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Article

Controlled Degradation of Lubricating Media by Means of an Accelerated Electron Beam

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Abstract: The article deals with the possibilities of using electron accelerator for controlled aging of lubricating media used in special vehicles. During use, e.g., in combustion engines, the lubricants get contaminated and thermo-oxidative degradation also occurs. The pilot project confirms the hypothesis that ionizing radiation makes it possible to simulate the operating load of lubricating media, which was repeatedly confirmed by long-term monitoring of changes in viscosity of statistically significant samples of motor oils used in special equipment. Preliminary test results also show that there are likely to be possibilities to influence other selected properties, such as the coefficient of friction depending on the radiation dose. The authors describe physicochemical processes during irradiation and, in the example of kinematic viscosity, present summary results for selected lubricating media.

Keywords: engines; ionizing radiation; accelerated electron beam; changes in the properties of the lubricating medium

1. Introduction

Engine and gear oils are decisive, it can be even said that they are a significant structural component of power machines (combustion engines, etc.) and their functional systems (transmissions, hydraulic systems, etc.) for mobile technology. They support the basic lubrication functions, such as the separation of surfaces of dynamically stressed moving parts, reduction of friction and wear, cleaning, heat dissipation—cooling, sealing, protection against corrosion, thermal, oxidation and shear stability, transmission of forces and moments. They affect the power, fuel consumption, reliability and service life, etc. Engine and gear oils are technologically complex products whose utility properties are classified by several technical parameters. In terms of users, there are two basic specifications, namely viscosity and performance. The basic parameters of the oils include viscosity, temperature and oxidation stability, additive content, water content, rheological properties at low temperatures, etc.

Ionizing radiation is increasingly being used in the world to optimize the properties of different materials [1]. Electron accelerators are already industrially assigned to various technological processes (production lines of cables, tires, plastics, etc.). At present, electron accelerators are typically used in the field of radiation treatment and sterilization of materials (sterilization of medical devices, polymer and plant crosslinking, treatment of contaminated environmental compartments, modification of semiconductor structures, protection of historical artefacts, food protection, radiation aging simulation of components working in a radiation environment and others) [2,3]. The article presents basic information from the research of changes in basic properties of engine oils after high-energy electron irradiation.

The undesirable thermo-oxidative degradation of lubricating media and hydraulic fluids occurs during their use in combustion engines, gearboxes, hydraulic systems, etc. Ionizing radiation as a

massive energy source enables the simulation of heavy loads on these media and the acceleration of their degradation. Based on this, we assume that exposure to ionizing radiation could have been a significant alternative method of accelerated aging and degradation of lubricating fluids and hydraulic fluids, which in a positive case would allow deriving correlation relationships of their service life. Based on a thorough search of the available literature, carried out by a professional organization in the ScienceDirect, Springer and EBSCO databases, the authors concluded that the accelerated degradation of selected lubricant parameters has not been systematically addressed in the world yet. The present article includes findings from the first phase of the research, where the main objective was to develop a new alternative method of accelerated degradation of selected basic property of the lubricating media—viscosity by irradiation from accelerated electron beam. The second phase of the research will be focused on influencing the life of selected lubricating media using radiation-affected material. This paper presents the basic information from stability testing or degradation of the final product under extreme conditions [4–6].

In general, it would be correct to select lubricating oils for testing based on the origin of the base oil (selective raffinate, hydrocracked or polyalphaolefin base), but the selection of the tested samples was motivated mainly by the very specific practical needs and the possible use of the tested lubricating media in various operating conditions based on the specific assignment. These products were: (a) Ursa SUPER mineral motor oil for large and extremely loaded turbocharged and non-turbocharged diesel engines of special vehicles; (b) Shell Hellix HX7 synthetic motor oil for daily used petrol and diesel engines of passenger cars using biodiesel or ethanol as fuel; (c) Castrol EDGE Professional A5 synthetic engine oil for cars with powerful petrol and diesel engines; (d) Shell S4 ATF HDX hydraulic fluid for automatic and manual transmissions, hydraulic systems, or power control systems.

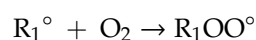
2. Physicochemical Processes in Irradiation

Radiation technology induces three kinds of physical changes in organic matter, namely (1) changes in the position of particles formed by a crystalline lattice (breakage of the lattice—vacancy defect and/or interstitial intergranular atoms), (2) formation of another kind of atoms due to nuclear reactions and (3) complex crystal disorders caused by massive heating of the crystal (different orientation of the crystal in relation to the original one). In the field of organic matter, two basic types of chemical changes are known, namely (1) disruption and rearrangement of covalent bonds in molecules and (2) polymerization of monomers. In the field of inorganic substances, in petroleum products to be specific, two kinds of chemical changes arise; they are (1) the formation of primary free electrons or molecular products and (2) biomolecular chemical reactions (capture of electrons on high affinity parts) to form negative ions, or recombination of electrons and positive ions to form radicals as a secondary process.

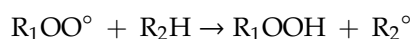
Petroleum oils are hydrocarbons with different chain length which is essential for viscosity values. When irradiating hydrocarbon materials with an electron beam, it can be accepted that the primary step is the interaction of electrons with hydrocarbon resulting in C-H bond excitation and subsequent formation of radicals. These phenomena can be described by three stages, namely initiation, propagation, and termination [4,7–9].



The forming radical oxidizes immediately in the atmospheric oxygen:



Peroxy radical R_1OO° is formed and reacts with another hydrocarbon molecule R_2H to hydroperoxy and a new radical R_2° :



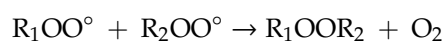
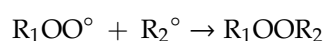
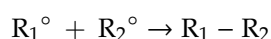
Hydroperoxyl is split easily due to low activation energy:



The resulting radicals may participate in the decomposition of other radicals, e.g., hydroperoxides and trigger the formation of new radicals. Carbonyl compounds of the type R-CO-R and RHO aldehydes are formed.

Propagation of the degradation process means cutting hydrocarbon chains into shorter fragments [8]. The physical manifestation of the described process, such as the decrease in molar mass, is also the decrease in viscosity of the oil.

Termination in which the generated radicals interact (recombination) to produce inactive products:



Terminally inactive products may have different chain lengths and branching. The creep of branched structures with lengths of branches comparable to the linear chain is made more difficult by creating multiple nodes with other chains. If a two-function radical is formed during the initiation of the chain, it can be recombined with two other radicals from two different chains. This creates a crosslinked structure that does not allow the independent movement of the connected chains and makes the flow of oil through the viscometer tube (capillary) more difficult, i.e., increases the viscosity. In the extreme case, so many crosslinked chains can be formed by forming slightly visually observable gel particles (the so-called gel point) in the oil, which can lead to a blockage of the viscometer capillary. Cleavage of hydrocarbon chains due to electron beam irradiation (in an inert atmosphere to prevent oxidation) could be compared to cracking oil to reduce the molar mass of high-molecular fractions (kerosene) and produce lower fractions, e.g., diesel and petrol.

Oils also contain unsaturated bonds that are more sensitive to the effect of the electron beam compared to the saturated bond. It is caused by lower dissociation energy of hydrogen in the unsaturated bond $R_x-CH=CH-R_y$ in comparison to the saturated bond $(R_xR_y)C-H_2$. When cleavage, branching, and linking of hydrocarbon chains occur, other unsaturated structures are also formed during hydrogenation. Increasing amount of unsaturated bonds results in a darker coloration of the irradiated oil even in carbonization [4,10,11]. This is related to the gradual change in colour of the irradiated oil, which has been experimentally verified repeatedly.

3. Experimental Part

The opinions of experts in the field of lubricant chemistry prefer the hypothesis that additives added to the base oil significantly affect the changes in the functional properties of the oils after ionizing radiation. These views led the authors to the decision that they would also test base oils without an additive package. These were the two samples of base oil. The first sample marked ZOT600-Chevron Texaco 600R and the second sample marked ZOT160 PUR160 from an anonymous manufacturer. The results of the kinematic viscosity testing are shown in the lower part of Table 1 and in Figures 2–4. The main goal of the present research is to test the coefficient of friction of irradiated oil samples in the cylinder-piston system with additionally applied ceramic coating SiO_2 from the combustion engine of a special vehicle on the UMT Tribolab equipment (BRUKER corporation, Manning Park Billerica, MA, USA), using Ball-on-Disc and Block-on-Ring methods (scheme) measurement. The possibilities of influencing (reducing) friction and wear are a complex scientific and practical task. It turns out that the friction and wear of the material surfaces are not the same in all cases and often depend on the specific conditions of use. The friction force is often not proportional to the loading force, the coefficients of

friction of the materials are not absolute and the environmental conditions (temperature, humidity, etc.) fundamentally affect them.

Table 1. Results of measurement of kinematic viscosity of irradiated oils.

Sample	Kind of Oil	Sample Treatment	KV100 (mm ² ·s ⁻¹)
R1	URSA SUPER (new)	before irradiation	12.5
R2	URSA SUPER	223.59 kGy (±3%)	19.2
R13	Shell HELIX HX7 5W-30 (new)	before irradiation	14.9
R14	Shell HELIX HX7 5W-30	23.36 kGy (±3%)	14.9
R15	Shell HELIX HX7 5W-30	42.88 kGy (±3%)	14.7
R16	Shell S4 ATF HDX (new)	before irradiation	11.1
R17	Shell S4 ATF HDX	59.99 kGy (±3%)	10.7
R18	Shell S4 ATF HDX	92.94 kGy (±3%)	10.6
R19	Castrol EDGE A5 0W-30 (new)	before irradiation	12.1
R20	Castrol EDGE A5 0W-30	23.36 kGy (±3%)	11.7
R21	Castrol EDGE A5 0W-30	42.88 kGy (±3%)	11.6
R22	Castrol EDGE A5 0W-30	59.99 kGy (±3%)	11.1
R23	Castrol EDGE A5 0W-30	92.94 kGy (±3%)	10.9
R24	Castrol EDGE A5 0W-30	223.59 kGy (±3%)	10.1
R29	Castrol EDGE 0W-30 (after 45.000 km)	before irradiation	11.2
R30	Castrol EDGE 0W-30	23.36 kGy (±3%)	11.1
R31	Castrol EDGE 0W-30	42.88 kGy (±3%)	10.8
R32	Castrol EDGE 0W-30	59.99 kGy (±3%)	10.7
R33	Castrol EDGE 0W-30	92.94 kGy (±3%)	10.5
R34	Base oil TOT600 (new)	before irradiation	17.6
R35	Base oil ZOT600	23.06 kGy (±3%)	18.6
R36	Base oil ZOT600	72.87 kGy (±3%)	18.8
R37	Base oil ZOT600	156.14 kGy (±3%)	18.9
R38	Base oil TOT160 (new)	before irradiation	7.7
R39	Base oil ZOT160	23.06 kGy (±3%)	7.9
R40	Base oil ZOT160	72.87 kGy (±3%)	8.0
R41	Base oil ZOT160	156.14 kGy (±3%)	8.9

Note: The values of the changes in kinematic viscosity are the arithmetic mean of three tests, while other statistical characteristics (variance, standard deviation, and coefficient of variation) were always calculated. The authors do not provide this information in the article.

For the irradiation, the UELR 5-1S linear accelerator was used at UCEA SMU (University Center of Electron Accelerators of Slovak Medical University) located in Trencin [3]. This accelerator produces electrons with energy in the range of 3.6 MeV to 6.2 MeV or X-ray radiation to 6.2 MeV energy. Doses from mGy up to MGy can be used for the radiation treatment of materials. Dosimetric workplace systems respect international standards and allow metering from low (Gy) to high doses (100 kGy) with an accuracy up to 3%. The overall view of the used equipment is in Figure 1.

The formation and acceleration of the electron beam is carried out in the accelerator structure (Figure 1, item 4) of the accelerator radiator. The electrons are emitted indirectly by a glowing oxide-nickel cathode and are formed by an electron-source optical system. The kinetic energy of the electrons increases by an electric field of the electromagnetic wave. The source of electromagnetic energy is an impulse magnetron, from which the energy is transmitted to a high vacuum accelerator structure. The beam of accelerated electrons is a sequence of short current pulses (3.5 μs) with frequency of 5 to 240 Hz. Beam focussing is realized by a high frequency field. Behind the accelerator structure is an inductive sensor whose signal is used by the control program to control the beam current. The output device provides the entry of the beam into the atmosphere, its decomposition, the length control and the measurement of its current. Beam decomposition takes place in a vacuum chamber [3,12–14].

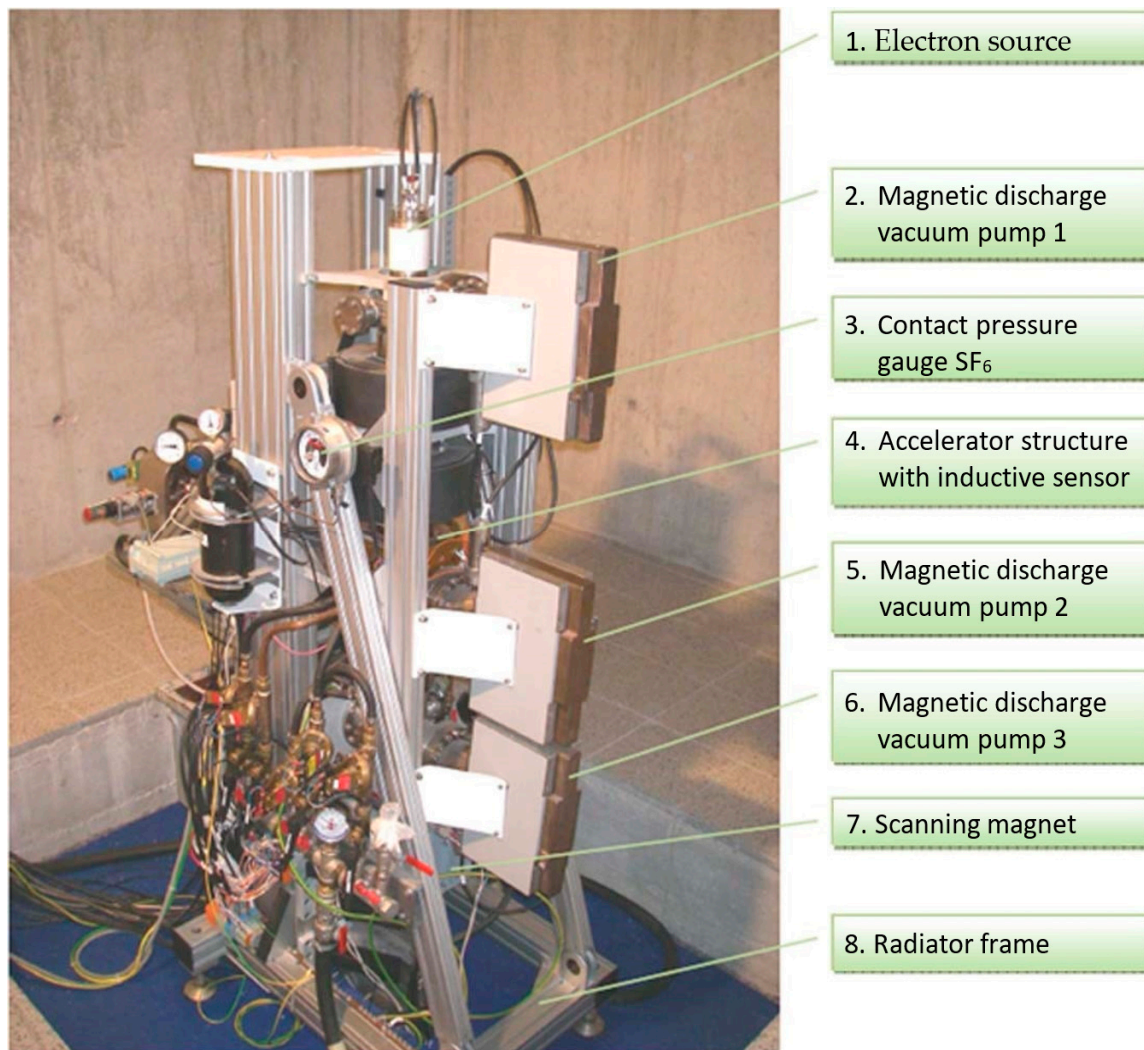


Figure 1. Overall view of the accelerator.

The basic quantity by which we characterize the effect of radiation on the substance is the absorbed dose. It is the amount of energy transmitted to the substance by radiation, the dose unit being 1 gray (Gy) with the dimension Jkg^{-1} . For the actual measurement of the irradiation dose, the radio-chromium film B3 was used as a dosimeter, allowing measurements in the range of 1 to 100 kGy, with an accuracy of 3%. The irradiation dose is determined from the spectrophotometer result.

While planning the oil radiation exposure experiment, it was decided, on the basis of available theories, literature research, and the practice of authors in similar areas, to examine the effect of irradiation on the essential property of lubricating oils, namely kinematic viscosity, which is a physical quantity indicating the ratio between tensile stress and the change of velocity depending on the distance between adjacent layers when the actual fluid flows. The viscosity clearly characterizes internal friction and is a function of attractive forces between particles. The relations of viscosity, temperature, pressure, and shear gradients were taken into account [4]. Oil samples came from two independent sources: Castrol EDGE 0W30 engine oils from a private company running Volvo XC60 vehicles and URSA SUPER, HELIX HX7 5W30 engine oils and Shell S4 ATF HDX automatic transmission oil from a state-owned organization using special vehicles [5,6]. The samples were irradiated with the same physical parameters of the accelerator, which included the beam scanning frequency 1 Hz, beam pulse frequency 120 and 240 Hz, the beam width of 40 cm and electron accelerating energy 5 MeV. The experimental procedure included 50 mL oil samples, Petri dishes of 90 mm diameter and 15 mm height. Dosimeters were fixed on the bottom of the Petri dishes and in front of them. The dishes and

dosimeters were placed on the accelerator conveyor and the conveyor speed was changed under the irradiation window in compliance with the desired irradiation dose.

4. Evaluation of Results and Discussion

The cumulative results of kinematic viscosity measurement of irradiated oils obtained during the experiment are shown in Table 1. Selected results of changes in kinematic viscosity due to irradiation for unused and worn Castrol EDGE 0W30 oil are shown in Figures 2 and 3. A Spectro VISC Q300 automatic viscometer from SPECTRO INC Industrial Tribology Systems, Littleton, MA, USA, specified according to standards: ATSM-D445, D446, D7279, IP 71 and ISO 3104, was used to measure kinematic viscosity. Furthermore, an AES (Atomic Emission Spectroscopy) spectrometer in the AES/RDE modification (with disk rotating electrode) Spectroil Q 100 RDE from SPECTRO INC Industrial Tribology Systems was used, supporting the JOAP program (U.S. Military's Joint Oil Analysis Program), Bruker Corporation's UMT TriboLab test system specified according to ISO, ASTM, and DIN standards and, for FTIR spectrometry, a NIVOLET Impact 410 instrument, manufactured by SpectroLab, Sylmar, CA, USA.

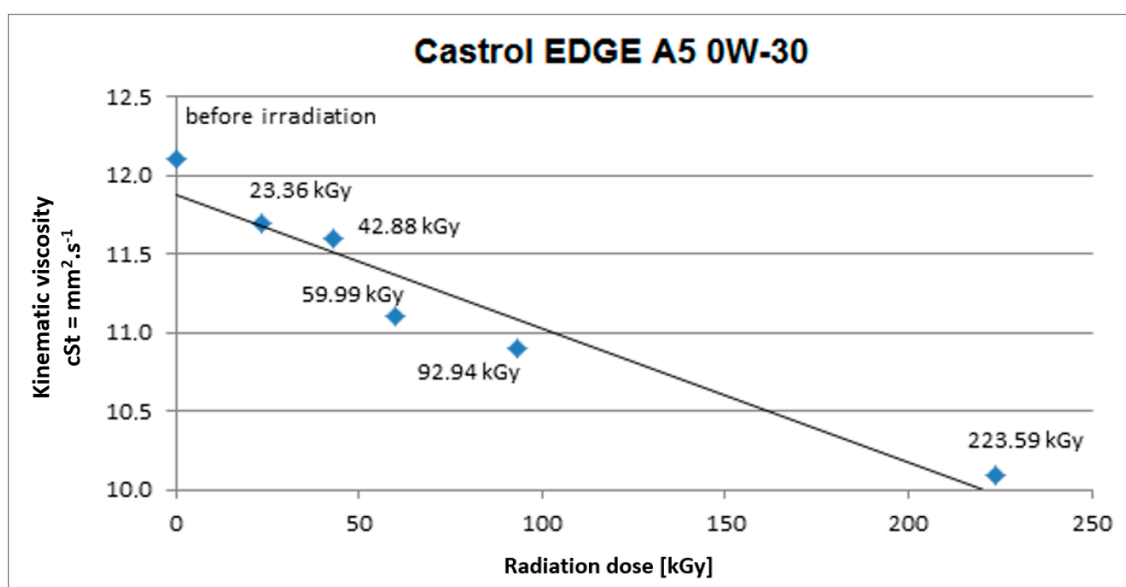


Figure 2. Change of kinematic viscosity due to irradiation of new oil.

For the selected worn lubricant, the kinematic viscosity was reduced by up to about 17% after irradiation and for the new lubricant the kinematic viscosity was reduced by about 12.5%. These results have not yet been verified by statistically significant data, but according to the first conclusions the exposure of dose 223.59 kGy appears as wear after about 45,000 km. However, this information is only an expert estimate and will be another part of the project to be solved.

Reduction in the kinematic viscosity of the oils after irradiation indicates the tearing of the original longer hydrocarbon or silicone chains to shorter ones, which is the first stage of the process. On the contrary, the increase in viscosity is caused by the so-called crosslinking, chemically indivisible linking of chains, which is a later stage of the process. This slows the flow of oil, which in the last stage leads to gel formation. These individual stages depend on the type of oil to be tested, the absorbed energy dose during irradiation, but also on the additive package and the ingredients present.

The decrease in viscosity of the R13–R34 samples indicates gradual degradation of the oil, tearing of chains (shortening of the chain length). The unchanged viscosity of the sample R14 in comparison to R13 can be interpreted in such a way that the dose ≈ 23 kGy in the given oil still did not induce such a degree of structural changes that would affect the viscosity. The rate of oxidation products in all samples could be identified by measuring infrared spectra in the range $160\text{--}170\text{ cm}^{-1}$. The highest applied irradiation dose ≈ 224 kGy in sample R2 caused an increase in viscosity, Figure 4. Since in

synthetic polymers the measurable gel is observed at an irradiance of about 200 kGy [1–3], it can be assumed that the presence of this fraction is responsible for the relatively high viscosity measured in this sample [4].

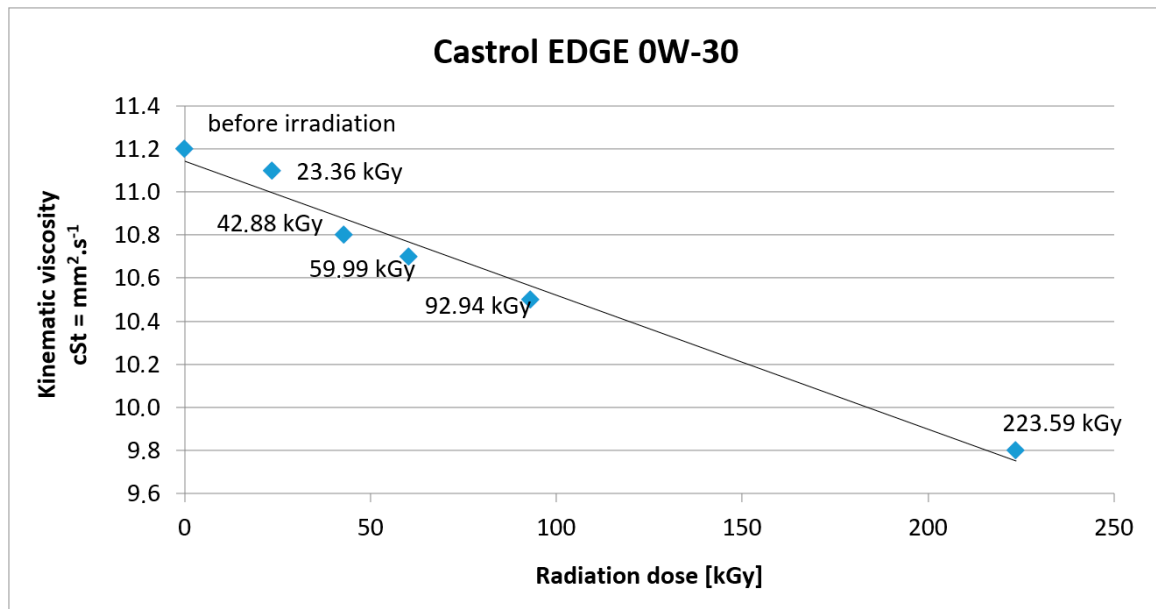


Figure 3. Change of kinematic viscosity due to irradiation of used oil (45,000 km).

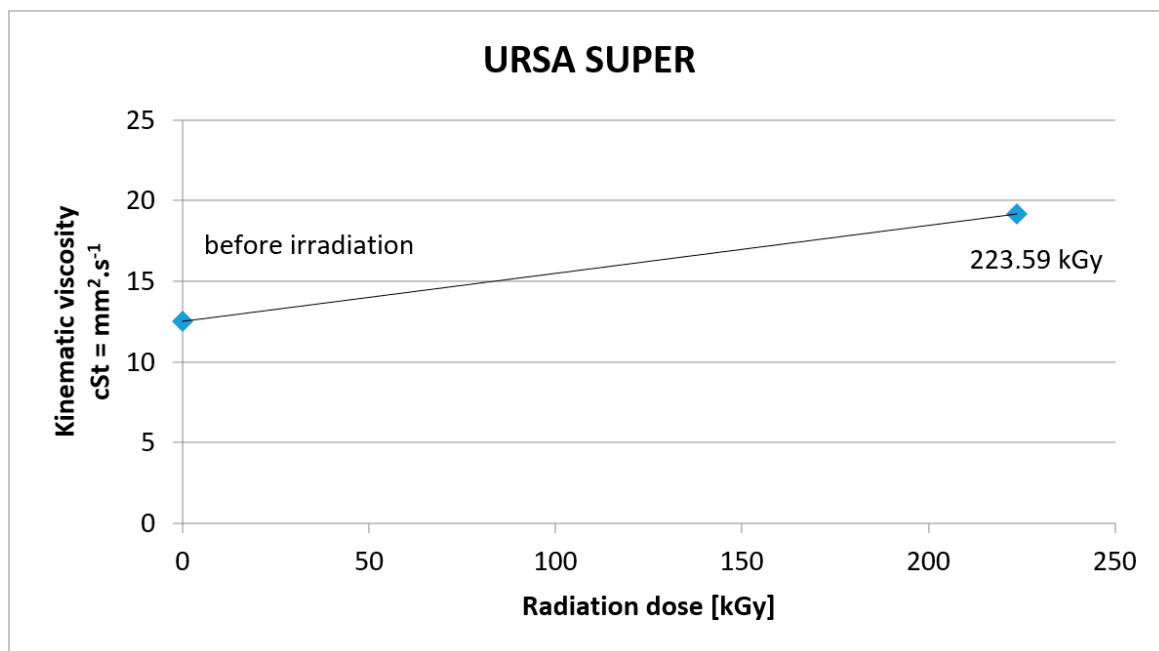


Figure 4. Increase of kinematic viscosity at high dose of irradiation.

According to the experimental results, the kinematic viscosity of the base oil did not change much for the two samples, as shown in Figures 5 and 6.

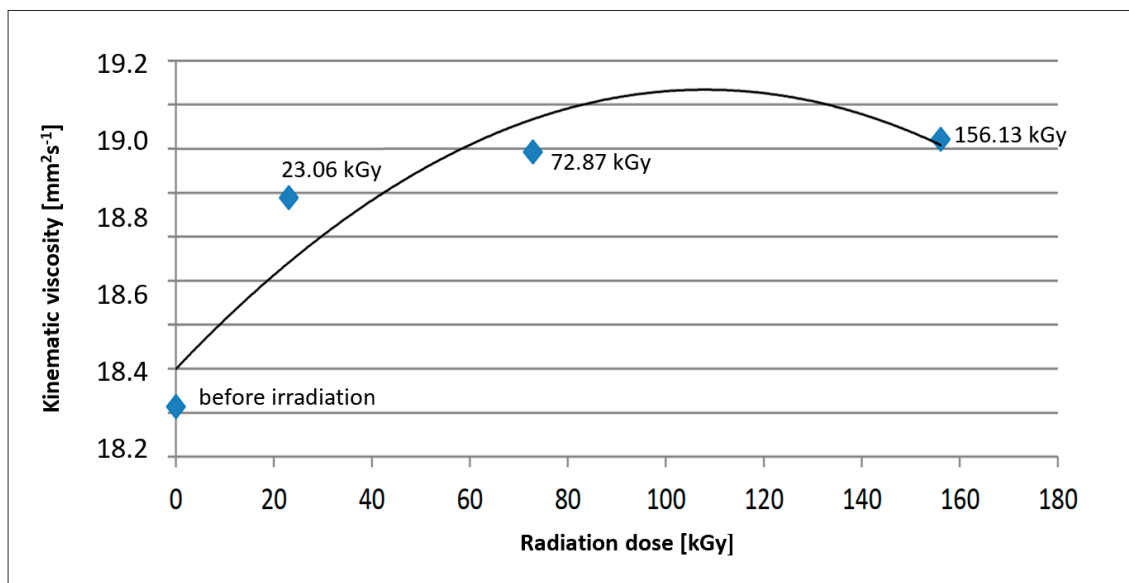


Figure 5. Change in kinematic viscosity of ZOT600 base oil due to irradiation.

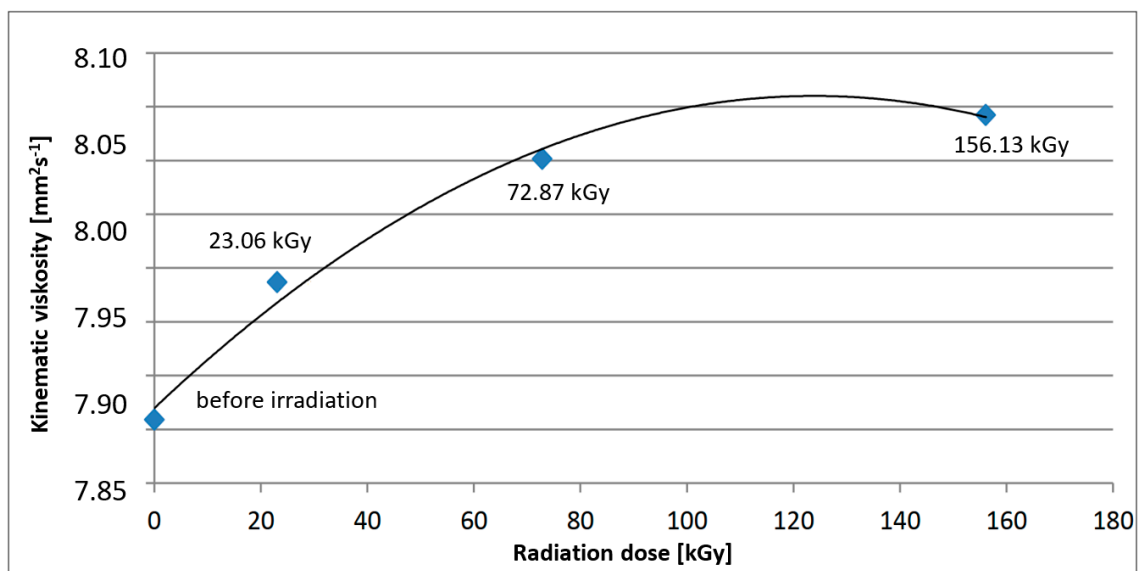


Figure 6. Change in kinematic viscosity of ZOT160 base oil due to irradiation.

The hypothesis that additives have a significant effect on the functional properties of oils after irradiation has not been confirmed, which leads the authors to state that it is sufficient to test finished oils with a package of additives in the future. Figure 7 shows the change in the coefficient of friction of the piston system (with ceramic coating SiO_2)—unirradiated and irradiated base oil using Ball-on-flat analysis (first irradiation dose 23.06 kGy and second irradiation dose 72.87 kGy).

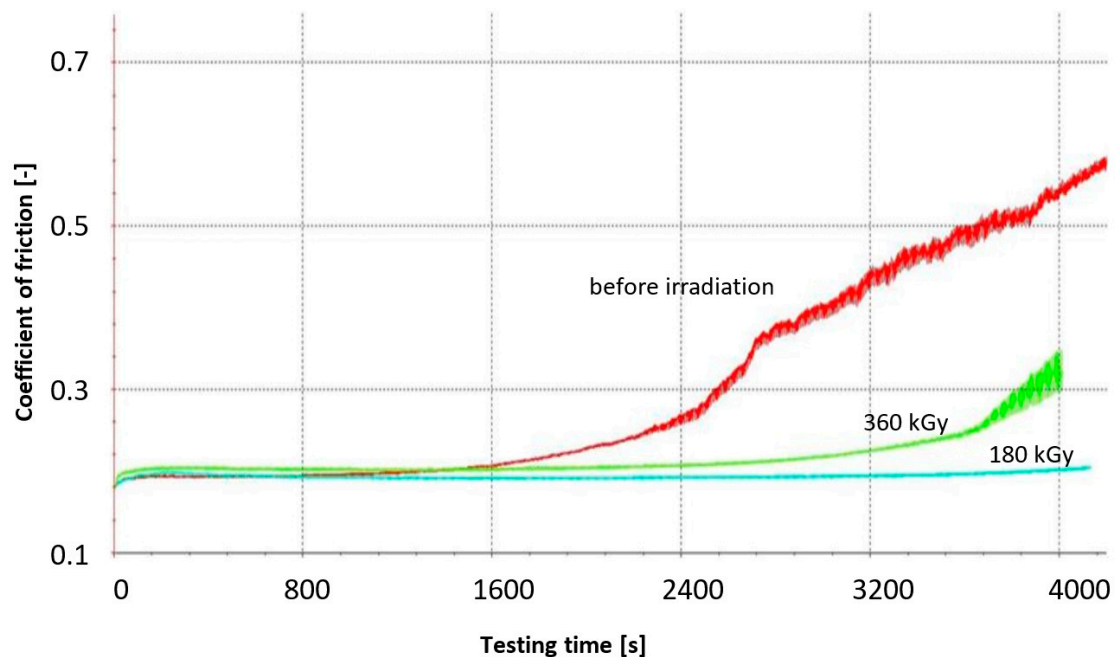


Figure 7. Change of the coefficient of friction of the base oil and piston system with SiO₂ coating due to irradiation by Ball-on-flat analysis.

5. Conclusions

The presented pilot research was focused on changes in the kinematic viscosity of selected lubricated medium, commonly used in the automotive industry, due to the irradiation by accelerated high-energy electrons. Selected new and used oils were irradiated, tested, and analyzed. The samples were irradiated with different doses, while maintaining the same physical parameters of the linear electron accelerator, while a relatively wide dose interval was chosen to increase the probability of finding the area of change of the observed phenomenon. The results show that almost in all samples the kinematic viscosity decreases due to high-energy ionizing radiation, proving the existence of degradation processes [4,15,16]. Only with one sample of mineral oil crosslinking was observed at the highest dose of irradiation. The result was a significant increase in kinematic viscosity [7,17,18].

Additional experiments and analysis have shown that the irradiation of oils with additives and base oils without additives does not change significantly. It is also assumed that base oils from different manufacturers are similar. On the other hand, there is a real assumption that base oils are very similar. The contents of these additive packages were not detected. On the contrary, a chemical analysis was performed for each irradiated sample, but the results are subject to anticipated industrial protection and for these reasons publications cannot yet be published. Experiments are currently underway to verify changes in the coefficient of friction of different frictional metallic materials, different lubricating media depending on the radiation dose. The research provides basic preconditions for a comprehensive analysis of changes in the basic properties of operating fluids used in the automotive industry due to ionizing radiation. The expected future research will focus on the exploration of other lubricating oil parameters (TBN/alkaline reserve, shear stability, oxidation products, etc.).

Author Contributions: Conceptualization, J.S.; methodology, J.S.; validation, P.S., formal analysis, P.S.; investigation, J.S.; resources, J.S.; data curation, P.S.; writing, J.S.; visualization, P.S.; supervision, P.S.; project administration, J.S. All authors have read and agreed to the published version of the manuscript.

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