Petroleum and Coal

Particulate Matter Index Profiles for Different Gasoline Samples Based on Hydrocarbon Groups

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Abstract

This project carries out research on the profiles of the Particulate Matter Index (PMI) of various samples of gasoline and petroleum products based on hydrocarbon groups. A detailed hydrocarbon analysis is used for qualitative and quantitative analysis of the profiles of the hydrocarbon groups. The PMI of the aromatic hydrocarbon group is found to be crucial for the overall PM index of the samples. The contribution of the aromatic hydrocarbon group composition to the PMI value is estimated to be 85%. Oxygen-containing components (about 10% ethanol, MTBE and ETBE) showed a limited improvement in the total PMI of the sample of 0.63% compared to the contribution of the PMI to aromatic hydrocarbons.

Keywords: *Particulate matter index; Gasoline; Gas chromatography; Hydrocarbon groups; Oxygen-containing components.*

1. Introduction

The increasingly stringent emission legislation of the European Union has led to increased scientific interest in particulate emissions into the environment. Respecting the problem, manufacturers of modern car engines are making a number of hardware and software changes to comply with the legislation for optimized reduction of particulate emissions [1]. Guided by policies to reduce greenhouse gas emissions and to achieve the goals of keeping global ambient temperatures increase below 2°C, automotive fuel producers are also developing new regulatory frameworks to specify fuels. Starting with specific regulations and showing how, what and where has been achieved in the production of automotive fuels, future prospects for fuel emission standards continue to be discussed [1]. A step in this direction is the introduction in fuel specifications concerning Euro 6 of a particulate matter (PN) emission limit of 6.0 × 10^{11} particles/km and a particulate (PM) emission of 4.5 mg/kg respectively [2].

One of the qualifying factors responsible for the composition of particulate emissions (PM) is of automotive fuels property [2]. Gasoline is a blend of several refinery streams (reformate, alkylate, FCC gasoline, oxygenates like MTBE, ETBE ethanol and others) the composition of which defines and satisfies the specified properties, established by regulatory or standard requirements. Particulate matter (PM) emitted by vehicles is known to be a complex combination of volatile and non-volatile substances. Volatile species usually contain sulphates, nitrates and organic matter, while non-volatile species are carbon and ash. The organic part contains thousands of compounds, incl. alkenes, aromatic hydrocarbons, ethers, ketones, alcohols [3]. There are publications in which attempts are made to link fuel composition to particulate emissions [4-6]. Exhaustive reviews and summaries of particulate matter emissions from diesel engines have found expression in a number of publications [7-9]. The properties and composition of gasoline engine exhaust gases are very different from those of diesel engines [9]. Various studies show that gasoline engines not only produce more particulate emis-

sions than diesel engines, but also produce smaller particulate matter. On one hand, particulate matter emissions from petrol engines are higher and on the other hand, these emissions are more toxic to public health [10].

Fuel quality is a key parameter for the assessment of the impact of petrol particulate matter over environment and a corresponding measure is needed for developing economies. Modern gas chromatographic instruments for gasoline qualification guarantee unambiguity in the identification of the types of components present [11]. Chromatographic analysis of petroleum fuels is the most reliable approach for obtaining information on both the presence of individual hydrocarbons and their distribution in the form of different types of hydrocarbon groups in gasoline [12-14].

Detailed hydrocarbon analysis (DHA) is still used in the petroleum industry to characterize light distillate fractions boiling up to 225°C. Many publications note that the analysis of gasoline fractions has already been sufficiently studied and evaluated in detail. Probably, these generalizations are related to the less complex hydrocarbon framework, which is easily and unambiguously determined at the level of individual components with the standard chromatographic test method. However, in the light of modern environmental protection requirements, there is an increased need for developing a reliable and detailed hydrocarbon method of analysis, which has to respond to these conditions.

A number of researchers have developed various indices in order to evaluate the particulate matter emitted from combustion of gasoline in spark ignition engines. They aim to relate the composition of the emissions to the quality of the fuel [6, 15-17]. While developing various indices, researchers rely on a number of basic physicochemical parameters of the fuel, such as vapor pressure, density, distillation and more. One of these indices is the particle index, developed by Aikawa, K. *et al.* that is a useful tool for guiding the trend of soot accumulation in fuel combustion [6, 15, 18, 19]. Currently, there is a lot of research, performed by various research laboratories and gasoline manufacturers, on particulate index evaluating the possibility of using it as a measure to clarify the impact of different types of gasoline on both soot formation on engine hardware and vehicles emissions [17, 20-22].

The present study focuses on the revealing of gathered data on particulate matter indices obtained from the distribution of the various hydrocarbon groups present in the specified types of gasoline, produced in LUKOIL Neftohim Burgas AD. The evaluation of the particulate index profiles of the different hydrocarbon groups in gasolines provides useful information for making efficient technological decisions in fuel production and, accordingly, the development of more effective technological control in order to achieve environmentally normalized conditions.

2. Experimental

2.1. Samples

Different types of gasolines and gasoline fractions manufactured by LUKOIL Neftohim Burgas AD Bulgaria were used in this study. The samples reformate, alkylate and C_5 fraction are one of the the main components for the production of gasoline in the refinery. The samples are sampled in glass bottles with screw caps. To prevent evaporation losses, samples were stored at 4°C in a refrigerator.

2.2. Apparatus

The samples were analyzed with a detailed hydrocarbon analyzer (DHA) equipped with an Agilent 7890A Series gas chromatograph, Agilent 7694A Series automatic injector. The instrument is equipped with a flame ionization detector. The analyzer is supplied with a capillary chromatographic column HP-1 (100% dimethyl polysiloxane stationary phase) with a length of 100 m and an inner diameter of 0.250 mm, with a film thickness of 0.50 μm and a pre-column DB-5 (5% diphenyl/95% dimethyl polysiloxane polymer) with an inner diameter of 0.250 mm and a film thickness of 1.00 μm. The control software of the analyzer is OpenLAB EZChrom Rev A.01.04 / B.04.04. Da Vinci Petro Reporter DHA software is used to simulate

the physical properties of the petrol-based quantitative results of the analysis (i.e. determination of hydrocarbon groups including paraffins, isoparaffins, olefins, naphthenic, aromatic components and oxygen-containing substances for each carbon number from C_2 to C_{15}). As carrier gas helium with a flow rate of 2.16 ml min^{-1} at a constant pressure of 41.064 psi was used. The temperature program of the column furnace is two-stage. The first step of the program is as follows: from 5°C (in 12 minutes) to 50°C at a speed of 5°C min^{-1} (in 51.7 min). The second temperature step of the program is from 50°C to 200°C (for 10 minutes) with a temperature programming speed of 1.5°C min^{-1}. The initial low temperature of the furnace is maintained by a cryogenic CO_2 refrigerant system. The tests performed are carried out in a carrier gas flow division mode in the ratio 150:1. The temperature of the injector and the detector is 250°C. The injection volume of the sample into the column is 0.1 µL.

3. Results and discussion

The identification of the individual components obtained by chromatography of the samples was performed by comparing certain retention data (retention times and Kovacs indices) with those of the compounds specified in the standard method ASTM D6730 [11]. After adjusting the peak area of each component using relative correction factors, the weight percentages of the compounds in each sample are calculated. Quantitative data for the individual components are grouped into a separate type of group such as n-paraffins, iso-paraffins, olefins, aromatic hydrocarbons, naphthenic, oxygen-containing components. The information on the quantitative content of the individual hydrocarbon groups are summarized and are presented in Table 1.

The data shows that the contents of the isoparaffin group and that of the aromatic hydrocarbons are in the highest concentrations and are quantitatively comparable except for the content of the isoparaffin group in samples F and G. All samples except samples C and G have more high quantitative content of aromatic hydrocarbon group compared to olefinic one. Aromatic hydrocarbons have the potential for high-impact components to form dust particles [16]. Their percentage in petrol must not exceed 35% (v / v) according to EN 228 [23]. The reformate produced in the refinery contains over 70%, mainly benzene, toluene, xylenes (BTX). The content of benzene (C_6) is comparable in all test samples, except samples E and G. The content of toluene (C_7) varies in the tested samples, and it is relatively comparable in quantitative terms for samples A, B and C. Most significant among the tested samples is the content of the aromatic compound with the number of carbon atoms of C_8-ethylbenzene, o-, m-, p-xylenes with the exception of sample F and G. Concentrations of aromatic hydrocarbons with carbon numbers C_9-C_{11} gradually decreased for all samples. For the highest boiling aromatic compounds with the carbon number C_{12}, their content in all samples is increased. Aromatic C_{11}-C_{12} hydrocarbons are represented by alkyl C_1- and C_2- substituted naphthalenes, C_1- and C_2-alkyl substituted indenes and C_1- and C_2-alkyl substituted indans. The content of the polycyclic aromatic hydrocarbon naphthalene in the samples varies from 0.072% to 0.130%.

The content of the olefinic hydrocarbon group is the important chemical properties of gasoline fuel. Their quantity in petrol must not exceed 18% (v/v) according to EN 228 [23]. Together with aromatic hydrocarbons, they are mainly responsible for VOC emissions from gasoline into the atmosphere [24]. In our studies, there were differences in the content of the olefinic hydrocarbon group among the tested samples. In this hydrocarbon group, the concentration of olefins with the C_5-C_7 hydrocarbon atoms are decisive. The petrol fraction (sample D), produced by the FCC plant (FCC), is the predominant base in the mixing petrol pool of the LUKOIL Neftohim Burgas refinery. Characteristic of this sample is that the content of the olefin group in its composition differs significantly from that in the other test samples.

The content of the iso-paraffinic hydrocarbon group is determined by the presence of mainly hydrocarbon components with C_5-C_8 atoms.

In our studies, there were no significant differences in the content of naphthenic hydrocarbon groups among the tested samples of petroleum products and fuels, except for the naphthenic group in sample D.

Table 1. Composition and content of the hydrocarbon groups in the samples investigated

Hydrocarbon groups %	A A95	B A95	C A98	D FCC gasoline	E Reformate	F Alkylate	G C5 fraction
C3 n-paraffin	0.13	0.08	0.06		0.28		
C4 n-paraffin	3.21	1.18	1.79	0.33	2.67	2.54	1.08
C5 n-paraffin	2.21	1.71	1.36	1.06	2.82		12.63
C6 n-paraffin	1.50	1.13	0.89	0.95	1.44		11.92
C7 n-paraffin	1.07	1.01	0.86	0.84	1.37		3.61
C8 n-paraffin	0.63	0.59	0.46	0.39	0.60		0.55
C9 n-paraffin	0.18	0.18	0.15	0.17	0.15		0.12
C10 n-paraffin	0.09	0.11	0.08	0.13	0.04		0.03
C11 n-paraffin	0.06	0.07	0.06	0.09			
C12 n-paraffin	0.02	0.02		0.05			
Total n-paraffins	9.10	6.08	5.71	4.01	9.37	2.54	29.94
C4 iso-paraffins	0.55	0.34	0.44	0.08	1.27	0.29	
C5 iso-paraffins	7.69	6.63	5.87	9.15	4.11	7.91	13.64
C6 iso-paraffins	6.49	5.80	4.92	7.25	3.74	1.52	22.93
C7 iso-paraffins	4.72	5.09	4.82	4.35	4.94	5.86	5.54
C8 iso-paraffins	8.59	14.16	14.99	3.46	2.52	69.87	1.32
C9 iso-paraffins	0.93	1.05	1.20	1.58	0.38	3.67	0.11
C10 iso-paraffins	0.59	0.59	0.57	1.04	0.07	3.18	0.02
C11 iso-paraffins	0.22	0.31	0.49	0.44		4.96	
C12 iso-paraffins	0.22	0.29	0.14	0.32		0.17	
Total iso-parafins	30.00	34.26	33.44	27.67	17.03	97.43	43.56
C6 aromatic	1.08	1.03	0.79	0.97	1.40		1.88
C7 aromatic	6.77	6.56	5.68	4.60	11.76		1.47
C8 aromatics	12.88	10.96	9.29	8.36	24.66		0.57
C9 aromatics	11.86	9.91	8.25	7.50	22.92		0.05
C10 aromatics	6.43	5.34	5.35	5.65	9.21		
C11 aromatics	0.73	0.74	0.40	0.89	0.69		
C12 aromatics	0.94	0.96	0.70	1.13	1.02		
Total aromatic hy-drocarbons	40.69	35.51	30.46	29.10	71.66		3.97
C5 naphthenes	0.23	0.14	0.10	0.15	0.04		3.80
C6 naphthenes	1.57	1.26	0.88	2.07	0.26		11.24
C7 naphthenes	1.82	1.48	1.03	2.56	0.24		5.94
C8 naphthenes	1.01	0.98	0.55	2.21	0.19		1.39
C9 naphthenes	0.18	0.06	0.19	0.66			0.16
C10 naphthenes	0.03			0.12			
Total naphthenes	4.84	3.92	2.75	7.77	0.73		22.53
C4 olefins	0.64	0.59	0.82	2.19	0.03	0.03	
C5 olefins	5.54	6.00	4.16	12.79	0.11		
C6 olefins	2.74	4.33	2.19	7.34	0.08		
C7 olefins	1.72	1.85	9.13	5.08	0.14		
C8 olefins	0.11	0.12	0.08	0.54	0.01		
C9 olefins	0.02			0.25			
Tottal olefins	10.77	12.89	16.38	28.19	0.37	0.03	
Ethanol content,(%)		4.48	4.51				
Mtbe content, (%)	4.60	1.10	5.62				
Etbe content, (%)		0.82	0.55				

Aikawa *et al.* is developed PMI [15]. It is represented by the following equation:

$$PMI = \sum_{i=1}^{n}\left(\frac{DBEi+1}{VP(443K)i} \times wti\right) \qquad (1)$$

where: wt_i is the weight fraction (%) of each component i identified in the sample composition; $DBEi$ is the number of double bonds in the molecule of component I; n is the total number of compounds in the sample; VP_i (443 K) is the vapor pressure of component i measured at 443 K.

DBEi binds to the unsaturation of the hydrocarbon molecule. It is calculated mainly from the number of carbon and hydrogen atoms in the molecular formula of the compound.

The vapor pressure of component is based on an empirical relationship between the physical properties such as boiling point of component i and its vapor pressure at 443 K (169.85°C). The value is not obtained by direct measurement of this property.

The constituent component of the PMI which is essential for its interpretation and study, is the quantitative content of component i in the sample. The remaining component in the PMI formula is an expression of the tendency of each component to form soot and its tendency to evaporate and physically mix with air [10].

The calculated PMI of each component, present in the test samples, is grouped according to the hydrocarbon type and are summarized in Table 2. The value of the PMI varies from 0.16 to 3.18 for the different test specimens. The accuracy of the determination of the PMI for each hydrocarbon group is presented with the accuracy of the determination of the hydrocarbon group by the developed gas chromatographic method. Accuracy of determining the content of the aromatic hydrocarbon group is 5%, the olefin group is 2.5%, the naphthenic group is 1.3%, the n-paraffin group and the iso-paraffinic hydrocarbon group are 1.7% and 1.4%, respectively. Accuracy of ethanol and MTBE content determination is 2.0% and 3.7%, respectively.

Table 2. Distribution of the PMI profiles of the hydrocarbon groups in the samples investigated

PM Index	A A95	B A95	C A98	D FCC gasoline	E Reformate	F Alkylate	G C5 fraction
PMI total n-paraffins	0.0113 ±0.0002	0.0104 ±0.0002	0.0080 ±0.0001	0.0119 ±0.0002	0.0091 ±0.0002	0.0005	0.0266
PMI total iso-paraffins	0.0465 ±0.0006	0.0679 ±0.0009	0.0712 ±0.0010	0.0596 ±0.0008	0.0220 ±0.0003	0.2906	0.0332
PMI total aromatic hydrocarbons	1.920 ±0.096	1.764 ±0.088	1.374 ±0.069	1.919 ±0.096	3.142 ±0.157		0.044
PMI C6 aromatic	0.006	0.006	0.005	0.006	0.008		0.011
PMI C7 aromatic	0.082	0.079	0.069	0.055	0.142		0.018
PMI C8 aromatics	0.303	0.258	0.189	0.197	0.581		0.013
PMI C9 aromatics	0.561	0.475	0.371	0.372	1.057		0.002
PMI C10 aromatics	0.493	0.549	0.455	0.605	0.677		
PMI C11 aromatics	0.303	0.269	0.198	0.492	0.315		
PM C12 aromatics	0.172	0.128	0.087	0.192	0.362		
PMI total naphthenes	0.0432 ±0.0006	0.0407 ±0.0005	0.0342 ±0.0004	0.0373 ±0.0005	0.0026 ±0.0001		0.0609
PMI total olefins	0.0162 ±0.0004	0.0209 ±0.0004	0.0406 ±0.0004	0.0529 ±0.0004	0.0008 ±0.0001		
PMI oxygen-containing compounds							
Ethanol	0.0032	0.0053	0.0053				
MTBE		0.0006	0.0040				
ETBE		0.0008	0.0005				
PMI total oxygen-containing compounds	0.0032 ±0.0002	0.0067 ±0.0002	0.0098 ±0.0002				
PMI (PIANO)	**2.0404**	**1.9106**	**1.5378**	**2.0807**	**3.1765**	**0.2911**	**0.1647**

The values of the PMI presented in Table 2 for the different samples are comparable to those determined by researchers who studied the impact of fuel properties on particulate emissions [25]. Our project studies the effect of composition of the aromatic hydrocarbon group on PMI value. In general, aromatic hydrocarbons are difficult to evaporate [26]. The conditionally aromatic hydrocarbon group is divided into two subgroups: the group composed of light (C_6-C_8) aromatic components and the group composed of heavy ones (C_9-C_{12}). The PMI values of light aromatic hydrocarbons in the studied samples are relatively close and their contribution to the compilation of the total PMI of the samples varies between 17-23%. The calculated PMI

values of the heavy aromatic hydrocarbons in the tested samples are in a positive correlation with the determined contents. They make a major contribution (around 85%) to the compilation of the overall PMI of the samples. The obtained data are consistent with similar studies [26]. The content of aromatic hydrocarbons is reduced in the direction of sample A to C, whereby the PMI of aromatic hydrocarbons also decreases in the described direction.

The value of the PMI of the olefin group in sample D differs from the calculated indices for this group in the other test samples. However, in quantitative terms, the contribution of this hydrocarbon group to the formation of the total PMI of the test samples varies from 0.8% to 2.6%, except for samples E and G. In general, the reduced quantitative content of the olefin group (in the direction of samples C → A) does not show an obvious improvement in the overall PMI of the samples.

Quantitative data from the calculated PMI of the iso-paraffinic hydrocarbon group in this study shows some benefits of 2.3% to 4.6% in forming the total PMI of gasoline samples. For the other gasoline fractions this trend is not observed - the contribution of the iso-paraffin group reaches 99.8% for sample F.

The calculated PMI of the other hydrocarbon groups show their insignificant contribution to the formation of the total PMI of the samples with the exception of sample F.

It can be seen from Table 2 that the tested quantitative contents of ethanol and MTBE in the different samples were not decisive for the formation of the PMI value of the sample. It can be concluded that these components do not make a significant contribution to the overall PMI of the test samples, which has also been discussed by other authors [27]. Oxygen-containing components (about 10% ethanol, MTBE and ETBE) (sample C) showed a limited improvement in the total PMI of the sample of 0.63% compared to the contribution of the PMI to aromatic hydrocarbons. Higher boiling aromatic components in gasoline lead to a much higher PMI of the tested samples.

Obtained total PMI of the samples A and E demonstrated that with the relatively comparable quantitative data for PMI of the different hydrocarbon groups, each of them shows some sensitivity which is in favor of experimental understanding of the influence of chromatographic composition on the distribution of the PMI of the different hydrocarbon groups.

4. Conclusion

The potential contribution of PMI of hydrocarbon groups to the formation of dust particles are assessed. Hydrocarbon groups are studied for their contribution to the formation of the values of the PM Index of various types of gasoline and refinery flows. Totally 6 hydrocarbon groups for 7 different gasoline and petroleum sample are used as objects. The most informative hydrocarbon group for shaping the profile of the PMI for the test samples is that of aromatic hydrocarbons.

The present research demonstrates successful application of the PMI profile of the different hydrocarbon groups in providing detailed quantitative information for assessing the potential of the chromatographic composition of petrol samples as a measure to clarify the impact of petrol on vehicle emissions.

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