COMPOSITION OF THE OIL FROM WASTE TIRES 2. Fraction boiling at 160–180 °C

Anne ORAV, Tiiu KAILAS, Mati MÜÜRISEPP, and Jüri KANN

Institute of Chemistry, Tallinn Technical University, Akadeemia tee 15, 12618 Tallinn, Estonia

Received 5 October 1998, in revised form 4 November 1998

Abstract. The qualitative and quantitative composition of the oil fraction from waste tires boiling at 160–180 °C were determined with a capillary GC with OV-101 and SW 10 columns and GC/MS. A total of 97 components representing 92% of the total fraction were identified by means of retention indices and mass spectra. The major contribution of this waste tire oil fraction came from aromatic hydrocarbons, which constituted 48% of the oil composition. The main components of the fraction were dipentene (35%) and *p*-cymene (10%). From the other identified compounds alkanes, alkenes, alkadienes, and cycloalkadienes made up 7% of the total oil fraction and sulphyric compounds (alkylthiophene, benzothiazole) only 0.9%.

Key words: waste tire oil, fraction boiling at 160-180 °C, composition, GC, GC/MS.

Oil was obtained from waste tires with laboratory equipment. The oil obtained was rectified on the APH-2 apparatus. The fraction boiling at 160–180°C constituted 10.2% of the total oil [1].

In order to find possible applications of this product its qualitative and quantitative composition was studied using capillary GC and GC/MS techniques.

EXPERIMENTAL

The gas chromatographic and mass-spectrometric analyses of waste tire oil fraction boiling at 160–180 °C were performed using the same equipment as reported for the fraction boiling at up to 160 °C [2]. The column temperature on the OV-101 column was programmed from 50 °C to 160 °C at 2 °/min and on SW 10 column from 70 °C to 170 °C at 2 °/min. The injector temperature was about 200 °C.

The constituents of the fraction were identified by using our retention index data bank, GC/MS, and literature data [2–4]. The concentration of the individual compounds was expressed as their percentage in the total GC peak area. The results presented are mean values of three injections.

RESULTS AND DISCUSSION

The identified components (76 peaks in the chromatogram) and their concentrations in the waste tire oil fraction boiling at 160–180°C are listed in Table 1.

Peak number	Component	RI on OV-101	Concentration, %	Identification
1.	Isoprene	< 500	0.10	GC. MS
2.	2-Pentene	< 500	0.09	GC. MS
3.	1.3-Cyclopentadiene	590	0.11	GC. MS
4.	2.5-Dimethylfuran	700	0.05	MS
5.	1,4-Cyclohexadiene	738	0.07	GC, MS
6.	Toluene	753	0.10	GC
7.	1,3,5-Cycloheptatriene	758	0.29	GC, MS
8.	2,5-Dimethyl-2,4-hexadiene	821	0.17	GC, MS
9.	2,6-Dimethylheptane	828	0.12	GC, MS
10.	2,3-Dimethyl-1-heptene	842	0.07	GC
11.00	Ethylbenzene	851	0.88	GC
12.	1,3-Dimethylbenzene	860	1.47	GC, MS
	1,4-Dimethylbenzene			GC, MS
13.	Styrene	875	0.11	GC
14.	Vinylbenzene	877	0.23	GC
15.	1,2-Dimethylbenzene	881	0.69	GC, MS
16.	1-Nonene	886	0.14	GC
17.	Nonane	900	0.17	GC
18.	trans-2-Nonene	906	0.13	GC
19.	Isopropylbenzene	912	1.47	GC, MS
20.	2,2-Dimethyloctane	921	0.37	GC
21.	1,5-Cyclooctadiene	928	0.32	GC, MS
22.	Allylbenzene	934	0.19	GC
23.	n-Propylbenzene	943	0.98	GC, MS
24.	1-Methyl-3-ethylbenzene	952	0.51	GC
	Dimethylhexadiene		0.72	MS
25.	1-Methyl-4-ethylbenzene	954	3.28	GC
	Hexahydroindene		0.55	MS
26.	1,3,5-Trimethylbenzene	958	0.75	GC, MS
27.	1-Methyl-2-ethylbenzene	967	1.65	GC
	α-Pinene		0.55	GC, MS
28.	Alkylthiophene	976	0.58	MS
29.	1,2,4-Trimethylbenzene	981	0.42	GC, MS
	2- and 3-Methylstyrene		1.63	GC

Table 1. Identification data and composition of the waste tire oil fraction boiling at 160-180 °C

Table	1	continued	
I duic		commucu	

Peak number	Component	RI on OV-101	Concentration, %	Identification
30.	terts-Butylbenzene	984	0.73	GC
31.	1-Decene	988	0.83	GC
32.	sec-Butylbenzene	996	1.35	GC
33.	1.2.3-Trimethylbenzene	1005	1.22	GC, MS
34.	1-Methyl-3-isopropylbenzene	1008	1.08	GC, MS
35.	1-Methyl-4-isopropylbenzene	1013	9.65	GC
36.	2.3-Dihydroindene	1017	2.79	GC
37	Dipentene	1025	34.69	GC. MS
511	Indene	1020	5 1107	00,110
38	1-Methyl 2-isopropylbenzene	1029	0.45	GC. MS
50.	Butylcyclohexane	1025	0.15	00, 110
30	Not identified	1033	0.24	
10	1.3-Diethylbenzene	1035	0.24	GC
40.	1,5-DictilyIDenzene	1037	0.27	GC MS
41.	1 4 Disthylbanzana	1039	0.55	CC, MIS
42.	n,4-Dietityibenzene	1045	0.00	CC
12	n-ButyIdenzene	1045	0.48	GC MS
43.	1-Metnyi-2-propyibenzene	1054	0.47	GC, MS
44.	1,3-Dimethyl-4-ethylbenzene	1064	0.29	GC
45.	1,4-Dimethyl-2-ethylbenzene	1066	0.48	GC
000000	trans-Decahydronaphthalene		ane de la regeneratione	GC
46.	1,2-Dimethyl-4-ethylbenzene	1069	0.88	GC
47.	1,2-Dimethyl-2-ethylbenzene	1072	0.95	GC
	2-Methyl-propenylbenzene			MS
48.	1,3-Dimethyl-2-ethylbenzene	1074	1.05	GC
	trans-Decalin	1075	0.92	GC
49.	Terpinolene	1070	1.09	GC, MS
50.	1,2-Dimethyl-3-ethylbenzene	1088	0.52	GC
	1-Undecene			MS
	terts-Pentylbenzene		0.76	GC, MS
51.	Diene (MW=152)	1095	1.02	MS
52.	Undecane	1100	0.36	GC, MS
	1,2,4,5-Tetramethylbenzene			GC, MS
53.	1,2,3,5-Tetramethylbenzene	1103	1.04	GC, MS
	cis-Decahydronaphthalene			GC
54.	Isopentylbenzene	1113	0.34	GC
55.	Methylindane	1116	0.31	GC. MS
200	Tetralin		0.30	MS
56	1.2-Dimethyl-4-isopropylbenzene	1120	0.60	GC
50.	Phenyl hutene	1122	0.63	MS
57	1 2-Dimethylindane	1130	1 38	GC
51.	Phenyl hutene	1150	1.50	MS
58	1 2 3 4 Tetrahydronanhthalene	1136	0.40	CC MS
50	1 4-Diethyl-2-methylbenzene	1144	0.33	GC, MIS
59.	n Dentylbenzene	1144	0.55	GC MS
	1.2.3.4 Tetromethylbonzone			GC, MS
60	1.4. Dimethyl 2. promite	1140	0.21	CCM
61	1.2 Diothyl 4 mothylbergene	1148	0.51	GC, MS
01.	1 Mathul 2 hutulhannen	1153	0.12	GC
(0)	1-ivietnyi-2-outyibenzene	1150	0.00	GC
02.	Naphthalene	1158	0.32	GC, MS

Table 1 continued

Peak number	Component	RI on OV-101	Concentration, %	Identification
63.	1,3-Dimethyl-2-propylbenzene	1170	0.56	GC
64.	1,2-Dimethyl-3-propylbenzene	1178	0.46	GC
65.	1-Dodecene	1189	0.20	GC, MS
66.	1,2,3-Trimethyl-5-ethylbenzene	1194	0.17	GC
67.	Benzothiazole	1200	0.30	GC, MS
	<i>n</i> -Dodecane	1200	0.26	GC
68.	Phenylcyclopentane	1212	0.19	GC, MS
69.	1,3-Dimethyl-5-butylbenzene	1230	0.09	GC
70.	1,2,4-Trimethyl-5-isopropylbenzene	1234	0.20	GC
71.	Methyltetralin	1238	0.13	GC, MS
72.	1,3,5-Trimethyl-2-isopropylbenzene	1244	0.16	GC
73.	1,2,3,4-Tetrahydro,5-methylnaphthalene	1261	0.19	GC, MS
74.	Pentamethylbenzene	1269	0.18	GC
	2-Methylnaphthalene			GC
75.	1,2,5-Trimethyl-3-propylbenzene	1276	0.24	GC
76.	1-Methylnaphthalene	1282	0.13	GC
77.	Phenylcyclohexane	1289	0.17	GC, MS
78.	Not identified	1348	0.12	telasted. The
	Total		02.05	

Total

Table 2. Group content of the waste tire oil fraction boiling at 160–180 °C

Group of components .	Concentration, %
<i>n</i> -Alkanes	0.79
Isoalkenes	0.49
Alkenes	2.22
Alkadienes	2.01
Cyclic hydrocarbons	1.34
Monoterpenes	36.33
Aromatic hydrocarbons	48.12
Sulphyric compounds	0.88
Oxygen compounds	0.05
Total	92.23

Like in the fraction boiling at up to 160° C aromatic hydrocarbons dominated also in this fraction (Table 2). The total content of aromatic hydrocarbons (48%) was higher than in the fraction boiling at lower temperatures (27%) [2]. The major individual components in the fraction boiling at $160-180^{\circ}$ C were dipentene (35%) and *p*-cymene (10%), in the fraction boiling at up to 160° C these components made up 12% and 3%, respectively [2]. Other components (alkanes, alkenes, alkadienes, and cycloalkadienes) constituted about 7% of the

total fraction. Alkyl thiophene and benzothiazole were identified in minor amounts (0.9%) as sulphur consisting compounds. Owing to a high concentration of aromatic hydrocarbons in this tire waste oil fraction the results for main constituents determined on two columns with different polarity coincided well.

Dipentene (limonene), the main component of the waste tire oil fraction boiling at 160–180 °C, is widely used for its aromatic value as flavouring in foods and beverages and as fragrance in cosmetic and industrial products. It could be used as raw material for synthesizing other flavouring compounds.

REFERENCES

- Kann, J., Marguste, M., Orav, A. & Kriis, J. Pyrolysis of rubber waste. Proc. Estonian Acad. Sci. Chem., 1999, 48, 1, 40–43.
- Orav, A., Kailas, T., Müürisepp, M. & Kann, J. Composition of the oil from waste tires.
 1. Fraction boiling at up to 160 °C. *Proc. Estonian Acad. Sci. Chem.*, 1999, 48, 1, 30–39.
- Matisova, E., Kovacicova, E., Pham Thi Ha, Kolek, E. & Engewald, W. Identification of alkylbenzenes up to C₁₂ by capillary gas chromatography and gas chromatography-mass spectrometry. II. Retention indices on OV-101 columns and retention molecular structure correlations. J. Chromatogr., 1989, 475, 113–123.
- 4. Bredael, P. Retention indices of hydrocarbons on SE-30. J. High Resolut. Chromatogr. Chromatogr. Commun., 1982, 5, 325-328.

SÕIDUAUTO KASUTATUD RADIAALKUMMI ÕLI KOOSTIS 2. Fraktsioon keemispiiriga 160–180 °C

Anne ORAV, Tiiu KAILAS, Mati MÜÜRISEPP ja Jüri KANN

Kapillaargaasikromatograafia ja massispektromeetria meetoditega on uuritud sõiduauto kasutatud radiaalkummi termilisel lagundamisel saadud 160–180 °C juures keeva õlifraktsiooni keemilist koostist. Retentsiooniindeksite ja massispektrite abil identifitseeriti õlis ligi sada komponenti, mis kokku moodustasid 92% fraktsiooni koostisest. Peaaegu pool fraktsioonist koosnes aromaatsetest süsivesinikest. Põhikomponentideks osutusid dipenteen (35%) ja *p*-tsümeen (10%). Teised süsivesinike rühmad (alkaanid, alkeenid, alkadieenid ja tsükloalkadieenid) moodustasid kokku 7% fraktsiooni koostisest ning väävli- ja hapnikuühendid alla 1%.