

Article

Compatibility of Different Automotive Elastomers in Paraffinic Diesel Fuel

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Abstract: The introduction of new fuels to power internal combustion engines requires testing the compatibility of such fuels with materials commonly used in fuel supply systems. This paper investigates the influence of alternative fuels on the acrylonitrile-butadiene rubber and fluoroelastomer used in the automotive industry. In the study, conventional diesel fuel, its blend with 7% of fatty acid methyl esters and paraffinic diesel fuel produced with the Fisher Tropsch synthesis from natural gas were interacted with the elastomers. The immersion tests were carried out at room temperature (20 °C) for 168 h. The effect was evaluated based on changes in the selected rubber's volume, mass and hardness. It has been confirmed that the synthetic component without aromatic hydrocarbons had a different effect on the tested rubber than did conventional fuel. In follow-up work, the selected rubbers were also subjected to microscopic observation. The most frequently observed effect was the washing out of the seal protective layer.



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1. Introduction

The basic units powering means of transport and the equipment used for producing energy in the construction, agricultural and other sectors are diesel engines. Their widespread application is the result of their high effectiveness, economic efficiency and reliability [1]. The primary fuel used to power these engines is diesel fuel, which is composed of hydrocarbons obtained from conservative or destructive crude oil processing. Diesel fuel distillates exhibit a boiling point range of ca. 180–350 °C, however, due to the high sulfur content, it has to be removed through hydrogen treatment within catalytic processes (hydrodesulfurization) [2]. Due to the method of ignition of the fuel-air mixture in the engines, the key parameter in terms of these fuels is the ability to rapidly self-ignite, which is measured by the cetane number. Its high value is brought about by paraffin hydrocarbons [3].

Diesel fuel (diesel) is a very complex mixture containing thousands of individual compounds, most of which have a carbon atom number of 10 to 22 (C₁₀–C₂₂) [4]. Individual hydrocarbons differ in terms of carbon atom arrangement and the hydrogen to carbon ratio (H/C). The composition and mutual proportions of paraffin (straight and branched), cycloparaffin and aromatic hydrocarbons contained in diesel fuels vary depending on the used crude oil, technological production processes and regulatory requirements of the market that a product is traded on [5]. For this reason, this type of fuel is not defined by the content of individual hydrocarbons, but through regulatory requirements.

In order to minimize greenhouse gas emissions and limit the use of petroleum products, non-petroleum components are introduced into diesel fuel. These include fatty acid methyl esters (FAME) that originate from rapeseed, sunflower and soybean oil processing

and the processing of animal fats [6]. FAME can be added to diesel fuel as a fuel component. The advantages of their incorporation include biodegradability [7], better lubricating properties [8] and low emission profiles. Such components are non-toxic [9]. At the same time, it should be noted that the chemical composition of FAME is different from diesel, hence the content of FAME in commercial fuels was limited to 7% (*v/v*) (B7), while fuels containing more esters can be used in vehicles dedicated exactly for this fuel type [10]. Incorporation of this component also comes with downsides, which include less stability than fossil diesel fuel, worse low-temperature properties [11], greater hygroscopicity and higher fuel consumption. It may also have an adverse impact on natural and nitrile rubber seals [12]. Similar to conventional diesel fuel, its properties strongly depend on the raw material used for its production [13].

The introduced amendment to the Directive of the European Parliament and of the Council (EU) 2018/2001 on 11 December 2018 [14] has limited the possibility of using conventional biofuels made of raw food materials. This promotes advanced alternative fuels of inedible raw materials or waste, and will intensify the growth in the share of these components in fuels over the next decade.

However, before the EU reaches zero emissions in the passenger car sector in 2050 [15] and the replacement of diesel engines by electric motors [16] comes about, during this transition period, vehicles will be powered by alternative hydrocarbon fuels. The literature contains studies in which their authors point to the possibility of using various fuels for powering diesel engines. These include FAME (in amounts higher than the standard 7% (*v/v*)) [17], vegetable oils [18], dimethyl ether DME [19], butanol [20], pyrolysis oil [21] or synthetic fuels [22]. Some of these can only be used as a fuel component, while DME and synthetic fuels constitute complete substitutes.

Two synthetic fuels for diesel engines can be distinguished at the current moment. These are paraffinic diesel fuels obtained through the hydrogenation of fatty acids from HVO (hydrotreated vegetable oil) and through synthesis process [23], e.g., gas to liquid (GTL). Unlike other alternative fuels, synthetic fuels are composed of the same hydrocarbons as fossil fuels, albeit with a lower variety. Therein, paraffin hydrocarbons are predominant and they contain no aromatic hydrocarbons at all. Aromatic compounds are characterized by very good low-temperature properties and calorific values; however, they exhibit poor self-ignition properties. Furthermore, they have worse thermal stability, and lead to the swelling of rubber seals, hence bringing about very tight connections in the distribution and fuel supply systems [24]. The requirements for paraffinic diesel fuels are set out in the standard EN 15940, however, this standard does not supersede EN 590 [25]; therefore, these fuels, such as B30 and B100 can be used in a dedicated fleet (approved by vehicle manufacturers). The producers of synthetic components point to their normative and operating properties being equivalent to fossil fuel [26,27]. However, it should be stressed that they are characterized by lower density, which does not fall within the diesel fuel range stipulated in EN 590. The studies conducted in terms of analyzing combustion and emission characteristics confirm that paraffinic fuels do not exhibit worse properties than fossil diesel fuel [28,29].

Based on many years of experience of the automotive industry with fossil diesel, it is assumed that the materials found in fuel supply systems are compatible with it. However, the process of introducing new, alternative fuels and fuel components generates additional questions as to the direction of their impact on the materials used by a given manufacturer.

The aviation industry has already introduced seven synthetic components into fuel in the amount of up to 50% (*v/v*). These fuels can power aircraft jet engines [30]. Furthermore, the aviation industry has also implemented the “Standard practice for evaluation of new aviation turbine fuels and fuel additives” procedure, which indicates the materials (metals and non-metals) that the new fuels can safely interact with, as well as the scope of required tests. With regard to non-metals, the test involves a 28-day immersion of the material in the fuel, followed by the determination of its properties, such as volume swell, tensile, elongation, hardness or resistivity [31].

The automotive industry has not yet developed such an industry-wide guidebook, rather vehicle manufacturers individually comment on the possibility of using paraffinic fuels in their engines. Given the constant growth of the interest in using alternative fuels, the market currently offers a wide range of biofuels, with new fuels or fuel components destined to appear.

Materials commonly used in seals and O-rings in vehicles include ethylene-propylene-diene monomer (EPDM), acrylonitrile-butadiene rubber (NBR), chloroprene rubber (CR), fluoroelastomers (FKM), silicone rubber (SR) or polytetrafluoroethylene (PTFE). Please note that FKM is best suited for use as a seal in fuel supply systems of vehicles powered by diesel fuel and biodiesel [32]. However, due to the different hydrocarbon composition (its lower diversity), synthetic fuel may interact with various materials, including rubber, differently to petroleum products.

Although the compatibility of fuel with the materials used in a fuel supply system is always a key issue, the source literature does not contain many publications in the field of synthetic fuel's impact on the elastomers used in the automotive industry (GTL [33] and HVO [34]). Most publications address the impact of fuels with a FAME additive [35–39]. Their authors select fossil fuel blends with methyl esters obtained from various raw materials, which are then exposed to selected rubbers. The obtained results are used as a base to develop ranking lists showing the elastomers most and least resistant to biofuels.

The authors of [40] identified all vehicle components that contain rubbers. These are, among others, fuel pump seals, fuel gauge seals, in-tank seals, fuel filter seals, fuel injector O-rings, fuel hoses, canister seals, etc. As already mentioned, there is currently no standardized list of materials to be introduced, therefore the author of [41] selected elastomers and plastics (NBR, FKM, nylon and Teflon) and metal components (copper, zinc, mild steel, matt sheet metal, brass and aluminum) to be tested for the impact of new fuels on structural materials. In addition, he suggested blend concentrations, conditions of interaction between the fuel and material and also the test scope itself.

The objective of the paper was to verify the impact of conditional fuel without FAME and with FAME, as well as paraffinic diesel fuel on selected O-rings used in fuel supply systems. Despite the abundance of articles related to biodiesels available in the literature, there are only a few studies which are focused on paraffinic diesel fuel. In view of the possible use of paraffinic fuel in the automotive sectors, the evaluation of its compatibility with elastomers becomes required.

2. Materials and Methods

The study of the impact of test fuels on rubber seals involved the injection system components specified below. They can be found in a common rail (CR) injection diesel engine: (i) CR injector overflow nozzle O-ring 3.2×1.6 —acrylonitrile-butadiene rubber (NBR) and (ii) CR injector overflow nozzle O-ring 3.2×1.6 —fluoroelastomer (FKM).

The aforementioned elastomers have been selected because they are widely used in fuel supply systems, primarily in the fuel lines and O-rings. NBR is a product of butadiene and acrylonitrile copolymerization, wherein increasing the acrylonitrile content simultaneously increases resistance to fuel impact. In contrast, FKM is a copolymerization of monomers, the molecules of which contain fluorine. It exhibits high mechanical strength and resistance to damage, extreme temperatures and aggressive substances, including aromatic hydrocarbons.

NBR and FKM rubbers are good as seals, since they maintain their shape when exposed to working fluids and are resistant to chemical substances [42].

The following fuel samples were selected:

- Conventional diesel fuel, produced from crude oil without FAME—DF,
- Conventional diesel fuel, produced from crude oil with 7%(v/v) FAME—B7,
- Paraffinic diesel fuel, produced through synthesis from natural gas, by means of the gas to liquid process—GTL.

B7 is a commercial fuel that is widely available at service stations. Diesel is also a fuel available at service stations. Moreover, it is primarily used by the armed forces, whereas GTL was produced from natural gas through the Fischer Tropsch synthesis. Synthetic fuel production process involves three key stages. These are synthesis gas production—an organic material is converted into synthesis gas through the gasification process; synthesis—synthesis gas is converted into liquid hydrocarbons with the use of steam and a catalyst, which leads to obtaining synthetic crude oil; and finally, hydrocracking and isomerization—synthetic raw material composed mainly of n-paraffins is converted to isoparaffins and separated through distillation into individual fuel fractions (diesel, jet), depending on carbon atom content [43].

The physico-chemical properties of fuel samples were investigated and are shown in Table 1.

Table 1. Properties of tested fuels.

Property	Unit	Test Method	EN 590 Requirement	DF	Results B7	GTL
Cetane index	–	EN ISO 4264	min 46.0	56.2	53.1	84.2
Density at 15 °C	kg/m ³	EN ISO 12185	820.0–845.0	822.9	833.9	777.9
Viscosity at 40 °C	mm ² /s	EN ISO 3104	2.000–4.500	2.685	2.772	2.660
Flash point	°C	EN ISO 2719	min 55.0	58.5	62.5	70.0
Sulphur content	mg/kg	EN ISO 20846	max 10.0	6.8	6.9	<3.0
Cold filter plugging point	°C	EN 116	–10 for D grade –15 for E grade –20 for F grade	–16	–15	–21
Lubricity HFRR at 60 °C	µm	EN ISO 12156-1	max 460	413	202	476
Aromatics	% (v/v)	D1319	-	20.3	19.7	<1.0
FAME content	% (v/v)	EN 14078	max 7.0	<0.1	6.6	<0.1
Copper strip corrosion (3 h, 50 °C)	classification	EN ISO 2160	1	1a	1a	1a

It should be noted that conventional and paraffinic diesel fuels are subject to different standards wherein their properties are specified. Paraffinic fuels are characterized by very good low-temperature and self-ignition properties, which result from the high content of paraffin hydrocarbons. The fact that there are only trace amounts of sulfur in the fuel positively impacts the environment during the combustion process. As already mentioned, GTL fuel does not contain aromatic hydrocarbons at all. Although they are characterized by good calorific values, their absence has a positive impact on unburned hydrocarbon and soot emissions during the combustion process.

The properties of selected rubbers in terms of changes in mass, volume and hardness (IRHD scale) were tested in order to determine the impact of prepared fuels on selected O-rings. The following fuel-rubber interaction conditions were selected: (i) interaction time—168 h (7 days), room temperature (20 °C) and (iii) 100 mL of tested fuel in a closed, glass container.

Percentage of mass change was calculated from:

$$\Delta M = \frac{(M_2 - M_1)}{M_1} 100 [\%] \quad (1)$$

where M_1 is the initial O-ring mass in air [g] and M_2 is the O-ring mass in air after immersion [g].

Percentage volume change was calculated from [44]:

$$\Delta V = \frac{(M_2 - M_4) - (M_1 - M_3)}{(M_1 - M_3)} 100 [\%] \quad (2)$$

where M_3 is the initial O-ring mass in water [g] and M_4 is the O-ring mass in water after immersion [g].

Each O-ring was measured 7 times, providing an arithmetic mean, later adopted as the measurement result. The measurement error for mass and volume was 0.5% of the measured value, and 0.4% for hardness. In the presented figures, the vertical error bars correspond to the measurement uncertainty.

The last stage of the research involved microscopic observations. Microscopic methods are commonly used to visualize the surface condition of polymeric materials. It is no different with the characteristics of rubbers exposed to fuels. A dedicated microscope is used depending on the needs and the order of magnitude of the studied changes and objects. Examples include the application of electron microscopy (e.g., SEM) and atomic force microscopy (AFM) to identify material and structural changes on a nanometric [45] and micrometric [46] scale, or the employment of optical microscopes for micro-scale surface imaging [24,47].

The observations were conducted by means of a Nikon Eclipse LV100ND optical microscope with a Nikon DS-Ri2i camera and NIS-Elements BR software. Microscopic imaging was undertaken using the bright field (BF) technology. The following magnifications were involved during the analysis: 10×10 – $200 \mu\text{m}$ scale, 10×20 – $100 \mu\text{m}$ scale, 10×50 – $50 \mu\text{m}$ scale. Prior to the observations, each seal was purged with dry air in order to remove potential contaminations, followed by rinsing with 96% ethanol of analytical grade and being left to dry.

3. Results and Discussion

3.1. Properties of Rubbers

A natural phenomenon occurring in the course of a fuel interacting with rubber compounds is their swelling, which causes the formation of a tight connection between fuel supply system elements. The change in the volume of conventional fuels depends, among others, on the aromatic hydrocarbon content in the fuel composition. With the regard to the use of synthetic hydrocarbons as fuel (without blending with a petroleum component), such fuels are free from aromatic compounds, indicating that rubber seals will not ensure sufficient tightness, leading to fuel leakage. The EN 590 standard only limits the content of polycyclic aromatic hydrocarbons to 8% (m/m), when the standard defining jet fuel properties [48] stipulate the aromatic hydrocarbon content range at 8–25% (v/v). However, the lower value is determined only to ensure adequate rubber swelling in order to avoid potential leakage from fuel and distribution systems.

Figure 1 shows the change in the volume of O-rings made from two types of rubber. The impact of NBR rubber used in the automotive industry on diesel fuel leads to a volume change of approx. 5%. Adding methyl esters slightly increases elastomer volume relative to diesel fuel. The GTL synthetic component (which does not contain aromatic hydrocarbons) causes virtually no changes to the rubber ester volume. Furthermore, it can be concluded that KFM rubber is less susceptible to volume changes resulting from contact with fuel.

The impact of petroleum fuels on rubber compounds also leads to their increased mass (Figure 2), however, this occurs at a lower rate than the volume change. Synthetic components free of aromatic hydrocarbons virtually do not react with rubber compounds. Generally speaking, the increase in the mass and volume is associated with elastomer swelling, which is induced by absorbing fuel and polymer chain relaxation and is related to the affinity between the fuel and the elastomer.

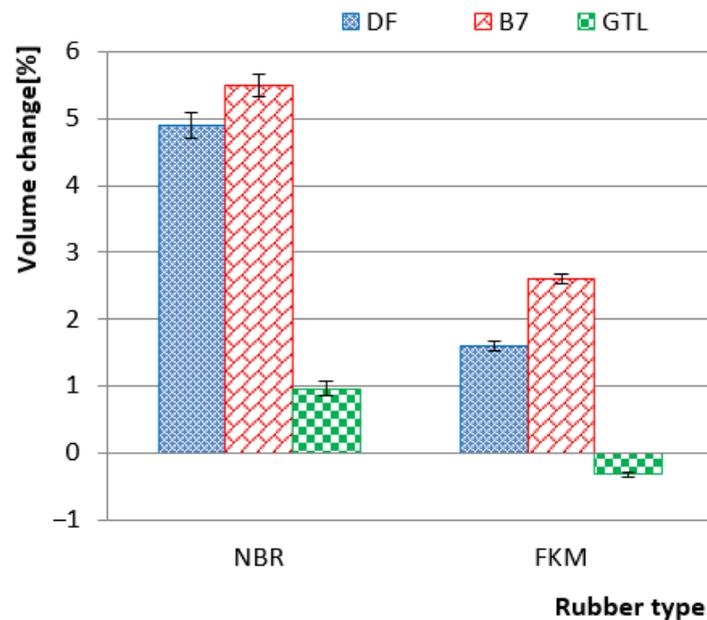


Figure 1. Volume change in different elastomers.

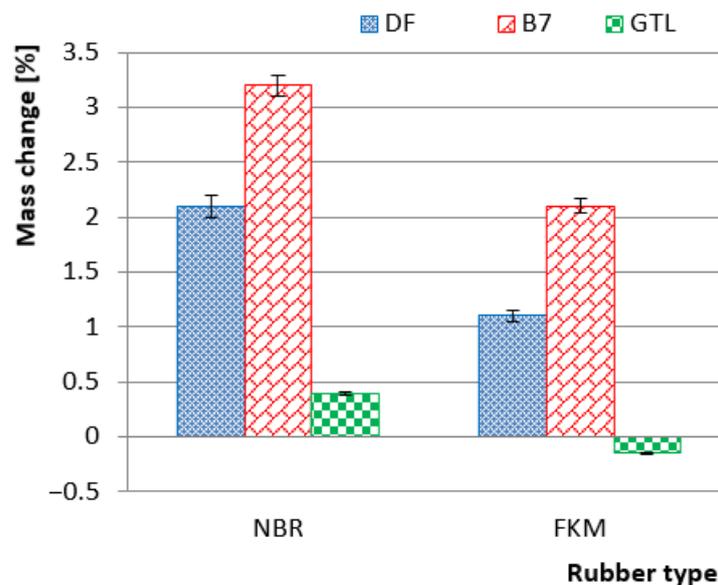


Figure 2. Mass change in different elastomers.

The interaction of all fuel samples with elastomers resulted in a decrease in elastomer hardness (Figure 3). This phenomenon is caused by the fact that hardening agents are added to elastomers in order to improve elastomer properties. This leads to cross-linking between their chains. After immersion in fuel, these agents dissolve, which results in decreased seal hardness.

3.2. Surface Morphology

Selected photos of the studied seal surfaces, prior to and after exposure to the fuel blends are presented below. The surface of an NBR seal with visible post-factory defects (minor decrements and cracks) is shown in Figure 4a. This is a reference surface, used to compare changes in samples immersed in DF, B7 and GTL fuels. A characteristic coat can be observed on its surface. Upon application of the bright field (BF) technique, this stands out due to its slightly opalescent bright color. It is a permanent element of the studied surface. In the experiment, the layer was changed by contact with the fuels. The surface of

the seal upon having been soaked in the various fuels became duller. A significant surface change is the drying effect, which is manifested by increased share of grooves and creases on the seal surface (Figure 4b,c), with the greatest changes identified after exposure to GTL (Figure 4d).

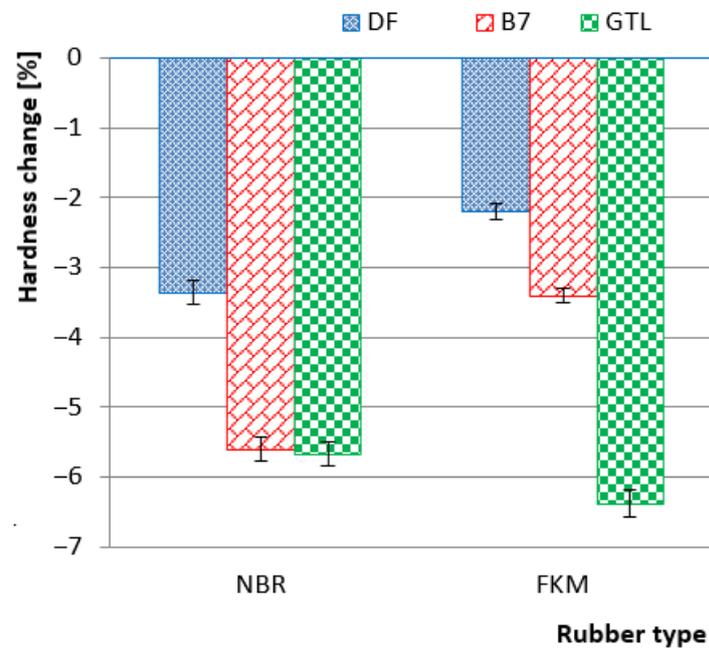


Figure 3. Hardness change in different elastomers.

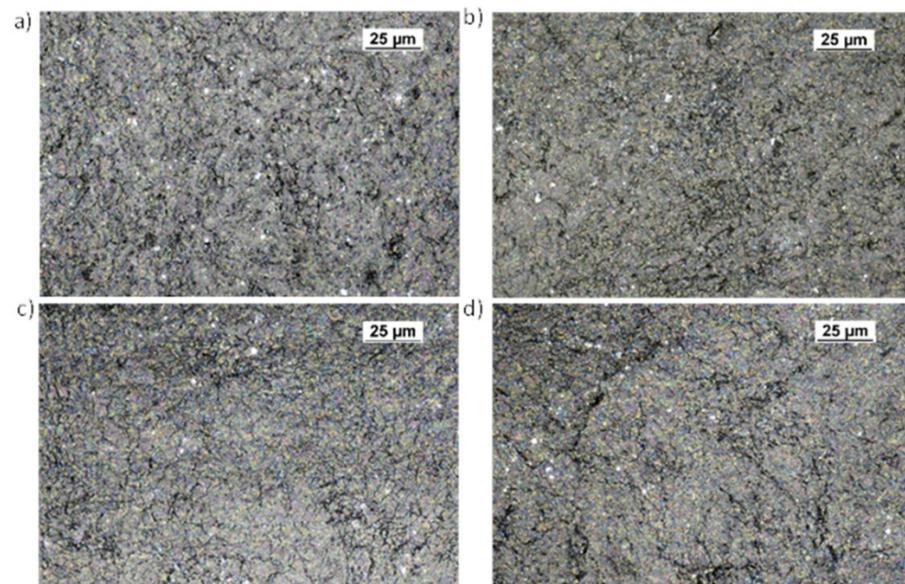


Figure 4. NBR surface, prior to (a) and after exposing to: (b) diesel, (c) B7 and (d) GTL; 10×50 magnification.

The analysis of FKM seal surfaces indicated that the samples have a layer that is characterized by the presence of numerous flake-shaped objects. They probably originate from the seal manufacturing process. The flake-shaped objects are probably the consequence of partial rubber foaming. Moreover, the outer layer had a tendency to wear off—resulting in the numerous foil flakes observed on the sample surface (Figure 5a). Another permanent element of rubber not exposed to fuels is the black elements that contrast with the light mass. There are the dye components and fillers used in rubber production (red arrows

on Figure 5a–c). They are statistically distributed and no direct interaction with fuel was identified, due to their location in the deeper rubber layers.

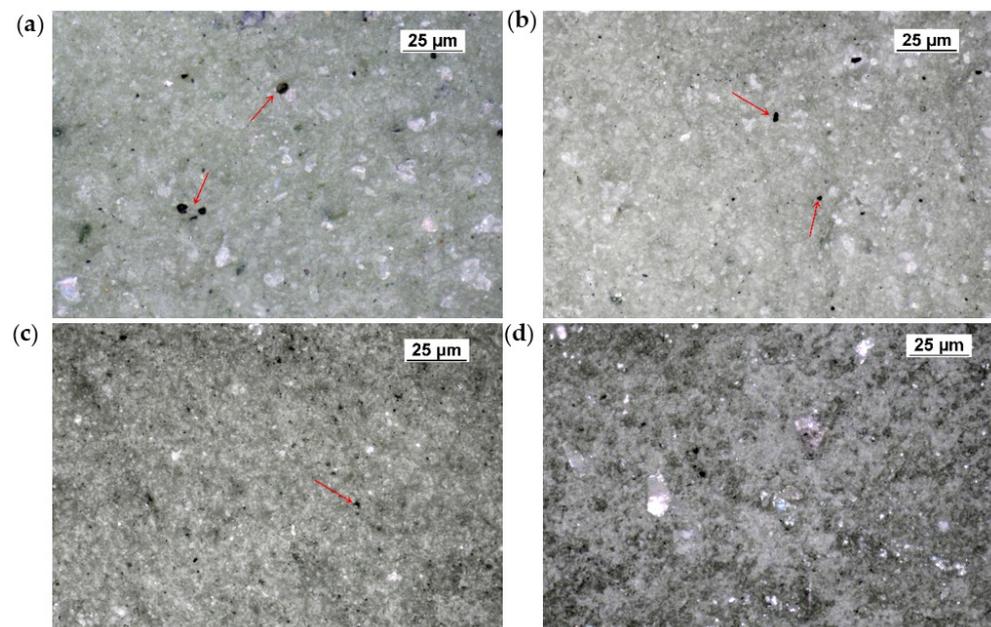


Figure 5. FKM surface, prior to (a) and after exposing to: (b) diesel, (c) B7 and (d) GTL; 10×50 magnification.

The tested fuels demonstrated a direct impact upon the sample surface. Herein, the thin, shiny layer was washed out, leading to surface dulling (Figure 5b,c). The greatest surface changes were observed on the seal exposed to GTL fuel (Figure 5d). Grooves on the surface were more visible as darker areas in the photo.

4. Conclusions

This work concentrated on the impact of diesel fuel without FAME, with FAME and paraffinic diesel derived from synthesis process on the selected rubber seals used in fuel supply systems.

Studies involving O-ring properties confirmed that the use of fuels with FAME did not adversely impact the elastomers, with the direction of changes similar to the changes caused by fossil diesel. In contrast, the paraffinic diesel fuel exhibited a different change direction, since it virtually did not lead to changes in O-ring volume and mass, which may translate to insufficient sealing of the components and leaks in distribution and fuel supply systems.

Microscopic surface tests demonstrate a negligible impact of DF, B7 and GTL fuels on NBR and FKM seals. The impact of the studied fuel blends on the surface of NBR seals is of a similar character and leads to similar changes. Seal surfaces undergo changes that are manifested by surface dullness, probably due to the washing out of the protective and preservation layer, which is left over from the industrial process. Removing this layer means that the creases and grooves on the rubber surface become more visible. The observed changes apply to all studied fuels. The changes observed on FKM seals are of a different nature. In this case, the post-factory grooves and defects are not distinguishing elements and occur to a smaller extent. The fact that draws attention is the shiny, thin surface layer, which easily undergoes mechanical damage, which leads to the formation of small rubber fragments resembling foil. The flakes observed on the surface are not abundant, which means they may have been washed out or swollen.

The study demonstrated a greater impact of the GTL on the FKM rubber surface—in the case of NBR, surface degradation is much weaker. In the future, other research

techniques will be considered, including SEM techniques for more accurate imaging of elastomer surfaces.

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