

CHARACTERIZATION OF THE SPENT TYRE CATALYTIC PYROLYSIS LIQUID PRODUCTS: GASOLINE AND THE REMAINING FRACTION BOILING ABOVE 200°C

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Abstract

Liquid product from pyrolysis of used tyres has been fractionated in naphtha and residual fraction in a rectification column with 15 theoretical trays. Gas chromatographic analysis of the naphtha indicated that it consisted of: normal + iso paraffins = 7.1%; normal + iso olefins = 8.9%; normal + iso di-olefins = 7.0%; cyclic paraffins = 11.7%; cyclic olefins = 18.6%; cyclic di-olefins = 1.4%; aromatics = 35.6%. The naphtha sulphur was 0.48% from which 0.2% was mercaptane sulphur and the remaining was thiophene sulphur. The most abundant individual hydrocarbon in naphtha was found to be limonene (7.65%). The naphtha research octane number was 93.6, and the motor octane number was 80.0. The physical and chemical properties (sp.gr.=0.937, gross calorific value = 41.4 MJ/kg) of the residual fraction indicated that it can be used as a low sulphur fuel oil (0.81% sulphur) or marine oil.

Key words: tyre pyrolysis; tyre pygas octane; residue; liquid characterization.

1. Introduction

Scrap tyres are a growing environmental problem because they are not biodegradable and their components cannot readily be recovered. It is estimated that 2.5 million tones per year are generated in the European Union, 2.5 million tones in North America and around 1 million in Japan [1]. Since tyres are designed to be extremely resistant to physical, chemical, and biological degradation, the possibilities for their reuse and recycling by mechanical or chemical means are very limited currently. Incineration may utilize the energy content of the waste tyres, but it is associated with the generation of SO₂, NO_x and other hazardous emissions. Pyrolysis is an alternative disposal method with the possibility for recovery of valuable products from waste tyres and also attractive environmentally [2-4]. The pyrolysis of used tyres has been widely studied for years [1 - 18]. It has been found that operating temperature, residence time, feedstock size, and feedstock origin (passenger car versus truck tyres) affect the product distribution from pyrolysis of tyres [6,15,19]. Mastral *et al.* [20] observed during tyre pyrolysis and hydrolysis that neither total conversion nor liquid increased with increasing temperature above 500°C. Rodriguez *et al.* [1] did not find significant influence of temperature on the amount and characteristics of pyrolysis products working over 500°C and proved that tyre-pyrolysis liquids are a complex mixture of C₅-C₂₀ organic compounds, with a great proportion of aromatics. After tyre pyrolysis, three phases are obtained: solid, liquid and gas and their composition is related to the temperature of the thermal treatment. The gas product can be used as a make-up heat source for the pyrolysis process [19]. The solid product after demineralization may be used as a suitable adsorbent [9,21]. It also may be used as a solid fuel in coal fired power station because its calorific value (28.9 MJ/kg) is close to that of the coal (33.2 MJ/kg). However, nearly all of the chlorine present in the original tyre ends up in the solid fraction. This should be taken into account when the solid fraction is to be considered as a fuel, since a gascleaning process should be necessary of the combustion gases [18]. The liquid product from the tyre pyrolysis was reported that may be used as fuel oil and diesel fuel [5,10-11, 14]. However,

the value of the liquid product flash point ($\sim 30^{\circ}\text{C}$) is much below the specification for fuel oil and diesel fuel – 60 and 55°C respectively [15]. Therefore, fractionation of the liquid product is required before suitable application of the light and heavy pyrolysis liquid product is to be found. Benallal [4] and Roy [19] reported that the light fraction of pyrolytic oil may be used as gasoline additives in amount of about 2%. However, they did not report its octane rating. Moreover, the current specification of sulphur in gasoline not higher than 10 ppm in the EU and 15 ppm in the US can not allow direct blending of high sulphur pyrolysis naphtha in the commodity gasoline.

A suitable application of the heavy pyrolytic oil can not be found without measuring of its physical and chemical properties and comparing their values with the ones specified in products like fuel oil or marine oil. An application as a diesel fuel may be considered as inappropriate without hydrotreatment and dearomatization because of the higher density, lower cetane and higher sulphur level of the pyrolytic diesel fraction.

The aim of this work is to separate the liquid product from pyrolysis of used tyres into light (gasoline) and heavy fractions, characterize these fractions and determine their suitable commercial application.

2. Experimental

The liquid pyrolytic product was obtained by using proprietary catalytic pyrolysis process [22] at following conditions: reaction temperature of 400°C ; residence time of 50 minutes; reaction pressure of 50 Pa. The process used a proprietary catalyst [23] consisting of 35-50% aluminosilicate; 15-30% active aluminum, 10-20% zinc oxide, 5-15% activated clay; 5-15% kaolin. The catalyst-to-tyre ratio was 5:1000 wt./wt. The yield of products obtained from the pyrolysis process was following: liquid product = 46%, Carbon black = 38%; Steel = 11%; Gas = 5%.

The total liquid pyrolytic product was analysed for its distillation characteristics according to the ASTM D-2887 method (simulation distillation). A fractionation of liquid pyrolytic product was carried out in the AUTODEST 860 Fisher column. that has 15 theoretical trays. The reflux ratio was 10. The liquid pyrolytic product was fractionated in two fractions: gasoline fraction (IBP- 200°C); and heavy pyrolytic oil fraction (200°C – FBP). The distillation characteristics of the gasoline fraction and the remaining fraction boiling above 200°C were determined in accordance with ASTM D-86 method.

The gasoline octane numbers (research octane number = RON, and motor octane number = MON) were measured according to ASTM D-2699 and ASTM D-2700 respectively. The liquid pyrolytic product density was measured according to ASTM D-4052.

Sulphur content in the heavy pyrolytic oil fraction (200°C – FBP) was determined by using a microcoulometric titration method after pyrolysis of the sample according ASTM D 3120. The microcoulometric titration system type MCTS "Dohrmann-130", which includes pyrolysis furnace with automatic temperature control (300 - 1000°C), titration cell with potentiometric determination of the equivalence point, control panel with monitor, and micro-processor.

Aluminium, silicon and vanadium contents in the heavy pyrolytic oil fraction were determined by using the standard methods ASDM D 5184 and ASDM D 5863 respectively. The measurements provided in our laboratory were performed on atomic absorption spectrometer type Varian SpectraAA 220 FS.

The gasoline fraction (IBP- 200°C) was analyzed directly by gas chromatography techniques. To quantify the different compounds, gas chromatography equipped with a flame ionization detector was used. To identify the compounds in the fraction, gas chromatography/mass spectrometry was utilized.

The gas chromatograph with flame ionization detector was a model 5890 series II Hewlett Packard (Agilent Technologies, Inc., USA). A capillary column, HP PONA (50 m length \times 0.20 mm id \times 0.5 μm film thickness), was used and was provided with split injector. The instrument parameters were as follow: initial oven column temperature of 40°C , then increased at increments of $2^{\circ}\text{C min}^{-1}$ to 130°C and second temperature gradient of $5^{\circ}\text{C min}^{-1}$ to 180°C and held for 20 min at 180°C . Helium was used as a carrier gas at a flow rate of 0.5 mL min^{-1} . The injector and the detector temperatures were 250°C and 260°C respectively. The volume that was injected and analyzed was 0.1 μL .

Data acquisition parameters, instrument operation and chromatographic data were collected and recorded by means of Clarity 2.6.

Gas chromatography/mass spectrometry analysis was performed with a 7890A GC System equipped with a HP PONA 50 length m \times 0.2 mm id \times 0.5 μm film thickness, capillary

column and 5975C Inert XL EI/CI mass selective detector (Agilent Technologies, Inc., USA). The oven column temperature conditions identical to those used with the gas chromatograph with flame ionization detector. High purity helium was used as carrier gas at a flow rate of 0.8 mL min⁻¹. The injection port was held at 250 °C and the injection volume of sample 0.1 µL of sample.

The mass selective detector was operated in the electron impact ionization mode (70eV) with continuous scan acquisition from 15 to 250 *m/z* at a cycling rate of approximately 1.5scan/s. The parameters were set up with the electron multiplier at 1224 V, source temperature of 230 °C, and transfer line temperature at 150 °C.

System control and data acquisition was achieved by HP G1033A D.05.01 MSD ChemStation revision E.02.00.493. The compounds were identified by means of the NIST MS Search version 2.0 library of mass spectra.

3. Results and Discussion

Results of the simulation distillation analysis according to ASTM D-2887 and distillation according to ASTM D-86 methods of the liquid product of the waste tyre pyrolysis are summarized in Table 1.

Table 1 Distillation characteristics of the waste tyre pyrolysis liquid product

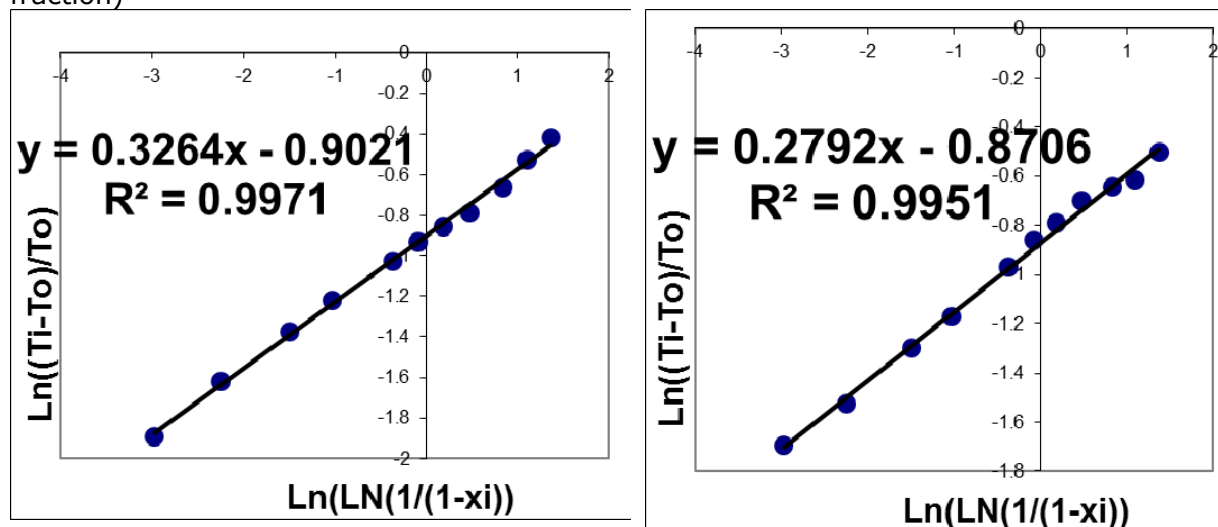
Density at 15°C, kg/m ³	902
Distillation	ASTM D-2887
wt. %	°C
1.2	36
5	80
10	110
20	130
30	145
40	168
50	177
60	203
70	236
80	271
90	335
95	391
98	448
FBP	503
Kw factor	10.34
Flash point, °C	< 40

It is evident from these data that the gasoline fraction (IBP-200°C) presents 59%, the diesel fraction (200 - 360°C) presents 33%, and the remaining residual fraction (360 - 503°C) presents 8% of the total liquid product. The flash point of the liquid product of the waste tyre pyrolysis is below 40°C that makes this product unsuitable for its utilization as a finished oil product. For that reason it was fractionated into gasoline fraction and fraction boiling above 200°C. The distillation characteristics according to ASTM D-86 method of both gasoline and heavy pyrolytic oil fraction are presented in Table 2. It is interesting to note here that similar to other hydrocarbon fractions derived from oil refining [27, 28] the distillation characteristics of the waste tyre pyrolysis gasoline and the fraction boiling above 200°C obey the Riazi's boiling point distribution model (Figure 1). The distillation characteristics of both waste tyre pyrolysis fractions show that these products could be used as gasoline and diesel fuels. In order to better understand the chemistry of the waste tyre pyrolysis gasoline fraction it was analyzed for its hydrocarbon composition and the content sulphur and oxygen species. The applicability as an automotive gasoline was tested by measuring the waste tyre pyrolysis gasoline fraction research and motor octane numbers (RON and MON). The data in Table III summarizes the composition of the pyrolytic gasoline fractions along with its octane ratings. The octane numbers of the pyrolytic gasoline fraction are relatively high: RON = 93.0; MON = 80.

Table 2 Distillation characteristics of the waste tyre pyrolysis liquid product fractions: gasoline and the fraction boiling above 200°C

Waste tyre liquid pyrolytic fraction	Pyrolysis gasoline	Fraction boiling above 200°C
Density at 15°C, kg/m ³	812.7	937
Distillation ASTM D-86 vol.%		
IBP	52	192
5	82	216
10	96	230
20	113	253
30	126	268
50	146	297
60	157	315
70	166	328
80	175	345
90	194	357
95	217	363
FBP	238	390
Recovery, vol.%	98	98
Kw factor	11.20	10.76
Cetane Index (ASTM D-4737)		25

Figure 1 Application of the Riazi's boiling point distribution model to the waste tyre pyrolysis liquid fractions: gasoline and the fraction boiling above 200°C (heavy pyrolysis fraction)



Waste tyre pyrolysis gasoline fraction boiling point distribution of the Riazi's model $(Ti-To)/To = [A_T/B_T \cdot \ln(1/(1-x_i))]^{1/B_T}$ and its linear form: $Y = C_1 + C_2 \cdot X$ where: $Y = \ln((Ti-To)/To)$, $X = \ln(\ln(1/(1-x_i)))$, $B_T = 1/C_2$, and $A_T = B \exp(C_1 B)$

Waste tyre pyrolysis heavy fraction boiling point distribution of the Riazi's model $(Ti-To)/To = [A_T/B_T \cdot \ln(1/(1-x_i))]^{1/B_T}$ and its linear form: $Y = C_1 + C_2 \cdot X$ where: $Y = \ln((Ti-To)/To)$, $X = \ln(\ln(1/(1-x_i)))$, $B_T = 1/C_2$, and $A_T = B \exp(C_1 B)$

However its high sulphur content (2000 ppm) does not allow direct blending of this material in the finished automotive near zero sulphur gasoline (< 10 ppm sulphur). Moreover the relatively high content of highly reactive diolefinic hydrocarbons (8.4%) makes the waste tyre pyrolysis gasoline fraction chemically unstable and prone to form undesirable gums. Therefore this gasoline fraction must be hydrotreated before its use as a component for production of automotive gasoline grades. The selection of the method for hydrotreatment is a subject of another study. Table IV lists the individual hydrocarbons, and sulphur, and oxygen species content in the waste tyre pyrolysis gasoline fraction. These data show that the individual hydrocarbon which has the highest concentration is limonene (7.65%). It has been reported in many studies that limonene is the most abundant hydrocarbon in the waste tyre pyrolysis gasoline fraction [4-6, 8,15,25-26]. It has been also mentioned that

limonene has high value and wide industrial application [25]. However no reports have appeared in the open literature to show that limonene can be efficiently separated from the other hydrocarbons present in the waste tyre pyrolysis gasoline fraction. For that reason we consider this gasoline fraction as a candidate for production of automotive gasoline after hydrotreatment or as a feedstock for the petrochemistry along with the pyrogasoline obtained during naphtha pyrolysis.

Table 3 Group hydrocarbon composition and octane numbers of the waste tyre pyrolysis gasoline

Group hydrocarbon composition	wt.%	Group hydrocarbon composition	wt.%
Normal alkanes	4.09	Oxygen containing species	
Iso-alkanes	2.97	Methylisobutyl ketone	0.7
Cycloalkanes	11.74	Sulphur containing species	%
Normal alkenes	1.14	Mercaptane sulphur	0.2
Iso-alkenes	7.72	2- Methylthiophene	0.3
Cycloalkenes	18.62	Unidentified compounds	10.96
		Octane numbers of waste tyre pyrolysis gasoline	
Normal dialkenes	1.49	RON	93.6
Iso-dialkenes	5.5	MON	80.0
Cyclodialkenes	1.37	Bromine number, gBr2/100 g oil	102.9
Arenes	35.6		

Table 5 presents physical and chemical properties of the waste tyre pyrolysis fraction boiling above 200°C. These data indicate that this fraction meets the specification of the heavy oil products: fuel oil and marine oil. The distillation characteristics of the waste tyre pyrolysis fraction boiling above 200°C as was mentioned earlier correspond to those of diesel fuel. However the low cetane index (25), and high viscosity, as well as high sulphur content do not allow the use of this material as a component for direct blending in the process of production of automotive near zero sulphur diesel (< 10 ppm sulphur). Nevertheless a hydrotreatment of the waste tyre pyrolysis fraction boiling above 200°C with refinery diesel streams may allow the use of this fraction for the production of automotive near zero sulphur diesel. Without any additional treatment the waste tyre pyrolysis fraction boiling above 200°C may be used as finished fuel oil or marine fuel.

Table 5 Measured values of the heavy pyrolytic liquid fraction and specified values for the products: heavy fuel oil and marine fuel oil

	Measured value	Specified value for heavy fuel oil and marine fuel oil
Kinematic viscosity at 50°C, mm ² /s	6.61	max 380
Kinematic viscosity at 50°C, mm ² /s	3.165	max 115
Relative viscosity, °E	1.24	max 15
Sulphur content, wt.%	0.81	max 3.0
Pour point, °C	-18	max 30
Freezing point, °C	-20	max 25
Ash	0.015	max 0.15
Flash point in closed cup, °C	77	min 60
Flash point in opened cup, °C	92	min 110
Water, vol.%	0.1	max 0.5
Lower heat content, MJ/kg	41.363	min 39.8
Water soluble acids and basis	absent	absent
Water and sediments, vol.%	0.4	max 1.0
Al, ppm	8	Al+Si = max 80
Si, ppm	11	
V, ppm	13	max 600

4. Conclusions

The catalytic pyrolysis of waste tyres produces about 50% liquid oil product. This product can not be used as a finished marketable product because of its low flash point value. A fractionation of the liquid pyrolytic product into gasoline fraction (IBP-200⁰C) and a fraction boiling above 200⁰C allows the heavier liquid pyrolytic product to meet the specification of the heavy oil products fuel oil and marine fuel. A hydrotreatment (selective or full) of the waste tyre pyrolysis gasoline fraction may allow the use of this fraction as a component for production of near zero sulphur automotive gasoline. The hydrotreatment of the heavy liquid waste tyre pyrolysis fraction along with the refinery diesel streams may allow the use it as component for production of near zero sulphur automotive diesel fuel.

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Appendix

Table 4 Waste Tyre Pyrolysis gasoline individual hydrocarbon composition

Aliphatic hydrocarbons	%		
Iso-butane	0.05	1,2,3-Trimethylcyclopentene	1.15
Butene-1+isobutene	0.9	2-Ethyl-3-Methylcyclopentene	0.22
Total C4	0.95	3-Ethyl-Hexene	0.69
C5		1,3-dimethylcyclohexane	0.22
3-Methylbutene-1	0.1	1-Ethyl-5-Methylcyclopentene	0.07
Iso-pentane	0.21	3-Ethyl-1,4-Hexadiene	0.32
Pentene-1	0.13	1,5 Dimethyl-Bicyclo[3.1.0]hexane	0.86
2-Methyl Butene-1	0.47	3,5-Dimethylcyclohexene	0.54
n-Pentane	0.32	4-Ethenylcyclohexene	0.42
1,3 Pentadiene	0.07	propylcyclopentane	0.56
1,2-dimethylcyclopropane	1.89	1-Ethyl-5-Methylcyclopentene	0.49
Cyclopentene	0.25	2,3-Dimethylhexadiene-1,3	0.35
Cyclopentane	0.2	Total C8	8.43
Total C5	3.64	C9	
C6		2,2,4,4-tetramethylpentane	0.79
4-Methylpentene-1	0.26	2,6-Dimethylheptene	0.64
1,2,3-Trimethylcyclopropane	0.52	3-Isopropylcyclohexene	0.71
3-methylpentane	0.17	3-Ethyl-2-Methylhexadiene	1.06
2-methylpentene-1	0.43	Ethylmethylcyclohexane	0.41
n-Hexane	0.49	2,6-Dimethylheptadiene	0.48
Hexene-2	0.11	n-Nonane	1.16
2-methylpentene-2	0.56	2-[1-Methyl-2-propynyl]bicyclohexane	0.48
3-methylpentene-2	1.31	Isopropylidenecyclohexane	0.52
methylcyclopentane	0.36	Dimethylheptadiene	0.73
trans+cis 2,4-hexadiene	0.44	Tetramethylpentene-2	0.95
1-Methylcyclopentene	0.93	Total C9	7.93
Cyclohexane	0.15	C10	
Cyclohexene	0.34	1-Isopropyl-4,5-Dimethylcyclopentene	0.37
1,1,2-Trimethylenecyclopropane	0.75	Isopropylidene-methylcyclohexene	1.1
1-Methyl-1,3-Cyclopentadiene	0.17	Isopropyl-Dimethylcyclopentene	0.49
Total C6	6.99	Di-methyloctadienes	1.9
C7		1,4,6,6-Tetramethylcyclohexene	0.55
2,4-dimethylpentane	0.49	4-Isopropyl-1-methylcyclohexene	1.52
5-Methyl-1,3 cyclopentadiene	0.38	4-Isopropenyl-1-methylcyclohexene (Limonene)	7.65
2,4 Dimethylpentene-2	0.46	1-Methyl-4-isoprylhexadiene	0.17
4-Methylhexene-1	0.28	Total C10	13.75
4-Methylhexene-2	0.33	C11	
3-Methylhexane	0.36	Undecandiene	0.98
1,2 Dimethylcyclopentane	0.5	C12	
trans+cis 3-Methylhexene-3	0.32	n-dodecane	0.68
4,4- Dimethylcyclopentene	0.51	Total aliphatics	54.32
n-Heptane	0.76	Aromatic hydrocarbons	%
trans+cis 3-Methylhexene-2	0.35	Benzene	0.26
2,4 Dimethyl-1,3 Pentadiene	1.29	Toluene	4.38
1-Methyl-2-Methylenecyclopentane	0.2	Ethylbenzene	3.41
3-Ethylcyclopentene	0.09	m+p Xylenes	4.67
Methylcyclohexane	0.17	Styrene	1.03
Ethylcyclopentane	0.26	O-Xylene	0.93
1,2-Dimethyl-1,3-Cylopentadiene	0.17	Isopropylbenzene	1.73
1-Methylethylidencyclobutane	2.5	n-Propylbenzene	1.53
1-Ethylcyclopentene	0.28	Ethylmethylbenzenes	3.8
1-Methyl-1,4 Cyclohexadiene	0.23	Trimethylbenzenes	3.2
3-Methyl-1,3,5-hexatriene	0.28	Methyl-isopropylbenzene	4.99
1-Methylcyclohexene	0.76	Methyl-propylbenzene	0.22
Total C7	10.97	Butylbenzene+Methylpropylbenzenes	1.52
C8		Dimethylethylbenzene	0.23
2,2,3,3-Tetramethylbutane	0.36	1-Ethynil-3-Ethylbenzene	0.4
3,4,4-Trimethylpentene-2	0.33	1,2-Di-methylpropylbenzene	0.18
1,2,3-Trimethylcyclopentene	0.18	Sec-Butylbenzene	0.38
1,1,2-Trimethylcyclopentane	0.27	3,4-Di-methylstyrene	0.85
2-Methylheptane	0.43	2,4,6-Trimethylstyrene	0.27
4-Methylheptane	0.11	Durol	0.44
1,3-dimethylcyclohexane	0.37	1-methylindane	0.45
2-Methylheptene-1	0.24	Dimethylindanes	0.51
1-Ethyl-2-Methylcyclopentane	0.25	Total aromatic hydrocarbons	35.38