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**PROBLEMS, TRENDS AND PROSPECTS OF PRESENT-DAY
AUTOMATIVE ENGINE OIL FORMULATIONS
(on the basis of analytical review of technical literature
and patents for the years 1990-2006)**

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Abstract. Based on the analysis of the issued for the past 15 years scientific papers dedicated to scientific aspects of the engine oil improvement and taking into account the requirement to oil performance resulting from economical and ecological factors, an attempt to formulate the scientific principles for engine oil formulations has been made.

Key words: engine oil, additives, additive ratio, performance criteria, scientific basis, quantitative concept.

The level of requirements for the automotive engine oils is conditioned by the problems to be solved at a certain stage of internal combustion engine improvement.

Due to environment pollution, global warming and lack of hydrocarbon energy resources exhaust gas emissions reduction along with the fuel economy became the main problems engine designers as well as fuel and lubricant formulators are facing.

Aimed at solving these problems changes in the engine construction (such as turbocharging, gasoline injection, exhaust gas recirculation (EGR), oxidation catalysts, diesel particulate filters (DPF), and NO_x storage reducing catalysts) as well as changes in the fuel composition (such as lead replacement, sulfur and aromatic levels reduction, utilization of alternative fuels like alcohols components, oxygenates, biodiesel, natural gas and biogas, et al.) formed rigid requirements to engine oils performance, primarily regarding their oxidation inhibition, antiwear and detergent-dispersing properties [1-10].

Attempts of improving oil performance by increasing additives variety and concentration only were a little success as they resulted in deterioration of some basic oil properties, such as colloidal stability, neutralizing and antiwear abilities, low temperature viscosity, foam tendency, and elastomer compatibility [8, 11-16].

The level of modern scientific investigations in the field of engine oil improvement as well as experimental results obtained for the last decades allow to formulate a quantitative scientifically based concept of oil formulations,

which would take into account in the first place chemical aspects, such as oil components structure, their interactions, influence on oil performance characteristics, and their changes in time [17-48].

This concept is based on the requirements to the engine oil performance set in ILSAC GF-4, PC-10, ACEA E 4, 5 specifications:

- reduction of phosphorus (< 0.1 %), sulphur (< 0.5 %) and sulphated ash (< 1.5 %) levels by reducing concentration of ash additives, primarily dithiophosphates (ZnDDF), sulphonates, and sulphurized phenates without diminishing the properties provided by these additives;
- improvement of antifriction properties and fuel economy;
- increase of colloidal stability, reduction of foam tendency, and improvement of elastomers compatibility;
- improvement of biodegradability and reduction of harmful emissions.

The basic ways of increasing engine oil performance in view of the aforementioned requirements is determining scientific criteria of quality for base oils and additives and optimization of oil formulations.

Among the base oils the preference is given to oils of III-V groups as specified in the API. These are hydrocracked mineral stocks and synthetic oils based on poly-alpha-olefins (PAO), alkyl esters of different carboxylic acids and alcohols, branched ethers with modified terminal hydroxyl groups, alkyl benzenes, Fischer-Tropsch products (GTL, gas to liquid oils), et al.

Mineral oils show good compatibility with additives and elastomers, but their rheological and inhibition properties essentially depend on refinement degree. Apart from that their biodegradability is low. Performance of such oils cannot be increased without employing such expensive processes as hydrocracking and hydroisomerisation. This is the only way the level of such undesirable components as hard aromatics and polycyclic naphthenes can be decreased [49-54].

Synthetic oils of definite structure may be obtained, but they exhibit own shortcomings and are too expensive.

PAO as decene and dodecene oligomers show high thermostability to deposit formation, but they can be subjected to oxidation in presence of oxygen due to tertiary hydrogens in their structure. They have good rheological characteristics (high VI), low pour point (< 243 K), and are less stable to biodegradation than mineral oils. On the other hand, PAO insufficiently dissolve additives and shrink elastomers. Due to their high viscosity and volatility a low molecular PAO alone do not allow to obtain engine oils of OW grade [55-58].

Esters and ethers are characterized by high rheological property, low evaporative loss, a good ability to improve antiwear and antifriction properties, as well as biodegradability. However, they are not compatible with elastomers and not sufficiently dissolve some additives. Due to double bonds esters based on plant oils exhibit low thermostability and thus need to be modified or stabilized with special oxidation inhibitors [59-62].

Alkyl benzenes have good rheological properties, high thermostability, low pour point, and are stable to temporary viscosity loss. Yet, their part in a composition cannot exceed 15 % not to affect detergent and neutralizing properties. Their biodegradability level is low [63-65].

Use of GTL oils in engine formulations is widening now. They are affordable, more biodegradable than mineral oils, practically do not contain sulphur and aromatics, have low viscosity and low evaporative loss (on their 100 % base engine oils of OW grade can be formulated). However, in order to ensure the high rheological properties and sufficient pour point the optimum degree of wax isomerisation is necessary. GTL oils are not sufficiently stable under thermal oxidation, that is why they need to be stabilized with antioxidants [66-69].

The common criteria for base oils performance are as follows: low kinematic viscosity at 373 K (3-10 mm²/s); low sulphur and nitrogen level (< 0,03 %); low degree of structural aggregation (low values of thermodynamic parameters of viscous flow, $H < 25$ kJ/mol, $T S < 15$ kJ/mol) and high viscosity index (VI, > 120); low evaporative loss, < 15 %; compatibility with additives and elastomers, aniline point 333-383 K; high flash point (> 473 K, COC); low level of aromatics (< 15 %); low level of polycyclic naphthenes (< 1 %, ratio between monocyclic and polycyclic naphthenes > 15 %); optimum degree of isomerisation (6-9 CH₃ groups for 100 C-atoms); pour point < 258 K [70-72].

Criteria for additives quality are based on the use of products with definite structure and predicted properties.

Metal detergents with high detergency and good neutralizing properties, sufficient antiwear and antifriction abilities, and a high colloidal stability are needed. Calcium highly based (150-300 mgKOH/g) disubstituted C10-40

alkyl salicylates and sulphur free phenates are preferred. These additives comprise not less than 50 mol % of all detergents in oil composition. Hydrocarbon substituent (secondary) and organic salt (one benzene ring in the molecule) structure are of great importance [73-77].

Formaldehyde treated calcium salicylates and sulphur free phenates known as saligenines, calixarenes, salixarenes, and linear polymers are also worth taking into consideration as detergents [78-80].

Despite the common tendency for reduction of sulphonates in modern formulations their overbased calcium dispersions (400-500 mg KOH/g) with regulative ratio of crystalline and amorphous structures of micellar carbonate cores, being responsible for friction modifying and deposit control, are advantageous of use [81-83].

Because of deterioration of oxidation inhibition and antiwear performance use of magnesium detergents, despite of their antirust pronounced property and ability of providing a low temperature viscosity as compared with calcium additive, is diminished in the modern engine oils [39, 84].

Sometimes in order to improve detergent and anticorrosion properties of the engine oils the simultaneous use of calcium and sodium detergents is practiced (sodium detergents amount to 10-15 % of common basity) [85, 86]. Still, in opinion efficiency of sodium additives seems to be overrated [38]. Thus these detergents hardly have any prospects at least without substantiation of optimum ratio between calcium and sodium components.

Although complete removing of ZnDDF from oil formulations is being predicted, at present only reducing of their treat rates is observed.

Among the additives alternative to ZnDDF, which impart the complex of oxidation inhibition, antiwear and friction modifying properties to engine oils, hindered phenols, aromatic amines, clusters of *d* and *f* metals, mainly, molybdenum (dithiocarbamates, amine and alcohol complexes), oil solubled copper salts (oleates, alkenylsuccinates et al.), N-heterocycles (thiadiazoles, triazines et al.), sulphidated olefins, esters and amides of carboxylic, phosphoric and thiocarbamoyl acids and different boron compound (succinimides, boron esters, borates of alkali and alkaline metals) are of wide use.

Structure preferences for these additives have been defined. As regards hindered phenolic antioxidants the liquid products containing amine or ester groups and possessing a high level of HOMO (highest occupied molecular orbital) energy so as to be functional above 423 K are of primary importance [87]. As aromatic amines diphenyl and phenyl-naphthyl amines alkylated (C⁴⁺) on aromatic ring are of exceptional use [88]. Boron containing additives seem to become mandatory components of the modern engine oils. Structures having high molecular hydrocarbon radical and possessing optimum ratio B/C, B/N, B/Ca are preferred. Increasing of hydrolytic stability of boron additives is of

special importance to be taken into consideration [74, 89-92]. The optimum structures of N-heterocycles are operated with electron density on N atom and availability of oxygen or sulphur containing substituents [93, 94]. Efficiency of molybdenum clusters depend on the number of the molybdenum (Mo) atoms (nuclei), its oxidation degree (three-nucleus complexes of Mo⁺⁶ are preferred), structure of alkyl substituent and number of oxygen atoms near molybdenum atoms (minimum) [25, 26, 32, 33, 95-101]. In order to enhance thermal stability molybdenum clusters should be used in combination with other antioxidants [25, 26, 97]. The preferred structure of sulphidated olefins is that having intramolecular sulphide linkages [102]. Optimum structure of phosphoric esters contains aryl substitutes [103], and esters of carboxylic acids are estimated by the value of the hydrophilic-hydrophobic balance (>15) [104] or by the value of in-polar index - ratio of molecular mass to number of ester groups (> 500) [88, 105].

Among antiwear and friction modifying additives attention should be drawn to nano dispersions of solid lubricants, such as metals, their oxides and carbonates, fluoroplastics, et al, as well as fullerenes and serpentines [106-112].

Ashless dispersants have not lost their importance for modern engine formulations, especially those of high temperature dispersing ability, improved detergency and antiwear properties, good compatibility with elastomers and low foam tendency. Alkenylsuccinimides on the base of highly reactive polybutenes (M_n = 1300-200, M_n/M_w < 4, a low molecular fraction (M_n < 500) content < 10 %, mostly terminal vinylidene double bonds) of bisimide structure with basic nitrogen content not exceeding 50 mol. %, so far meet these criteria. Modification of the additive with cyclic carbonates, boric acid, et al is desirable 45-48, 113-118].

As a rule the modern engine oils are multigrade formulations thickened with viscosity index modifiers. These additives have to meet the following requirements: thickening ability (a low polymer treat rate, < 1.0 %), viscosity temperature relationship ability (a low thermodynamic parameter of viscosity flow for oil solutions, a high viscosity index, > 120), high shear stability (after Bosch injector test oil is within viscosity grade), stability to temporary viscosity loss (oils of newtonian viscosity mode are preferred) and sufficient thermooxidative stability. Viscosity modifiers exhibiting dispersing ability are desirable [119-124]. Additives on the base of ethylene-olefins and hydrogenated styrene-diene copolymers in which ethylene moieties comprise ≥70 % largely meet these requirements (M_n = 5.000-30.000, M_n/M_w < 4, [η] = 1.3-1.8 dl/g, index of shear stability < 18 %, melt point < 303 K). Amphiphilic block copolymers and star polymers having definite structure and predictable properties are preferred [124].

Optimization of engine oil compositions consists in transition from empirical to scientific approach in oil formulation quantitatively joining together oil composition and its performance. Taking synergistic effects into account such approach imparts a complex of balanced performance features to engine oils by intensive use of additives along with minimization of their range and optimization of their concentration rate. Oil formulations are based on scientifically grounded additives ratio: Zn/N_{disp} = 1.5-3.5, Ca/Zn = 2-4.5 (7-8 in the absence of dispersant), B/N_{disp} = 0.3-0.9, Ca_{sulph}/Ca_{sulph} = 0.1-0.5, B/Ca = 0.1-1.0, Zn/Ca_{hi} ~ 5; Zn_{disp}/Zn_{dte} 4.7 [26, 36, 37, 41, 45, 73, 74, 125].

Conclusion

To sum up, a common tendency of up-to-date engine oil formulations consists in elaborating the scientifically grounded compositions consisting of oil components of definite structure and predicted performance in which by means of optimum additives ratio account is taken of the additives interaction and mechanism of their action.

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СУЧАСНІ МОТОРНІ ОЛИВИ - ПРОБЛЕМИ, ТЕНДЕНЦІЇ ТА ПЕРСПЕКТИВИ

Анотація. На основі аналізу опублікованих за останні 15 років результатів дослідження наукових аспектів підвищення якості моторних оливо зроблена спроба з урахуванням вимог, зумовлених економічними та екологічними чинниками, сформулювання наукових принципів формування сучасних композицій.

Ключові слова: моторна олива, добавки, співвідношення додатків, критерії якості, наукові засади, кількісна концепція.