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ABSTRACT

of the dissertation for the degree
of Doctor of Philosophy in Chemistry

**STUDY OF THE ANTIOXIDANT PROPERTIES OF
HETEROATOMIC CYCLIC COMPOUNDS AND MODIFIED
CARBON NANOTUBES**

Speciality: 2314.01 – «Petrochemistry»

Field of science: chemical science

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The work was performed at the Y.H. Mammadaliyev Institute of Petrochemical Processes of the Azerbaijan National Academy of Sciences at the "Liquid phase oxidation" laboratory

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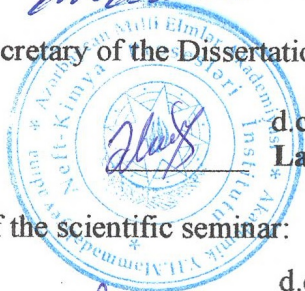
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GENERAL DESCRIPTION OF WORK

The current state of the problem and its relevance. Research, selection and use of effective antioxidants to stabilize the properties of fuels, mineral oils, lubricants and polymeric materials is one of the urgent tasks of petrochemistry. The oxidation of petrochemical industry products not only affects the presentation, but also contributes to the appearance in their composition of compounds that degrade performance. Fuels and polymers, like all organic materials, are also subject to oxidation. Oxidation of polymers or degradation leads to a change in viscosity, color and a deterioration in the physical and mechanical characteristics of polymers. For this reason, to improve the chemical stability of fuels and prevent the thermo-oxidative degradation of polymers, appropriate antioxidants (AO) are used, which are based on radical chain oxidation inhibitors of hydrocarbons and increase their resistance to atmospheric oxygen. Compounds of various classes are used as AO: phenols, aromatic amines, mixed aminophenols, hydroxylamines, phosphites, and also mixed-use AOs are used, in which synergistic effects are often manifested.

Inhibited oxidation is one of the largest sections of hydrocarbon oxidation in the liquid phase and solves the problem of selecting new effective AOs. One of the approaches to the selection and quantitative study of AO is the use of standard test reactions, for which the rate constants of elementary reactions are determined with high accuracy. Only with knowledge of such constants can these test reactions be used not only to determine the inhibitory effect of compounds introduced into the oxidative system, but also to determine the degree of their catalytic effect. This approach was first implemented in the present dissertation on the example of doped and modified carbon nanotubes.

It should be noted that, despite an intensive quantitative study, to date, only a small number of simple oxidation processes have measured the rate constants of elementary reactions and we can name only a few hydrocarbons (e.g. ethylbenzene, cumene) for which these

rate constants are determined with a high degree of accuracy. It was the use of such reactions that allowed us to obtain for the first time original quantitative information on a number of new objects - Lowinox CPL, Lowinox 22IB46, Naugard 445, Genox EP, Weston TNPP, 2,2' - methylenebis-[6-(1-methylcyclohexyl)-4-methyl]-phenol, investigate in detail the mechanism of their action, conduct targeted accelerated test methods in real systems (diesel fraction, polyolefin compositions) and recommend them for practical use. All of the above is the relevance of the undertaken dissertation research.

Object and subject of the research. Over the years of research dedicated to the study the processes of oxidation of organic materials and the ability of heteroatomic cyclic compounds to affect the stability and durability of systems, along with experimental work, theoretical studies were widely carried out, allowing both to explain the results obtained from the experiment and to predict new properties of substances, new effects in them and new areas of application.

According to worldwide research, it is reasonable to assume that the presence of the inherent antioxidant activity of carbon nanotubes will be a good tool for solving problems of oxidative stability of the polymer composites.

All this, and served as the basis for a set of research works related to the use of heteroatomic cyclic compounds (Lowinox CPL, Lowinox 22IB46, Naugard 445, Genox EP, Weston TNPP, 2,2'-methylenebis- [6-(1-methylcyclohexyl)-4-methyl]-phenol) and carbon structures (carbon nanotubes, fullerene soot) as antioxidant-inhibitors.

The purpose and objectives of the dissertation.

1. Determination of the activity of individual compounds (Lowinox CPL, Lowinox 22IB46, Naugard 445, Genox EP, Weston TNPP, 2,2'- methylenebis - [6 - (1 - methylcyclohexyl) - 4 - methyl] - phenol), in particular, the study of the rate of oxygen absorption in the reactions of aerobic oxidation of cumene in the presence of these compounds;
2. Determination of thermal stability of diesel fuel in the presence of

the above individual compounds;

3. Determination of the activity parameters of modern carbon nanostructures and the search for possible areas of their application:

- ❖ the study of the rate of oxygen absorption in the model reaction of cumene initiated oxidation in the presence of bromine-containing single-walled carbon nanotubes;
- ❖ the study of the rate of oxygen absorption in the model reactions of cumene and decalin initiated oxidation in the presence of the latest generation of Carbo Gran NI MWCNTs;
- ❖ the study of the rate of oxygen absorption in the model reaction of ethylbenzene initiated oxidation in the presence of fullerene soot and studying the effect of fullerene soot on the physico-mechanical characteristics and thermal properties of low density polyethylene;

4. Analysis of antioxidant-inhibitors in oil compositions of complex composition:

- ❖ the study of the rate of oxygen absorption in the model reaction of cumene initiated oxidation in the presence of heavy oil residues (fraction of heavy pyrolysis resin, resins, asphaltene-like compounds and oil phosphor of heavy pyrolysis resin) as potentially available and inexpensive antioxidants.

Investigation methods. On investigation there were used the following methods: X-ray - photoelectron spectroscopy, atomic absorption spectroscopy, spectral (IR) analysis, determination of qualitative measures of diesel fuel by GOST 9144-79, thermogravimetry (TG).

The main provisions submitted to the defense. Research, selection and use of the studied compounds as effective antioxidants for stabilizing the properties of diesel fuel and polymer materials, namely:

- determination of the inhibitory ability of the tested compounds;
- selection of new effective antioxidants that improve the thermo-oxidative stability of diesel fuel;
- study of the activity of carbon nanotubes and fullerene soot, and its

use as an inhibitor that improves the thermal properties of compositions with low density polyethylene.

The scientific novelty of the work. The effectiveness of new objects was investigated for the first time, in particular, 6 inhibitors - antioxidants - Lowinox CPL, Lowinox 22IB46, Naugard 445, Genox EP, Weston TNPP, 2,2' - methylenebis - [6 - (1-methylcyclohexyl) - 4-methyl] - phenol in the model reactions of cumene initiated oxidation and diesel fuel. For the first time, the inhibitory ability of fullerene soot in a model reaction was studied, and its effect on the physico-mechanical and thermal properties of a polyethylene composition. All the main parameters characterizing the activity of these objects in various oxidizing media were determined - the initiated oxidation of alkyl aromatic hydrocarbons (cumene, ethylbenzene), the oxidation of diesel fuel under conditions of intense decomposition of hydroperoxides, and climatic aging of polyethylene compositions. For the first time, numerical values of parameters such as inhibition rate constants, oxidation chain termination coefficients, and general inhibitory activity for individual synthesized compounds were found. The features of the mechanism of action of antioxidants were first established and interpreted. It has been established that some antioxidants (Naugard 445, Genox EP) exhibit a unique mechanism of multiplet action. The effect of bulky substituents in heteroatomic compounds, as well as the structural morphology of carbon nanocompounds (Carbo Gran NI, (Br)_n-SWCNTs) on the effectiveness of antioxidants, is evaluated.

The practical value of the work. The obtained antioxidant activity parameters are a reference material and can be used for targeted research of compounds of similar structure for stabilizing fuels and polyolefin compositions. It was determined that the studied individual AOs increase the thermo-oxidative stability of diesel fuel. The introduction of both individual compounds and complex compositions of AO into the reaction system increases the thermal oxidative stability of diesel fuel from 1,6 to 10 times. The work provides practical recommendations on the rational use of carbon nanostructures (CNS) as antioxidants, namely, an indispensable

condition is the purification of CNS from the residues of metal catalysts that are used for their synthesis, and modified CNS Br - containing compounds that provoke bromide oxidation catalysis should be avoided. The results obtained allow us to recommend the studied individual compounds as effective antioxidants.

Publications. On the topic of the dissertation published 19 scientific papers, including 11 articles, 8 abstracts.

Approbation of a work. The results of the work were published in national and international journals: Reports of the NAS of Azerbaijan - 1 article; News of higher technical educational institutions of Azerbaijan - 1 article; The journal "Scientific notes" of the Azerbaijan Technical University - 1 article; Petrochemical and Oil Refining Processes - 1 article; Oil Refining and Petrochemistry (Russia) - 2 articles; Oil and Gas technologies (Russia) - 3 articles; Journal of Vinyl & Additive Technology (USA) - 1 article; Applied Catalysis A-General (Netherlands) - 1 article.

The main results of the thesis were discussed: at the Republican scientific conference dedicated to the 100th anniversary of academician A.M. Guliyev (Baku, 2012), The Republican scientific conference dedicated to the 80th anniversary of M. Nagiyev Institute of Catalysis and Inorganic Chemistry (Baku, 2016), The International scientific and technical conference "Petrochemical synthesis and catalysis in complex condensed systems" dedicated to the 100th anniversary of academician B.K. Zeynalov (Baku, 2017), The International scientific-practical conference "Innovative prospects for the development of oil refining and petrochemicals", dedicated to the 110th anniversary of academician V.S. Aliyev (Baku, 2018), The scientific conference "Nagiyev Readings" dedicated to the 110th anniversary of academician M. Nagiyev (Baku, 2018), The international scientific conference "Actual Problems of Modern Chemistry" dedicated to the 90th anniversary of the academician Y.H. Mammadaliyev Institute of Petrochemical Processes (Baku, 2019), THERMAM (Germany, 2020), Republican scientific - practical conference on "Problems of modern chemistry and development trends" (Baku, 2020).

Place of dissertation. The work was performed in accordance with the work program of the IPCP ANAS 14/2006, registration number 0106Az00010.

Personal participation of the author. The author outlines the main goals of the study and the tasks to achieve them, identifies areas of research, carried out processing, systematization and discussion of the results. The author also took a direct part in the formulation and conduct of laboratory and pilot tests.

The structure and scope of the dissertation. The dissertation work consists of an introduction (15122 signs), five chapters (166920 signs), conclusions (3386 signs), a list of cited scientific literature of 208 titles (45969 signs), 28 tables, 11 figures and 22 graphs and includes a total of 185428 signs.

The introduction examines the relevance of the presented dissertation, the goals and objectives of the study, scientific novelty and practical value, as well as the scope of the results.

The first chapter, a literature review, reflects the current state of work on the classification of modern AO inhibitors and new areas of their application. The features of the mechanism of action of carbon nanostructures in the reactions of aerobic oxidation of organic substances and potential applications of functionalized fullerenes and nanotubes are considered. In particular, the role and importance of native oil antioxidants in oxidation processes are considered.

The second chapter is the experimental technique. It describes the objects used in the research and their cleaning methods, a description of the gasometric installation and finding the optimal experimental conditions - temperature and initiator. Then, the basic technique for processing kinetic oxidation curves and calculating the kinetic parameters of AO activity is shown.

The third chapter is a quantitative study of individual inhibitors. It defines the rate constants and inhibition coefficients of individual AOs. Tests were conducted to determine the thermal-oxidative stability of diesel fuel in the presence of individual AOs and their complex compositions.

The fourth chapter is the determination of the activity options

of carbon nanostructures. It summarizes the results of studies to determine the activity of carbon nanotubes containing Br and a metal of variable valency on the model reaction of the initiated oxidation of cumene and fullerene soot on the model reaction of the initiated oxidation of ethylbenzene.

The fifth chapter is devoted to the study of the inhibitory properties of fractions, resins, asphaltene compounds of pyrolysis origin and an oil phosphor.

The work ends with conclusions and a list of cited literature.

MAIN CONTENT OF THE WORK

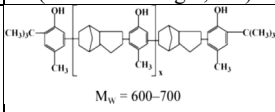
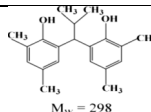
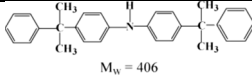
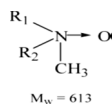
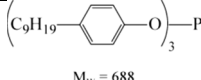
1. Quantitative study of individual inhibitors and determination of their effect on the thermo-oxidative stability of diesel fuel

Changes in the properties of petrochemical industry products lead to poor quality and reduced product life. One of the main reasons is the oxidation of products, which can be prevented by the introduction of small amounts of chemical compounds - highly effective antioxidants, and the determination of the main parameters of their activity is of great practical importance.

The properties of AO were studied using the model reaction of cumene oxidation¹ at the initiation rate $W_i = 1,7 \times 10^{-8}$ - $6,8 \times 10^{-8}$ Ms⁻¹, temperatures: 60 and 80 ($\pm 0,02$)°C, oxygen pressure $P_{O_2} = 20$ kPa (air) [4]. Cumene has a purity of 98% (Aldrich). 2,2'-azo-bis-isobutyronitrile (AIBN) was used as an initiator. The volume of the reaction mixture is 10 cm³. The chain development constants for the model cumene oxidation reaction at temperatures of 60 and 80°C are as follows: $k_3 = 1,75$ and $4,05$ M⁻¹s⁻¹; cumene concentration $[RH] = 6,9$ and $6,76$ mol/l. The main characteristics of individual AOs are presented in table 1.

¹Дахнави, Э.М., Кошель, Г.Н. Окисление изопропилбензола до гидропероксида в присутствии N-гидроксифталимида / Дахнави, Кошель, Г.Н. // Нефтехимия, -Москва:-2017. №2,-с.86-90.

Table 1.
Characteristics of individual antioxidants

Trade name	Melting range, °C	Molar activity of AO groups (lnH/ kg AO)	Chemical structures (molecular weight, M _w)
Lowinox CPL: 4-(4,6,8-trimethylnonan-3-yl) benzenesulfonic acid	>105	4,3 (x=1)	 M _w = 600-700
Lowinox 22IB46: 2,2'-isobutylidene-bis(4,6-dimethyl-phenol)	158-164	6,7	 M _w = 298
Naugard 445: 4,4'-bis(α,α dimethylbenzyl) diphenylamine	98-100	2,46	 M _w = 406
Genox EP: amins, bis (hydrogenated rape-oil alkyl)methyl, N-oxide (R ₁ , R ₂ =C ₁₄ -C ₂₄ alkyl chains)	85-105	1,63	 M _w = 613
Weston TNPP: trisonnylphenyl phosphate	liquid at room temp.	4,36	 M _w = 688

Lowinox CPL. By structure, Lowinox CPL is a typical sterically hindered phenolic AO and contains three functional hydroxyl groups (x = 1, n = 3). The main kinetic parameters of inhibitory activity are calculated and summarized in table 2.

Table 2.
Basic kinetic parameters of Lowinox CPL

f(n=3)	[C _Σ], mol/l	τ _Σ , min.	[C ₍₁₎], mol/l	τ ₁ , min.	[C ₍₂₎], mol/l	τ ₂ , min.	k ₇₍₁₎ , M ⁻¹ s ⁻¹	k ₇₍₂₎ , M ⁻¹ s ⁻¹
2,4±0,2	4×10 ⁻⁶	14±1	3,1×10 ⁻⁶	11±1	0,9×10 ⁻⁶	3±0,5	(1,5±0,2) × 10 ⁵	(2,8±0,3) × 10 ⁴
2,4±0,2	1,5×10 ⁻⁵	27±2	1,2×10 ⁻⁵	21±2	0,3×10 ⁻⁵	6±1	(1,9±0,3) × 10 ⁵	(5,5±0,6) × 10 ⁴
2,5±0,2	5×10 ⁻⁶	37±3	3,8×10 ⁻⁶	28±2	1,2×10 ⁻⁶	9±1	(1,4±0,2) × 10 ⁵	(4,4±0,5) × 10 ⁴
On average							lg k ₍₁₎ = 5,2±0,1	lg k ₍₂₎ = 4,6±0,1
Reduced to	1,0×10 ⁻⁴	180±3	0,8×10 ⁻⁴	140±2	0,2×10 ⁻⁴	40±7	(1,6±0,3) × 10 ⁵	(4,2±1,0) × 10 ⁴
A = Σ {k _{7(i)} (f _n [lnH]) _(i) } = [(1,6×10 ⁵)×6,0×(8×10 ⁻⁵)+(4,2×10 ⁴)×1,5×(0,2×10 ⁻⁴)] = 80 ± 10 c ⁻¹								

Lowinox CPL exhibits two active antioxidant functional groups. The first group is valid for 80% of the total induction time, the second - only for 20%. The experiments show that for each hydroxyl functional group the highest factors are $f = 2,4 - 2,5$, which indicates the presence of an additional antioxidant effect.

Lowinox 22IB46. This AO has two functional hydroxyl groups ($n = 2$) of potential inhibitory activity. The main kinetic parameters of this AO are calculated and summarized in table 3.

Table 3.
Basic kinetic parameters of Lowinox 22IB46

$f(n=2)$	$[C_2]$, mol/l	τ_2 , min.	$[C_{(1)}]$, mol/l	τ_1 , min.	$[C_{(2)}]$, mol/l	τ_2 , min.	$k_{7(1)}$, $M^{-1}s^{-1}$	$k_{7(2)}$, $M^{-1}s^{-1}$
$2,4 \pm 0,2$	$3,7 \times 10^{-5}$	43 ± 3	$1,9 \times 10^{-5}$	22 ± 2	$1,8 \times 10^{-5}$	21 ± 2	$(3,3 \pm 0,4) \times 10^5$	$(9,6 \pm 0,9) \times 10^3$
$2,45 \pm 0,2$	$2,0 \times 10^{-5}$	24 ± 2	$0,9 \times 10^{-5}$	11 ± 1	$1,1 \times 10^{-5}$	13 ± 1	$(1,5 \pm 0,3) \times 10^5$	$(1,05 \pm 0,1) \times 10^4$
$2,45 \pm 0,2$	$8,0 \times 10^{-5}$	96 ± 6	$4,2 \times 10^{-5}$	50 ± 4	$3,8 \times 10^{-5}$	46 ± 4	$(3,5 \pm 0,4) \times 10^5$	$(8,5 \pm 0,8) \times 10^3$
$2,5 \pm 0,2$	$9,4 \times 10^{-6}$	46 ± 6	$4,3 \times 10^{-6}$	21 ± 3	$5,1 \times 10^{-6}$	25 ± 4	$(1,9 \pm 0,3) \times 10^5$	$(8,3 \pm 0,9) \times 10^3$
On average							$\lg k_{(1)} = 5,4 \pm 0,15$ $\lg k_{(2)} = 3,96 \pm 0,05$	
Reduced to	$1,0 \times 10^{-4}$	120 ± 8	$0,5 \times 10^{-4}$	60 ± 6	$0,5 \times 10^{-4}$	60 ± 7	$(2,5 \pm 0,8) \times 10^5$	$(9,2 \pm 1,0) \times 10^3$
$A = \sum \{k_{7(i)}(f_i [\ln H]_{(i)})\} = [(2,5 \times 10^5) \times 2,5 \times (5 \times 10^{-5}) + (9,2 \times 10^3) \times 2,5 \times (5 \times 10^{-5})] = 32 \pm 8 \text{ c}^{-1}$								

As a result of the experiment, it was found that Lowinox 22IB46 contains two inhibitory centers of different activity, and the inhibition coefficient f for the hydroxyl group of this AO is also $2,4 - 2,5$. Calculations showed that the total inhibitory activity for Lowinox 22IB46 is 2,5 times less than that of Lowinox CPL.

During the model oxidation, two OH groups of Lowinox 22IB46 break two oxidation chains, but they exhibit different antioxidant activity: for the first hydroxyl group, the inhibition constant is $k_7 = (2,5 \pm 0,8) \times 10^5 \text{ M}^{-1}\text{s}^{-1}$, while the other show weaker inhibition: $k_7 = (9,2 \pm 1,0) \times 10^3 \text{ M}^{-1}\text{s}^{-1}$

Naugard 445. Although the Naugard 445 molecule has only one functional NH - group, two centers of different activity are manifested here. This was confirmed by higher values of the inhibition coefficient for the NH - group. As can be seen from the table 4, the inhibition coefficient was greater than expected, $f = 2$,

which was achieved due to the additional antioxidant effect of the compounds obtained during the inhibited oxidation of Naugard 445.

Table 4.
Basic kinetic parameters of Naugard 445

$f(n=1)$	$[C_{\Sigma}]$, mol/l	τ_{Σ} , min.	$[C_{(1)}]$, mol/l	τ_1 , min.	$[C_{(2)}]$, mol/l	τ_2 , min.	$k_{7(1)}$, $M^{-1}s^{-1}$	$k_{7(2)}$, $M^{-1}s^{-1}$
2,5±0,2	$4,9 \times 10^{-5}$	30±2	$3,8 \times 10^{-5}$	23±2	$1,1 \times 10^{-5}$	7±1	$(7,6 \pm 0,6) \times 10^4$	$(1,8 \pm 0,2) \times 10^4$
2,5±0,2	$7,4 \times 10^{-5}$	46±3	$5,8 \times 10^{-5}$	36±3	$1,6 \times 10^{-5}$	10±1	$(8,8 \pm 1,0) \times 10^4$	$(2,0 \pm 0,2) \times 10^3$
On average							$\lg k_{(1)} =$ 4,91±0,04	$\lg k_{(2)} =$ 4,28±0,05
Reduced to	$1,0 \times 10^{-4}$	62±6	$0,8 \times 10^{-4}$	48±5	$0,2 \times 10^{-4}$	14±6	$(8,2 \pm 0,8) \times 10^4$	$(1,9 \pm 0,2) \times 10^4$
$A = \Sigma \{k_{7(i)} (f_n [\text{InH}])_{(i)}\} = [(8,2 \times 10^4) \times 2,0 \times (0,8 \times 10^{-4}) + (1,9 \times 10^4) \times 0,5 \times (0,2 \times 10^{-4})] = 13 \pm 2,0 \text{ c}^{-1}$								

The total inhibitory activity of Naugard 445 decreased to a certain standard concentration of AO, and in comparison with previous AOs, this is 2,5 and 6 times less than in the case of Lowinox 22IB46 and Lowinox CPL, respectively.

Genox EP. The chemistry of this hydroxylamine in the reactions of aerobic oxidation of hydrocarbons is quite complex. Genox EP effectively inhibits the model reaction of cumene oxidation (Table 5).

Table 5.
Data of the antioxidant activity of Genox EP in a model reaction of cumene initiated oxidation

T, °C	(Genox EP) × 10 ⁴ , mol/l	τ , min.	f_n	W_{constO_2}	
				$\text{mm}^3 \text{O}_2 \text{ min}^{-1}$	$M \text{ s}^{-1} \times 10^6$
60	1,0	15 ± 2	0,6 ± 0,1	88	6,0
60	2,5	37 ± 4	0,6 ± 0,1	85	5,8
60	5,6	85 ± 5	0,6 ± 0,1	67	4,6
80	1,1	10 ± 3	0,4 ± 0,1	131	8,9
80	2,3	22 ± 6	0,4 ± 0,1	121	8,2
80	3,3	35,5 ± 4	0,4 ± 0,1	102	6,9

As can be seen from the table 5, the apparent inhibition coefficient f for Genox EP is significantly lower than that of previous AOs. This means that some parts of the reactive forms from Genox EP act as acceptors of peroxide radicals, thus, as a result, there is a sharp

decrease in oxidation rates at the beginning of the reaction, while the rest interact with alkyl radicals, slowing down the post - induction oxidation rate.

Table 6.
Basic kinetic parameters of Genox EP

f n=3	$[C_7]$, mol/l	τ_7 , min.	$[C_{(1)}]$, mol/l	τ_1 , min.	$[C_{(2)}]$, mol/l	τ_2 , min.	$[C_{(3)}]$, mol/l	τ_3 , min.	$k_{7(1)}$, $M^{-1}s^{-1}$	$k_{7(2)}$, $M^{-1}s^{-1}$	$k_{7(3)}$, $M^{-1}s^{-1}$
0,2	5,6 $\times 10^{-4}$	85 \pm 5	3,1 $\times 10^{-4}$	47 \pm 3	1,4 $\times 10^{-4}$	21 \pm 2	1,1 $\times 10^{-4}$	17 \pm 2	(7,0 \pm 0,5) $\times 10^4$	(3,0 \pm 0,2) $\times 10^4$	(1,0 \pm 0,1) $\times 10^4$
On average									$\lg k_{(1)} =$ 4,85 \pm 0,04	$\lg k_{(2)} =$ 4,48 \pm 0,03	$\lg k_{(3)} =$ 4,0 \pm 0,05
$A = \Sigma \{k_{7(i)}(f_i [\text{InH}])_{(i)}\} = [(7,0 \times 10^4) \times 0,2 \times (5,5 \times 10^{-5}) + (3,0 \times 10^4) \times 0,2 \times (2,5 \times 10^{-5}) + (1,0 \times 10^4) \times 0,2 \times (2,0 \times 10^{-5})] =$ $1,0 \pm 0,1 \text{ c}^{-1}$											

During test trials, Genox EP shows numerous inhibitory centers: three types of fixed inhibitory forms with different rate constants $k_7 - k_{7(1)} = (7,0 \pm 0,5) \times 10^4$, $k_{7(2)} = (3,0 \pm 0,2) \times 10^4$ and $k_{7(3)} = (1,0 \pm 0,1) \times 10^4 \text{ M}^{-1}\text{s}^{-1}$ (Table 6). However, the level of antioxidant activity is not so high, due to the low values of inhibition coefficients and the fact that relatively high concentrations of AO are required to obtain similar inhibitory effects, as in the case of primary phenols and amines. However, it should be borne in mind that Genox EP is a double-type AO and, therefore, has a long-lasting antioxidant property.

Thus, the stabilizers used in this study were very strong AOs with high inhibition rate constants. Drawing conclusions from the foregoing, the studied AOs can be placed in the following series in terms of their antioxidant effectiveness: Lowinox CPL > Lowinox 22IB46 > Naugard 445 > Genox EP.

2,2' - methylenebis- [6- (1-methylcyclohexyl) -4-methyl] - phenol.

The structure of this AO was interesting from the point of view of the presence of bulky side substituents. AO activity is determined by a number of factors, among which the most important is the stability of the phenoxyl radical. Therefore, sufficiently voluminous cyclohexyl fragments should have ensured such stability and minimized chain extension reactions involving the phenoxyl radical. On the other hand, in the literature there are practically no exact

quantitative data on the activity of the investigated 2,2'-methylenebis - [6- (1-methylcyclohexyl - 4-methyl)] - phenol (2,2'-MBMCHMP). Thus, the obtained kinetic parameters for the antioxidant activity of 2,2'-MBMCHMP were to replenish the bank of similar data existing for other phenols.

The main parameters of inhibitory activity are calculated and summarized in table 7.

Table 7.
The main parameters of inhibitory activity
of 2,2'-MBMCHMP

№ №	Quantity of 2,2'- MBMCHMP		$W_{inh.o_2} \times 10^7$, mol/l·s	τ , min.	f_n	$k_7 \times 10^4$, l/mol·s	A, s ⁻¹
	$[InH] \times 10^{-4}$, mol/l	ml					
1.	0,5	0,1	4,2	16±4	1,3±0,5	3,0	1,95±0,1
2.	1,0	0,2	3,4	23,5±5	1,0±0,5	2,5	2,5±0,1
3.	2,0	0,4	2,2	43±2	0,9±0,3	2,1	3,78±3
4.	3,0	0,6	0,9	76±3	1,0±0,5	3,0	9,0±6
5.	4,0	0,8	0,7	101±2	1,0±0,5	2,9	11,6±8
On average				$\lg k_7 = 4,43 \pm 0,03$			

From the obtained values of the inhibition coefficient, we can conclude that the 2,2'-MBMCHMP molecule breaks only one oxidation chain with an average inhibition rate constant of $4,43 \times 10^4$ l/mol·s.

Over the years, in connection with the tightening of the quality indicators of fuels, research has been intensively carried out related to the thermo-oxidative transformations of fuels and the search for effective ways to stabilize them. Among the compounds used as stabilizing additives, using diesel as an example, phenolic, amine, and multicomponent compound systems are most widely used. Thus, in the search for the fields of application of the investigated AOs, in the present dissertation, studies were conducted to determine their thermo-oxidative stability as the AO of diesel fuel. The essence of the method is to determine the mass of sediment formed during the oxidation of diesel fuel on a JICAPT device at 120°C for 4 hours (GOST 9144-79).

Table 8.
The amount of sediment in the samples
of diesel fuel after testing

The concentration of the introduced compounds is 0.004% wt.

Test compound	Draft quantity, mg /100 sm ³ fuel
Without AO	6,0
Lowinox 22IB46	0,6
Weston TNPP	7,0
Naugard 445	5,48
2,2'- MBMCHMP	4,4
Lowinox 22IB46 + Weston TNPP	3,8
Lowinox 22IB46 + 2,2'-MBMCHMP	2,5
Weston TNPP + 2,2'- MBMCHMP	2,8

Analyzing the obtained data (Table 8), Lowinox 22IB46 showed the best result, and this confirmed the results of studies on the determination of antioxidant activity. This AO increases the thermal oxidative stability of diesel fuel by 10 times [17]. Also, the introduction of the Lowinox 22IB46 + Weston TNPP mixture into the reaction system increases the thermal oxidative stability of diesel fuel by 1,6; Lowinox 22IB46 + 2,2'-MBMCHMP - in 2,4; Weston TNPP + 2,2'-MBMCHMP – 2,2 times, respectively [13,16].

2. A quantitative study of the activity of functionalized carbon nanotubes in a model reaction of initiated cumene oxidation

Carbon nanotubes (CNTs), which have their special chemical, structural, mechanical and electronic properties, are becoming increasingly important materials for use in various fields of the chemical, polymer and petrochemical industries.

In this work, samples of functionalized CNTs that have been tested are bromine-containing single-walled CNTs ((Br)_n - SWCNTs), grades Br-80-120504, Br-81-120509, Br-82-120514. In the process of preparing (Br)_n - SWCNTs, SWCNTs of the commercial sample “Elicarb” manufactured by Thomas Swan & Co. Ltd., were used [9]. The composition of the investigated (Br)_n - SWCNTs was determined by X-ray photoelectron spectroscopy

(XPS). The elemental composition is as follows:

1. Br-80-120504 (1 h of exposure): C = 94,8, O = 3,3, Br = 2,0%, O / C = 0,035, Br / C = 0,021;
2. Br-81-120509 (2 h of exposure): C = 87,3, O = 6,3, Br = 6,4%, O / C = 0,072, Br / C = 0,073;
3. Br-82-120514 (crude): C = 96,9, O = 3,1%, O / C = 0,032.

The model oxidation of cumene was carried out in the presence of various concentrations of $(\text{Br})_n$ - SWCNTs with an initiation rate of $W_i = 6,8 \times 10^{-8} \text{ mol / l}\cdot\text{s}$, $60 (\pm 0,02)^\circ\text{C}$ and $P_{\text{O}_2} = 20 \text{ kPa}$.

Table 9.
The rate of cumene initiated oxidation
in the presence of $(\text{Br})_n$ - SWCNT

Samples	Quantity of $(\text{Br})_n$ - SWCNT,		$W_{\text{O}_2} \times 10^{-6}$, mol/l·s
	$[\text{InH}] \times 10^{-4}$, mol/l	g/l	
Br-80-120504	20,0	4,0	6,6
Br-81-120509	10,0	2,0	7,6
Br-81-120509	20,0	4,0	9,0
Br-82-120514	20,0	4,0	8,0

The $(\text{Br})_n$ - SWCNTs introduced into the reaction medium showed a catalytic effect. Having examined the data obtained table 9 it can be said with accuracy that the presence of $(\text{Br})_n$ - SWCNTs in the reaction system causes a catalytic effect, which means that the data studied $(\text{Br})_n$ - SWCNTs are potential catalysts for liquid-phase oxidation of hydrocarbons.

A possible mechanism for the catalytic action of $(\text{Br})_n$ - SWCNT samples can be explained by the formation of reactive oxygen species due to the distribution of electrons on the CNT surface. It is known that the accumulation of Br - containing groups on the surface of CNTs continues in an unusual order perpendicular to the surface of CNTs and, as a result, the area on which bromine accumulates has a shifted electron density due to the excess of the inductive effect, which in turn can provoke the formation of reactive oxygen species.

3. Determination of the activity of the latest generation Carbo Gran NI highly walled carbon nanotube highly dispersible in polar solvents in the aerobic oxidation of hydrocarbons

This chapter is devoted to the test determination of the activity of MWCNTs Carbo Gran NI on the model reaction of the initiated oxidation of cumene and decalin. MWCNTs Carbo Gran NI are prepared using special technology from Future Carbon GmbH (Germany). As a result of the experiments, it was found that the Carbo Gran NI introduced into the MWCNT system exhibit a catalytic effect. A possible mechanism of catalytic action can be explained by the presence of variable valence metal in the composition of MWCNTs. In order to detect the presence of metals in the studied MWCNTs, inductively coupled plasma atomic absorption spectroscopy was performed on an IRISInterpidiIXPL instrument (ThermoElectronCorp., USA) by radial and axial observations at wavelengths of 310 and 95,5 nm. It was found that Carbo Gran NI contains 0,33% wt. gland.

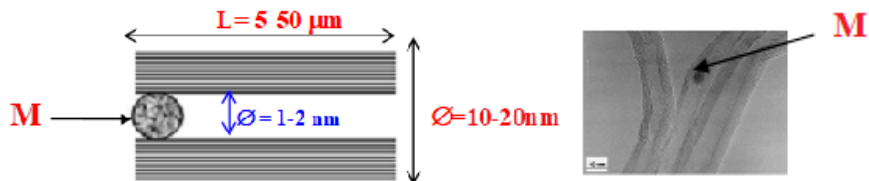


Figure 1. Schematic image and a photograph of the electron microscope of a Carbon Gran NI MWCNT

**Table 10.
The rate of cumene initiated oxidation in the presence of MWCNTs Carbo Gran NI**

№	Quantity of the Carbo Gran NI		W _{O₂} × 10 ⁻⁶ , mol/l·s
	[InH] × 10 ⁻⁴ , mol/l	g/l	
1.	0	0	6,1
2.	2,5	0,5	9,5
3.	7,5	1,5	13,0
4.	12,5	2,5	14,3

Based on the calculated parameters presented in table 10, where the catalytic effect is quite clearly observed, it became advisable to conduct studies on the model reaction of the initiated oxidation of decalin. The experiments were carried out under standard conditions: the volume of the reaction mixture was 10 cm³, the amount of AIBN initiator was 10 mg, and the temperature was 60°C. A control experiment (in the absence of a catalyst) showed that oxygen uptake was not observed within 80 minutes of the reaction. However, the results of a model reaction of initiated decalin oxidation in the presence of 25 mg Carbo Gran NI MWCNTs show that the reaction rate is 8 mm³O₂ / min. or 0,5 × 10⁻⁶ mol O₂ / l · s. It seems that in the case of cumene and decalin oxidation in the presence of Carbo Gran NI, the carbon carrier greatly facilitates electron transfer from the metal (Figure 2).

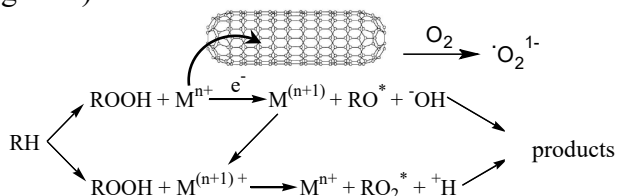


Figure 2. Schematic representation of electron transfer in an oxidative process in the presence of Carbo Gran NI

4. Correlation between the rate constant of inhibition of fullerene soot in the model reaction of the initiated oxidation of ethylbenzene and its stabilizing activity in polyethylene compositions

In the present work, the kinetic parameters of fullerene soot as AO are first studied using by the model reaction of the initiated oxidation of ethylbenzene. The antioxidant effect of fullerene soot was studied by its consumption rate in oxidizable ethylbenzene under standard conditions. From the literature data, the values of the rate constant of elementary reactions for the oxidation of ethylbenzene at 60°C are as follows: $k_3=2,38$ l/mol·s, $k_6=1,7 \times 10^7$ l/mol·s, $W_i = 6,8 \cdot 10^{-8}$ mol/l·s, $[RH] = 7,84$ mol/l.

Table 11.

The main parameters of the inhibitory activity of fullerene soot

№ №	Quantity of fullerene soot		W _{inh.O2} , mm ³ /min.	τ, min.	f _n [InH]×10 ⁻⁵ , mol/l·s	k ₇ ×10 ⁴ , l/mol·s	A×10 ⁻¹ , s ⁻¹
	[InH]×10 ⁻³ , mol/l	mg					
1.	2,77	2	7,2	6±2	2,45 ± 0,3	10,7	2,6
2.	6,93	5	5,3	13,5±4	5,5 ± 0,3	6,4	3,5
3.	13,87	10	2,5	24,5±2	10,0 ± 0,5	7,4	7,4
4.	27,7	20	1,4	29,7±3	11,8 ± 0,5	11,2	13,2

The results obtained indicate that fullerene soot exhibits inhibitory properties. The model reaction proceeds with an induction period that increases in proportion to the amount of added antioxidant.

Relying on a literary material in which fullerenes and fullerene soot embedded in a polymer matrix effectively prevent the thermal oxidative degradation of polymer compositions, the effect of fullerene soot on the physicomechanical characteristics and thermal properties of low density polyethylene (LDPE) was studied by derivatographic analysis. Studies to determine the physicomechanical and thermal properties of fullerene soot in LDPE compositions have shown that thermal properties and weight loss when heating the composition are improved when fullerene soot is introduced into the sample LDPE, and the optimal percentage of fullerene soot is 0,5% wt.

5. Analysis of inhibitors - AO in complex oil compositions

In this work, we determined the parameters of the inhibitory activity of heavy petroleum residues (HPR) by the method of cumene initiated oxidation. In addition, a change in the inhibitory activity of the fraction (F_{HPR}), resin (R_{HPR}), and asphaltene-like compounds (A_{HPR}) of the heavy resin of pyrolysis (HRP) during photo irradiation was revealed.

Having examined the obtained data (Table 12), we can conclude that if we take the average molecular weight of the components of F_{HPR} as 500, R_{HPR} and A_{HPR} as 1000, then approximately 30% of the

total number of molecules have antioxidant activity.

Table 12.
The main parameters of the inhibitory activity
of FrHRP, RHRP and AHRP

Samples	W_{inhO_2} , mm ³ O ₂ /min	τ , min.	$f_n[\text{InH}]_{TCH} \times 10$, mol/l	$k_7 \times 10^3$, l/mol·s	$A \times 10^{-3}$, l/s·kg
FrHRP	13	46±5	6,3 ± 0,7	5,7±0,6	3,5 ± 0,5
RHRP	15	16±2	3,3 ± 0,5	0,8±0,2	0,3 ± 0,1
AHRP	14	18±3	2,8 ± 0,5	1,4±0,2	0,35 ± 0,1

Further, photoirradiated samples – FrHRP-3 (3 hours), RHRP-1 (1 hour), RHRP-3 (3 hours), AHRP-1 (1 hour) and AHRP-3 (3 hours) were introduced into the model system of cumene initiated oxidation and were tested for inhibitory activity.

Table 13.
The parameters of the inhibitory activity of
FrHRP-3, RHRP-1, RHRP-3, AHRP-1, AHRP-3

Samples	W_{inhO_2} , mm ³ O ₂ /min	τ , min.	$f_n [\text{InH}]_{HRP(hv)} \times 10$, mol/kg	$k_7 \times 10^3$, l/mol·s	$A \times 10^{-2}$, l/s·kg
FrHRP-3	12	25 ± 3	3,3 ± 0,3	10 ± 1,0	3,3 ± 3
RHRP-1	13	19 ± 2	3,8 ± 0,3	1,3 ± 0,1	4,9 ± 0,5
RHRP-3	12	23 ± 3	4,6 ± 0,5	1,1 ± 0,1	5,0 ± 0,5
AHRP-1	12	20 ± 2	3,9 ± 0,3	1,3 ± 0,1	5,2 ± 0,5
AHRP-3	11	24 ± 3	4,8 ± 0,5	1,2 ± 0,1	5,6 ± 0,6

Comparison of the results of experiments given in tables 12 and 13 show the effect of secondary inhibition upon photoirradiation of FrHRP, RHRP and AHRP. In all cases, during photo irradiation, the effective concentration of inhibitory centers increases, on average, by 1,5 times, the total inhibitory activity, on average, by 1,6 times, and the rate of inhibition constant, on average, by 1,2 times. Drawing conclusions, we can state the fact that during the photoirradiation of RHRP and AHRP there is an effect of a certain increase in the level of inhibitory activity due to structures exhibiting an additional antioxidant effect.

Then, a series of experiments were conducted to identify the antioxidant activity of the oil phosphor (OPh) on the model reaction of the initiated oxidation of cumene. In addition, it was quite interesting

to follow the change in the inhibitory activity of OPh_{HRP} during photo irradiation. OPh obtained from the fraction > 400°C of heavy pyrolysis resin of straight-run gasoline of the Sumgait Ethylene-Polyethylene plant was used in the experiments. It was found that OPh additives inhibit the oxidation reaction to some extent, but the oxidation rate when leaving the induction period is only $50 \pm 5 \text{ mm}^3\text{O}_2 / \text{min.}$, while it should be twice as high, because displays the oxidation rate of pure cumene. Photoirradiated samples also exhibit slight inhibitory activity. Moreover, it is possible that during the oxidation of these samples, the formation of active particles ($\text{R}\cdot$, $\text{RO}_2\cdot$, $^1\text{O}_2$) occurs, leading to a deterioration of their inhibitory properties.

Given the very high rate constant of the attachment of alkyl radicals to oxygen, which is $10^9 \div 10^{10} \text{ l} / \text{mol}\cdot\text{s}$, the antioxidant activity of OPh_{HRP} seems to be very insignificant.

CONCLUSIONS

1. For the first time, the inhibitory properties of primary basic AOs were studied on a model reaction of initiated cumene oxidation. According to the total inhibitory activity, the studied AOs can be arranged in the following row: Lowinox CPL > Lowinox22IB46 > Naugard445 > Genox EP > Weston TNPP [4, 10, 11].
2. Based on the model reaction of the initiated oxidation of cumene, the antioxidant activity of the synthesized 2,2'-MBMCHMP was studied. It was found that the introduction of 2,2'-MBMCHMP in the reaction system leads to a more intense inhibition of the process due to an increase in the concentration of functional active centers of AOs, leading to an increase in the chain termination rate [5, 13].
3. Individual AOs have been investigated for their ability to improve the thermal oxidative stability of diesel fuel. Lowinox 22IB46 showed the best result, as increases the thermal-oxidative stability of diesel fuel by 10 times, and the mixture Lowinox 22IB46 + Weston TNPP – 1,6 times, Lowinox 22IB46+2,2'-MBMCHMP –

- 2,4 times, Weston TNPP+2,2'-MBMCHMP – 2,2 times [14,16, 17].
4. The inhibitory activity of $(Br)_n$ - SWCNTs was studied. It was established that introduced $(Br)_n$ - SWCNTs into the system show a catalytic effect under experimental conditions. A possible mechanism for the catalytic action of $(Br)_n$ - SWCNT samples can be explained by the formation of reactive oxygen species [9].
 5. For the first time, the activity of a new generation MWCNTs - Carbo Gran NI, was studied. An analysis of the obtained kinetic parameters showed that Carbo Gran NI introduced into the MWCNT system exhibit a catalytic effect. It has been established that the possible mechanism of the catalytic action of Carbo Gran NI MWCNTs is explained by the presence in its composition of a metal of variable valence - iron (0,33% wt.), which is confirmed by atomic absorption spectroscopy [8,15].
 6. Based on the results of studies in the field of antioxidant activity, fullerene soot manifests itself as an inhibitor. It was found that the thermal properties of the PE composition are improved when fullerene soot is introduced into the PE sample, namely, the optimal percentage of fullerene soot is 0,5% wt. [19].
 7. It has been established that the inhibitory parameters of resins and asphaltene-like compounds of HRP are almost the same; the total inhibitory activity of the fraction of HRP is 10 times greater than that of resins and asphaltene-like compounds. It was determined that during photoirradiation of the studied resin and asphaltene-like compounds of HRP, the effective concentration of inhibitory centers increases by a factor of 1,5, the rate of inhibition constant is $\sim 1,2$ times, and the total inhibitory activity is $\sim 1,6$ times [1-3].

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