03
A3	2.82E-03	-3.50E-08	8.14E-04
$\sigma(Y^E)$	4.78E-04	2.31E-03	2.09E-04

Figure 4. The experimental and calculated excess molar viscosities of the binary mixture of xUTPO+(1-x)DF at 293.15 K and 303.15 K.

In Figure 4, it is seen that the excess viscosity values calculated from the Redlich–Kister equation are in agreement with the experimental values, and the deviation in the experimental excess viscosities is greater at 293.15 K. Experimental excess viscosities at 303.15 K were found to be lower than the values calculated from the Redlich–Kister equation. Deviations in excess viscosity values calculated from the polynomial equation are probably due to the increase in dispersive forces and dipole interactions at low temperatures and the increase in charge-transfer forces at high temperatures. As a result, a decrease in viscosity values was observed due to volumetric expansion as a result of the increase in intramolecular chemical bonds and the decrease

in dispersive forces and dipole bonds. As seen in Figure 4, as the temperature increase disperses localized π-electrons, π-bonds weaken and a more homogeneous mixture forms, and the flow of the liquid mixture accelerates. The negative values in the viscosity deviations of binary mixtures support weak interactions between *UTPO* and *DF* molecules. It is seen that the viscosity deviation becomes more clearly negative depending on the temperature increase. The reason for this is the acceleration of free flow due to the weakening of intermolecular interaction due to volumetric expansion.

The excess Gibbs energy of the binary mixture of *UTPO* + *DF* is given in Eq. (7) depending on viscosity

$$\frac{\Delta G^{*E}}{RT} = ln\left(\frac{\eta v}{\eta_2 v_2}\right) - x_1 ln\left(\frac{\eta_1 v_1}{\eta_2 v_2}\right) \qquad (7)$$

where v is the molar volume of the mixture, v_1 and v_2 is the molar volume of the pure component, R is the gas constant, T is the absolute temperature, x_1 is mole fraction [19]. The dynamic viscosity of the mixture and pure components are η, η_1 and η_2, respectively. The excess Gibbs energy values of binary mixture at 273.15 K and 303.15 K are given in Table 3. Figure 5 show the excess molar Gibbs energies calculated from experimental data and Redlich–Kister equation.
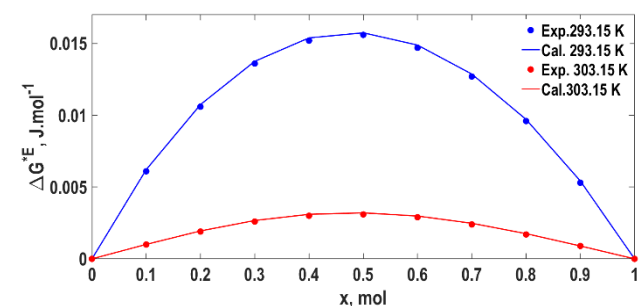

Figure 5. The experimental and calculated excess molar Gibbs energies of the mixture of xUTPO+(1-x)DF at 293.15 K and 303.15 K.

A larger positive deviation was seen in the excess Gibbs energy at 273.15 K. At low temperature, higher activation energy is required for the flow of the mixture due to dispersive and dipole interactions between molecules. At 303.15 K, the activation energy required for the flow of the mixture decreased due to intramolecular charge-transfer in *UTPO*. A positive deviation in the ΔG^{*E} indicates that there is no exothermic interaction in the mixture. As seen in Figure 4, viscous flow is slower at low temperature. Since chemical interactions become more dominant as *UTPO* concentration increases, the positive deviation in ΔG^{*E} decreases at *UTPO* concentrations greater than 50%. ΔG^{*E} obtained from the Redlich and Kister equation and calculated from experimental data are more compatible with each other. A coefficients and standard deviation values in the Redlich and Kister equation are given in Table 4. Redlich–Kister equation coincided with the experimental results in great agreement.

4. Conclusion

The interactions between pyrolytic oil and diesel fuel occur depending on the concentration of both components, molecular structure and temperature. At low *UTPO* concentrations, dispersive forces and physical interactions dominate between *DF* and pyrolytic oil molecules. At high

UTPO concentrations, the disordered π-electrons in the aromatic structure in *UTPO* become localized and intramolecular π-π bonds between aromatic structures become more dominant than physical forces.

The increase in temperature caused a decrease in the viscosity of the binary mixture and reduced the activation energy required for flow. From these data, it was understood that there were no chemical interactions in mixtures of *UTPO* and *DF* in amounts less than 50%, and the volumetric expansion increased slightly with temperature.

Nomenclature

A_i	Redlich–Kister equation Coefficients
M_i	Molar masses of components,
ρ_{ii}	Densities of pure components
ρ	Density of the mixture,
$\sigma(V^E)$	Standard deviation
η	Viscosity of binary mixture,
η_i	Viscosity of component,
$\Delta\eta$	Viscosity deviation of binary mixture
ΔG^{*E}	Excess molar Gibbs energy
v	Molar volume of the solution
v_i	Molar volumes of pure components
V^E	Excess molar volume
exp.	Experimental
cal.	Calculated
x_i	Mole fractions of components
x	Mole fractions of mixture
TPO	Tire pyrolytic oil
UTPO	Upgraded tire pyrolytic oil
DF	Diesel fuel
v_{UTPO}	Pyrolytic oil volume in mixture

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