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# Compatibility of Sealing Materials with Biofuels and Biodiesel Heating Oil Blends at Different Temperatures

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## Abstract

Biofuels including ethanol and biodiesel (fatty acid methyl ester, FAME) represent an important renewable fuel alternative to petroleum-derived transport fuels. Increasing biofuels use would bring some benefits, such as a reduction in oil demands and greenhouse gas emissions, and an improvement in air quality. Materials compatibility is a major concern whenever the fuel composition is changed in a fuel system.

The objective of this research was to determine the resistance of frequently used sealing materials such as CR (chloroprene rubber), CSM (chlorosulfonated polyethylene), EPDM (ethylene-propylene-diene rubber), FKM (fluorocarbon ruber), FVMQ (methyl-fluorosilicone rubber), IIR (butyl rubber), NBR (acrylonitrile-butadiene rubber), PA (polyamides), PUR (polyester urethane rubber) and VMQ (methyl-vinyl-silicone rubber), in heating oil with admixtures of biogenic sources such as E10 (fuel with max. 10% ethanol), E85 (fuel with 85% ethanol), non-aged and aged biodiesel, diesel fuel with 5% biodiesel, non-aged and aged B10 (heating oil with 10% biodiesel) at 20°C, 40°C and 70°C. Mass, tensile strength and breaking elongation of the test specimens were determined before and after the exposure for 84 days in the fuels. The visual examination of some elastomer test specimens clearly showed the great volume increase until break or partial dissolution. Shore hardness A and D (for PA) were determined before and after exposure of the test specimens in the biofuels for 42 days.

There is not determined a threshold for the reduction in tensile properties and Shore hardness in the international standards. Therefore, a threshold of 15% was set for the evaluation of the compatibility. The sealing materials CR, CSM, EPDM, IIR and NBR were generally not resistant to biodiesel and B10. In summary, it can be therefore stated that the chemical resistance of the fluoropolymers FKM and FVMQ in fuels and biofuels is the best one.

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Compatibility, Change in mass, Change in tensile properties, Change in Shore hardness, E10, E85, Fatty acid methyl ester (FAME), Heating oil with 10% Biodiesel (B10)

## Introduction

Biofuels including ethanol and biodiesel, FAME (fatty acid methyl ester), represent a renewable fuel alternative to petroleum-derived transport fuels. Increasing biofuel use would bring some benefits, such as a reduction in oil demand and greenhouse gas emissions, and an improvement in air quality. The relevance of biofuel production strongly depends on the respective potential in different countries, i.e. the availability of land or the usability of residues and by-products of important agricultural products. Furthermore, there is a significant technical potential for further development of biofuels as a renewable energy source [1].

Ethanol has become one of the main fuel components as it complies with environmental regulations. It is produced from carbohydrates such as sugar cane, sugar beet and corn. Ethanol-gasoline mixtures are commonly used in vehicles designed to run on gasoline thus the modification of some vehicle components is required as properties of the mixture are different from those of gasoline [2].

Biodiesel is manufactured from oilseeds, predominantly rapeseed, oil palm and soy. Differences between diesel and biodiesel are due to their different chemical nature. Diesel is composed of hundreds of compounds which boil at different temperatures, while biodiesel contains a few compounds, primarily the $C_{16}$-$C_{18}$ carbon chain, depending on the vegetable oil [3,4].

Changes in fuel composition and the introduction of alternative fuels often create problems of corrosion and degradation in materials. Polymeric materials can suffer from damage or degradation due to the use of ethanol-blended fuels, which is attributable to the absorption of oxygenated hydrocarbons that mainly cause swelling. The amount of swelling depends on the nature of the solvent and the polymer. Degradation processes are irreversible, producing changes in the elastomer because of the loss of its chemical structure. Besides the tendency of elastomers to swell, contact with ethanol may also alter the tensile strength and breaking elongation, causing weakening, cracking, leakage and brittle behavior [5].

The polarity of biodiesel increases its solvency and facilitates permeation and extraction. Solvation, swelling and/or extraction lead to changes in the physical properties. Extraction alters the fuel chemistry. These chemical changes could also accelerate the degradation (hydrolysis and oxidation) of the polymeric material with the loss of additives and stabilizers [6,7].

The aim of this work was to determine the physical and chemical behavior of different frequently used sealing materials (CR, CSM,

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EPDM, FKM, FVMQ, IIR, NBR, PA, PUR and VMQ) in biofuels such as E10 (fuel with max. 10% ethanol), E85 (fuel with 85% ethanol), non-aged and aged biodiesel, diesel fuel with 5% biodiesel, non-aged and aged B10 (heating oil with 10% biodiesel) at 20°C, 40°C and 70°C. First results of the exposure tests at 20°C, 40°C and 70°C were published in papers for CORROSION 2014, CORROSION 2015, CORROSION 2016, and CORROSION 2017 [8-11].

## Materials and Method

### Preparation of test specimens

Vulcanized rubber plates of CR, CSM, EPDM, FKM, FVMQ, IIR, NBR, PA, PUR and VMQ were used for the exposure tests. At least five test specimens of each polymer were cut out of the plates with the following dimensions according to DIN 53504 [12], Figure 1.

Total length: 110 mm

Breadth at the end: 12.5 mm

Length of the narrow parallel part: 25 mm

Breadth of the narrow parallel part: 4 mm

### Exposure tests in fuels

Five tensile test specimens of each sealing material were exposed to E10, E85, non-aged and aged biodiesel, diesel fuel with 5% biodiesel, non-aged and aged B10 at 20°C, 40°C and 70°C for 84 days in a specimen jar (diameter: 100 mm, height: 200 mm) with a sufficiently tight-fitting lid according to ISO 1817 [13]. The biodiesel was produced from rapeseed. The test specimens were fully immersed in the biofuels, Figure 1. Three circular test specimens of each sealing material for the determination of the Shore hardness were exposed for 42 days to the fuels at 20°C, 40°C and 70°C.

### Determination of change in mass and visual detection

The tensile test and the Shore hardness specimens were weighed before and after exposure to the biofuels at the standard laboratory temperature of 23°C and the standard laboratory humidity of 50 ± 10%. After exposure, the test specimens were inspected visually to detect damage such as greater swelling and volume change, as shown in Figure 1.

### Determination of tensile properties

The tensile properties (tensile strength and breaking elongation) were determined according to DIN 53504 before and immediately after exposure at the standard laboratory temperature of 23°C and the standard laboratory humidity of 50 ± 10% [12].

### Determination of shore hardness A and D

The Shore Hardness A and D (for polyamide) was determined before and after exposure to the biofuels according to DIN 53505 at the standard laboratory temperature of 23°C and the standard laboratory humidity of 50 ± 10% [14].

## Results and Discussion

### Change in mass, tensile properties and Shore hardness in E10

The immersion tests of elastomers in E10 and E85 at 60°C for 20 hours performed by Kass et al. led to the conclusion that the maximum swelling occurred at a content of 17-25% in the fuel for the fluorocarbon specimens. Fluorosilicone rubber achieved maximum swelling at 10% ethanol. The tests showed that NBR test specimens were less soluble in gasoline blends containing high levels of ethanol such as E85. Higher swelling was observed for gasoline blends with 10-17% ethanol [15].

The results of the present work confirmed the swelling of fluorocarbon specimens in E10 and the deterioration of NBR specimens at 70°C as shown in Figure 2. The measured weight gain of the test specimens caused by swelling was in the range of 2% (PA) to 83% (VMQ) at 20°C, of 7% (PA) to 88% (VMQ) at 40°C and of 2% (IIR) to 103% (VMQ) at 70°C. The lowest weight gain was measured for FKM, FVMQ and PA. A deterioration of NBR and CR test specimens was observed at 70°C.


Figure 1: Specimen jar for the exposure tests (left) and IIR tensile test specimens before and after exposure in diesel fuel with 5 % biodiesel at 40 °C (right).

Swelling influenced the tensile properties. Loss in tensile strength and breaking elongation was lower at 20°C and 40°C than at 70°C, as shown in Figure 3. Tensile strength decreased by 6% (VMQ) to 69% (CR) at 20°C, by 8% (VMQ) to 100% (CR) at 40°C and by 16% (VMQ) to 100% (NBR, CR) at 70°C. Breaking elongation decreased by 1% EPDM to 17% (CR) at 20°C, by 1% (EPDM) to 100% (CR) at 40°C and by 15% (EPDM) to 100% (CR, NBR, PA) at 70°C. The lowest decrease in tensile properties was determined for FKM, EPDM, VMQ and IIR, and the highest for NBR, CSM and CR at the three test temperatures. There is not determined a threshold for the reduction in tensile properties and Shore hardness in the international standards. Therefore, a threshold of 15% was set for the evaluation of the compatibility. Based on this threshold of 15% FKM, FMVQ, CSM, EPDM, NBR, VMQ and PA were evaluated as resistant in E10 at 20°C, FKM, FVMQ, EPDM and PA at 40 °C, and none of the materials at 70°C.

Swelling influenced the shore hardness of the test specimens too. Loss in Shore hardness increased with temperature rise to 70°C, as shown in Figure 4. Shore hardness A decreased by 3% (CR) to 36% (IIR) at 20°C, by 12% (PUR) to 59% (IIR) at 40°C and by 23% (VMQ) to 100% (NBR, CR, CSM, IIR) at 70°C. For PA was determined a decrease of Shore hardness D by 15% at 20°C, by 11% at 40°C and by 84% at 70°C. Based on a threshold of 15% for the reduction in Shore hardness FKM, FMVQ, EPDM, NBR, PA and PUR were evaluated as resistant in E10 at 20°C, FKM, FVMQ, PA and PUR at 40°C, and none of the tested materials at 70°C.

**Change in mass, tensile properties and Shore hardness in E85**

The weight gain and weight loss of the test specimens caused by swelling and separation of plasticizers in E85 were in the range of 6% (NBR) to 9% (PA) at 20°C, of 4% (EPDM) to 9% (PA) at 40°C and of 4% (CR) to 12% (NBR) at 70 °C. However, the weight gain of the fluorinated elastomers FKM and FVMQ were, together with IIR, at the lower end of this range at all test temperatures. CR test specimens lost 5% of their original weight at 20°C and 40°C, and 5% at 70°C, while IIR test specimens lost 1% of their original weight at 40°C and 70°C. The damage was attributed to peroxides which were formed by alcohol oxidizing into carboxylic acids.Weight gain and weight loss of the test specimens were lower at 20°C and 40°C than at 70°C.

Beside the tendency of elastomers to swell, contact with 85% ethanol affected the tensile properties too. Figure 5 shows that tensile strength decreased by 0.1% (FVMQ) to 25% (CSM, CR) at 20°C, by 9% (PA) to 52% (EPDM, CR) at 40 °C and by 22% (IIR) to 100% (PUR) at 70°C. Breaking elongation decreased by 0.3% (CSM) to 39% (CR) at 20°C, by 6% (VMQ) to 54% (NBR) at 40°C and by 17%


Figure 2: Specimens of FKM (left) and NBR (right) before and after exposure to E10 at 70 °C.

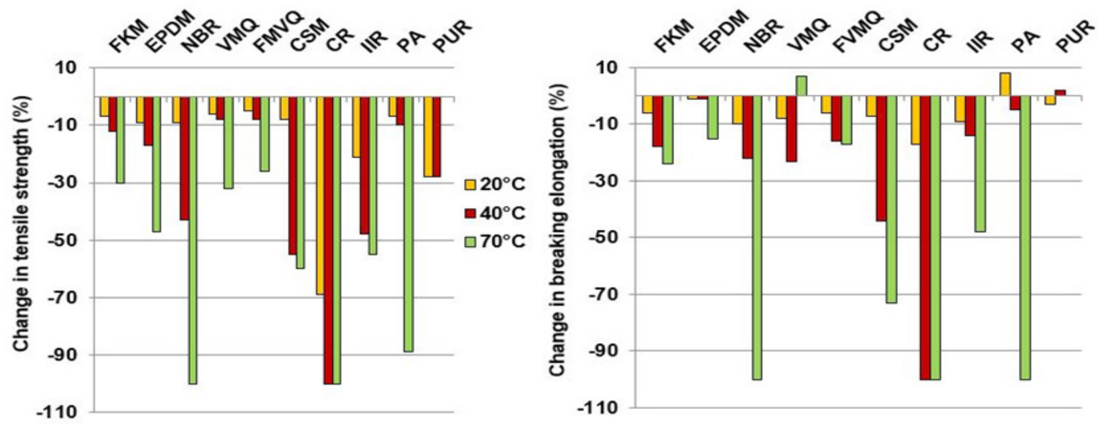


Figure 3: Change in tensile strength (left) and breaking elongation (right) of sealing materials after exposure to E10 for 84 days at 20 °C, 40 °C and 70 °C.

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(FKM) to 100% (PA, PUR) at 70°C. Loss in tensile strength and breaking elongation was lower at 20°C and 40°C than at 70°C.

Swelling influenced the shore hardness of the test specimens too. Loss in Shore hardness increased with temperature rise to 70°C, as shown in Figure 6. For Shore hardness A was determined a decrease

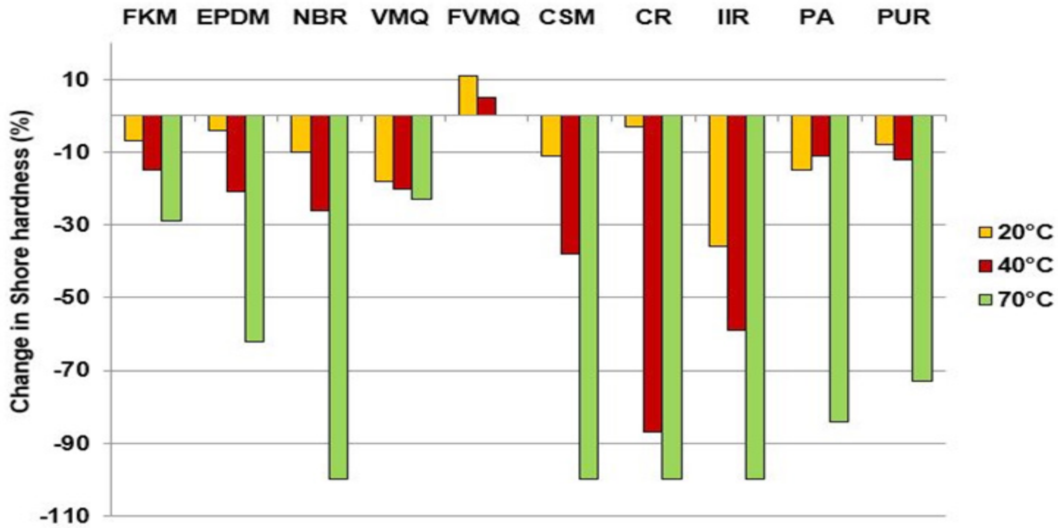


Figure 4: Change in Shore hardness of sealing materials after exposure to E10 for 84 days at 20 °C, 40 °C and 70 °C.

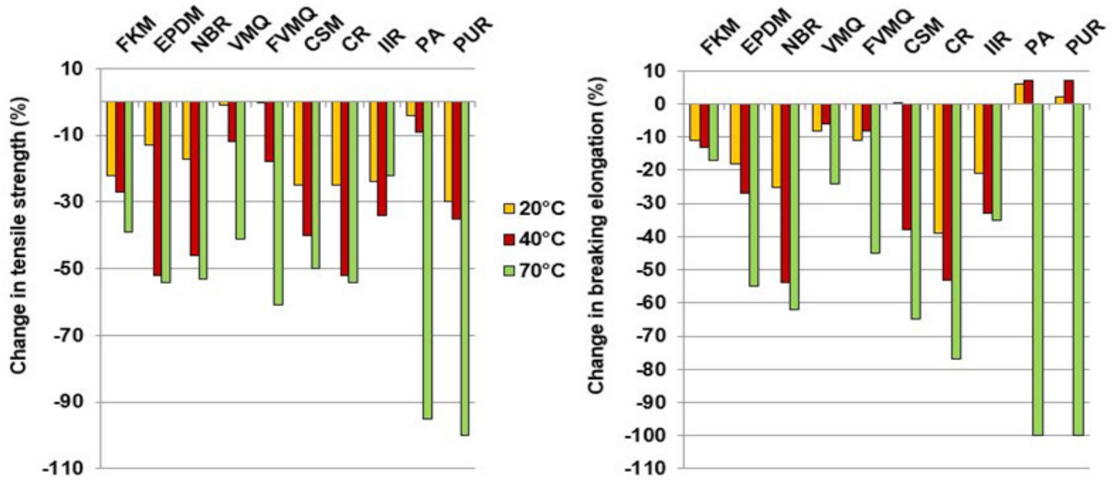


Figure 5: Change in tensile strength of sealing materials after exposure to E85 for 84 days at 20 °C, 40 °C and 70 °C.

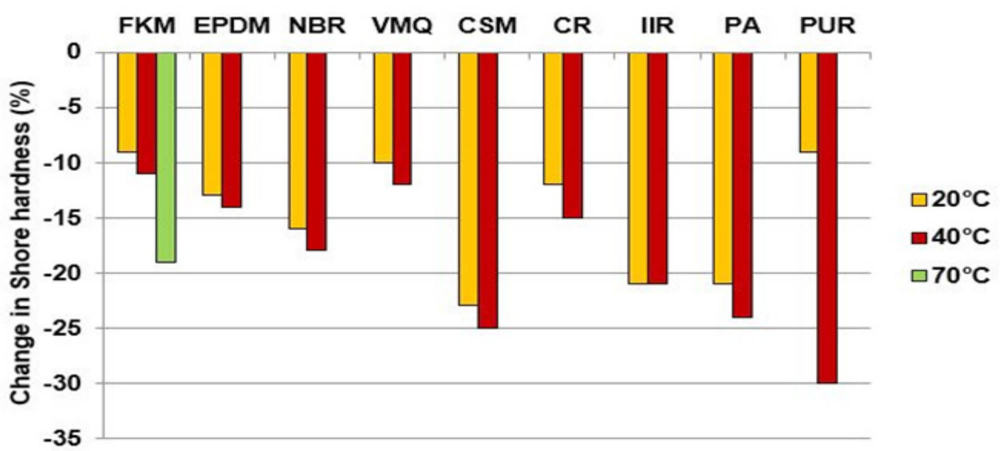


Figure 6: Change in Shore hardness of sealing materials after exposure to E85 for 84 days at 20 °C, 40 °C and 70 °C.

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by 9% (FKM, PUR) to 23% (CSM) at 20°C, by 11% (FKM) to 30% (PUR) at 40°C and by 19% (FKM) at 70°C. For PA was determined a decrease of Shore hardness D by 21% at 20°C and by 24% at 40°C. Based on a threshold of 15% for the reduction in Shore hardness FKM, EPDM, VMQ, CR and PUR were evaluated as resistant in E85 at 20°C, FKM, EPDM, CR and VMQ at 40 °C, and none of the tested materials at 70°C.

Note: the Shore hardness was not determined for FVMQ in E85.

**Change in mass, tensile properties and Shore hardness in non-aged and aged biodiesel**

When exposed to biodiesel, elastomers are affected in two ways: firstly, by absorption of liquid by the elastomers and, secondly, by dissolution of soluble components of the elastomers in the liquid medium. Swelling is the result of the high absorption by elastomers in comparison to their dissolution in the fuel. Test specimens of CR, CSM, EPDM, IIR and NBR were damaged to a high degree by swelling. The reason is their polar nature when they are dissolved in the biodiesel with its polar ester group.

The weight gain of the elastomers varied between 0.3% (FKM) and 78% (CR) in non-aged biodiesel and between 0.4% (FKM) and 108% (EPDM) in 2-year aged biodiesel. Aging of the biodiesel increased the swelling and weight gain of the sealing materials.

The tensile strength and breaking elongation were affected to a high degree. The values decreased to a large extent in the case of EPDM (100%), NBR (100%) and CR (100%) in 2-year aged biodiesel at 20°C, 40°C and 70°C. CSM lost 100 % at 40°C and 70°C, as shown in Figure 7. CR, CSM, EPDM, IIR and NBR were not resistant at all in biodiesel, independently of the age and the temperature.

FKM showed, with a loss in tensile strength and breaking elongation less than 10%, high compatibility with non-aged and 2-year aged biodiesel at 20°C and 40°C, which was attributed to the absence of polarity. FKM lost 18% of its tensile strength and 11% of its breaking elongation in non-aged biodiesel at 70°C. The reduction in tensile strength and breaking elongation was lower with 16% and 2% respectively in 2-year aged biodiesel at this temperature. Using a threshold of 15% in the reduction in tensile properties, FKM was evaluated as resistant in non-aged and 2-year aged biodiesel at all test temperatures.

The diagrams with changes in Shore hardness in Figure 8 show clearly that the elastomers CR, CSM, EPDM, IIR and NBR were not

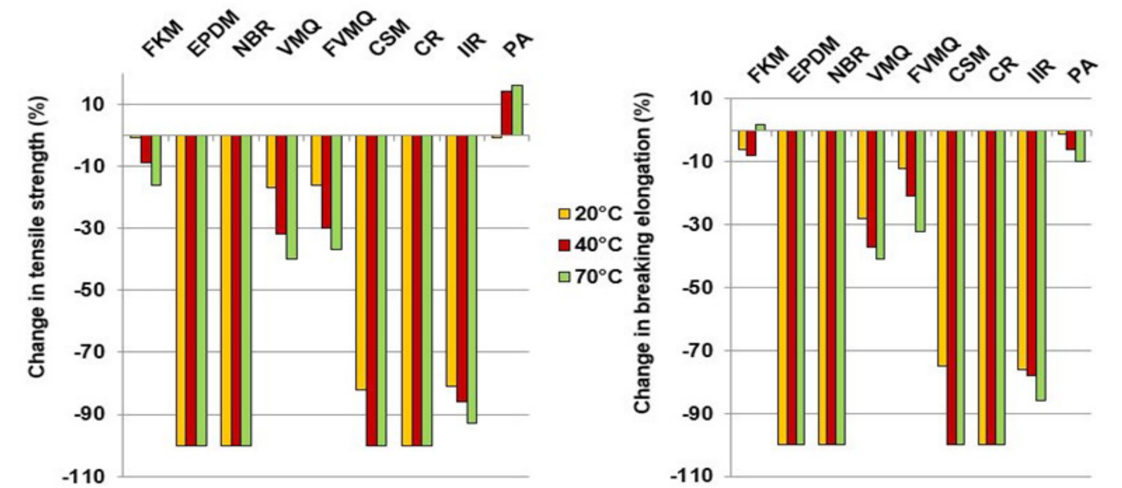


Figure 7: Change in tensile strength (left) and breaking elongation (right) of sealing materials after exposure to 2-year aged biodiesel for 84 days at 20 °C, 40 °C and 70 °C.

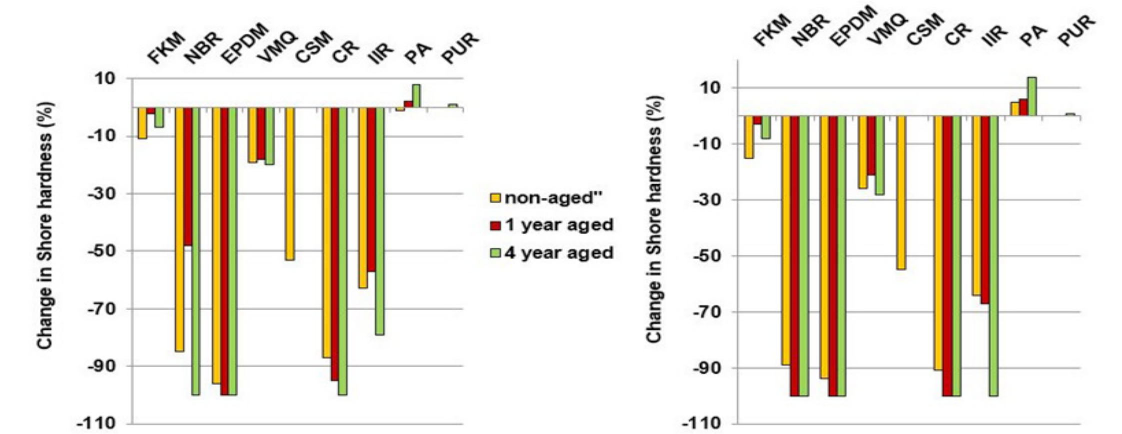


Figure 8: Change in Shore hardness of sealing materials after exposure to biodiesel of different ages at 20 °C (left) and 40 °C (right).

resistant at all in biodiesel of all ages at 20°C und 40°C. Whereas fluorocarbon rubber lost 2% of its Shore hardness at 20°C and 3% at 40°C in 1-year aged biodiesel, the Shore hardness was reduced by 7% at 20°C and by 8% at 40°C in 4-year aged biodiesel.

For polyamide was determined an increase by 2% at 20°C and 6% at 40°C in 1-year aged biodiesel, and an increase by 8% at 20°C and by 14% at 40°C in 4-year-aged biodiesel.

**Change in mass, tensile properties and Shore hardness in diesel fuel**

The weight gain of NBR test specimens caused by swelling in diesel fuel with max. 5% biodiesel at 20°C, 40°C and 70°C is shown in Figure 9.

However, the weight gain of the fluorinated elastomer FKM were, together with PUR, at the lower end of this range at all test temperatures, as shown in Figure 10. IIR and NBR test specimens showed the highest weight gain with 184% (IIR) and 32% (NBR) at 20°C, 195% (IIR) and 129% (NBR) at 40°C, and 134 % (NBR) at 70°C, while PA test specimens lost 10% of their original weight at 20°C and 40°C.

Loss in Shore hardness increased with temperature rise to 70°C, as shown in Figure 11. For Shore hardness A was determined a decrease by 1% (FKM, PUR) to 64% (CR) at 20°C, by 1.5% (FKM, PUR) to 70% (CR) at 40°C and by 12% (FKM) to 95% (NBR) at 70°C. For PA was determined an increase of Shore hardness D by 13% at 20°C and by 11% at 40°C. Based on a threshold of 15% for the reduction in Shore hardness FKM, NBR, PA and PUR were evaluated as resistant in diesel fuel with max. 5% biodiesel at 20°C, FKM, PA and PUR at 40°C, and only FKM at 70°C.

In Figure 12 is shown the influence of max. biodiesel addition to diesel fuel on the change in Shore hardness A at 40°C. The Shore hardness A of NBR test specimens was reduced after exposure to diesel fuel with max. 5% biodiesel by 18% more than in pure diesel fuel.

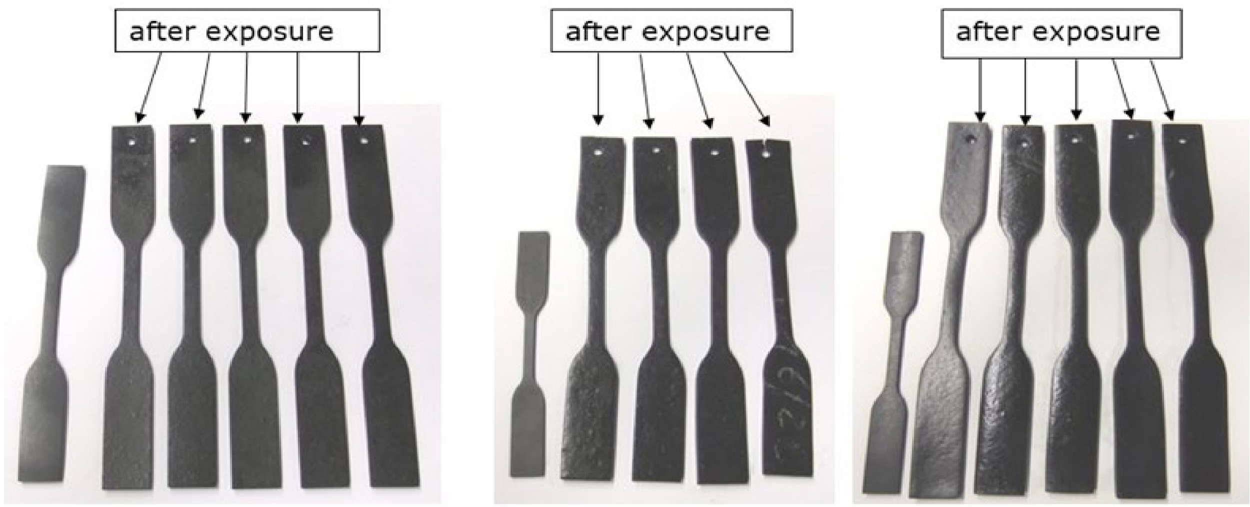

Figure 9: Specimens of NBR before and after exposure to diesel fuel with max. 5 % biodiesel at 20 °C (left), 40 °C (center) and 70 °C (right) for 84 days.

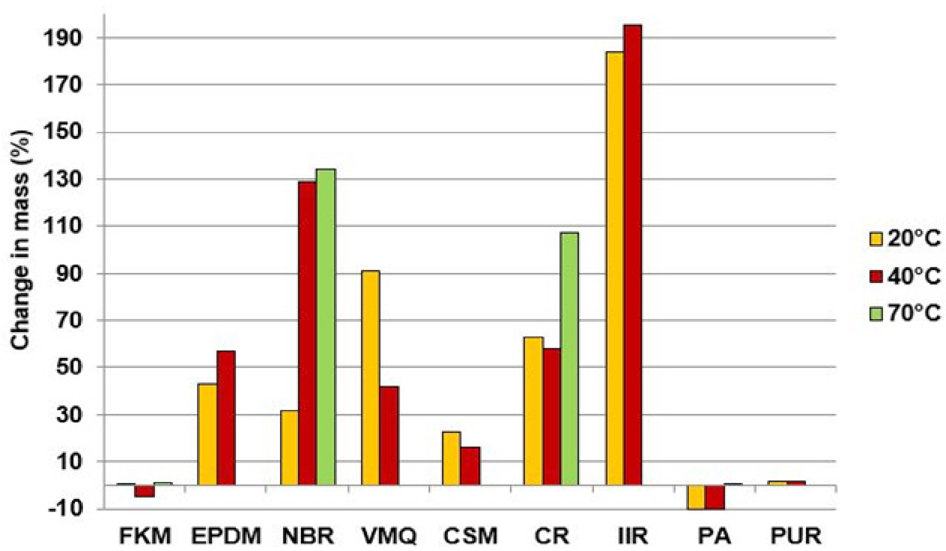

Figure 10: Change in mass of sealing materials after exposure to diesel fuel for 84 days at 20 °C, 40 °C and 70 °C.

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**Change in mass, tensile properties and Shore hardness in non-aged and aged B10**

Test specimens of CSM, CR, EPDM, IIR and NBR were damaged to a high degree by swelling in both non-aged and aged B10, which consists of 10% biodiesel (FAME) produced from rapeseed. Figure 13 shows EPDM specimens after exposure to 1-year aged B10 at 40°C and NBR specimens after exposure to 4-year aged B10 at 70°C. The highest weight at 20°C was determined for EPDM with 149% and for NBR with 102%, and at 40°C for EPDM with 140% and for NBR with 184% in 4-year aged B10. Weight gain was increased by increasing the temperature to 40°C and 70°C. The highest weight gain was


Figure 11: Change in Shore hardness of sealing materials after exposure to diesel fuel for 42 days at 20°C, 40°C and 70°C.

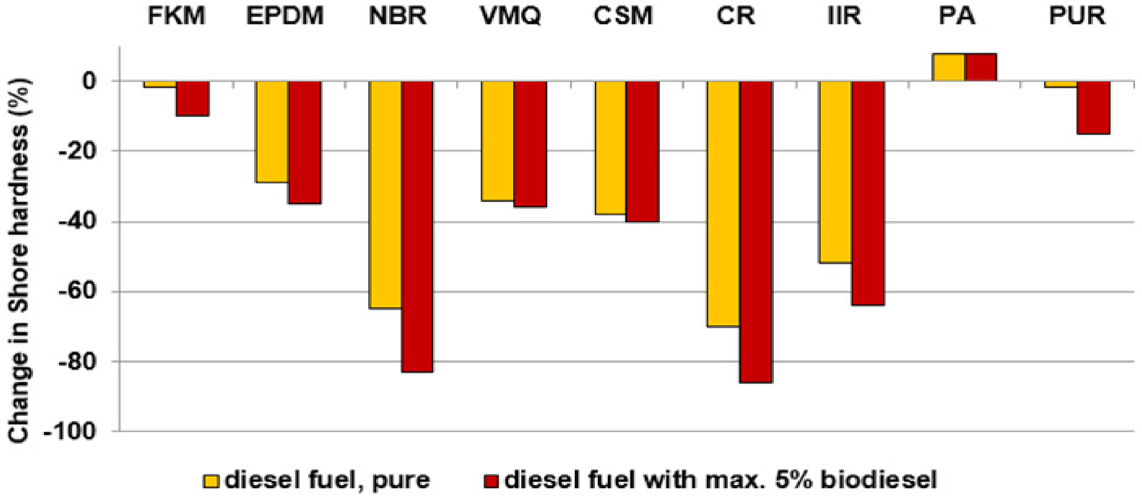


Figure 12: Comparison of the change in Shore hardness of sealing materials after exposure to pure diesel fuel and diesel fuel with max. 5% biodiesel at 40°C.

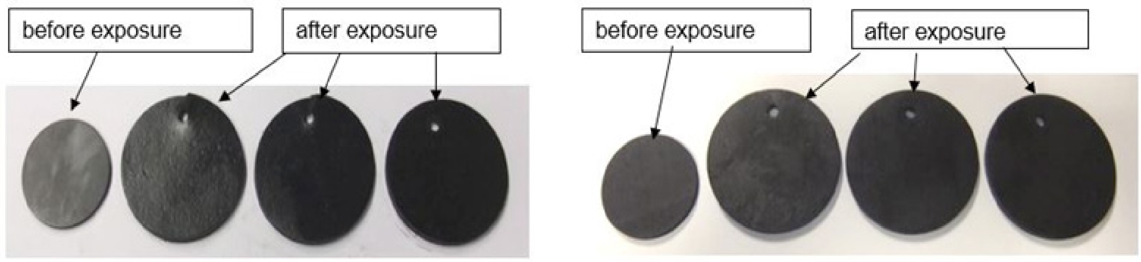


Figure 13: EPDM specimens after exposure to 1-year aged B10 at 40°C (left) and NBR specimens after exposure to 4-year aged B10 at 70°C (right).

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measured for CR with 240%, IIR with 192%, CSM with 106%, EPDM with 96% and VMQ with 54% in one-year aged B10 at 70°C, while the elastomers containing fluorine FKM (1%) and FVMQ (3%) absorbed much less B10 and swelled less.

As shown in Figure 14, the tensile strength and breaking elongation were affected to a high degree by swelling of the test specimens. The values decreased to a large extent in the case of CSM (100%), EPDM (100%) and NBR (100%) in 2-year aged B10 at 20°C, 40°C and 70°C. CSM lost 100% at 40°C and 70°C. CR, CSM, EPDM, IIR and NBR were not resistant at all in B10, independently of the age and the temperature.

Changes in Shore hardness confirmed that the elastomers CR, CSM, EPDM, IIR, NBR and VMQ were not resistant to heating oil with 10% biodiesel at all at 20°C, 40°C and 70°C, regardless of the age of the heating oil. The Shore hardness of fluorocarbon rubber specimens was reduced with the higher age of heating oil with 10% biodiesel and higher temperature. Shore hardness was reduced by 1% in one-year aged heating oil, by 4% in two-year aged heating oil, by 6% in three-year aged heating oil and by 7% in four-year aged heating oil at 20°C. The highest reduction in Shore hardness was measured with 12% in two-year aged heating oil, 13% in three-year aged heating oil and 18% in four-year aged heating oil at 70°C.

Figure 15 shows clearly the influence of 10% biodiesel on the reduction in Shore hardness of the sealing materials after exposure to standard heating oil and non-aged B10 for 42 days at 40°C. The reduction in Shore hardness is higher in B10 compared with standard heating oil.

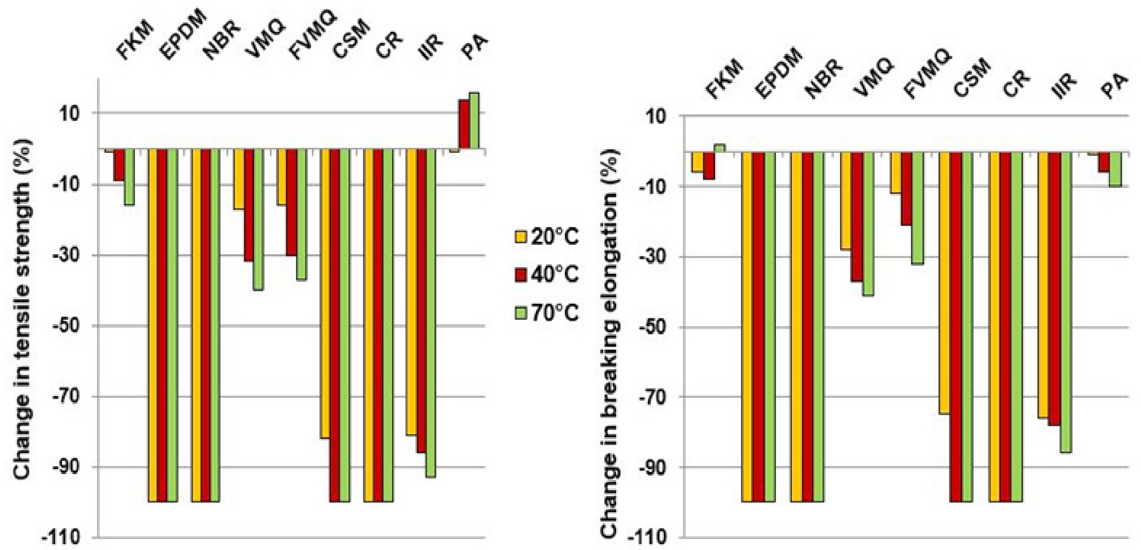


Figure 14: Change in tensile strength (left) and breaking elongation (right) of sealing materials after exposure to 2-year aged B10 for 84 days at 20°C, 40°C and 70°C.

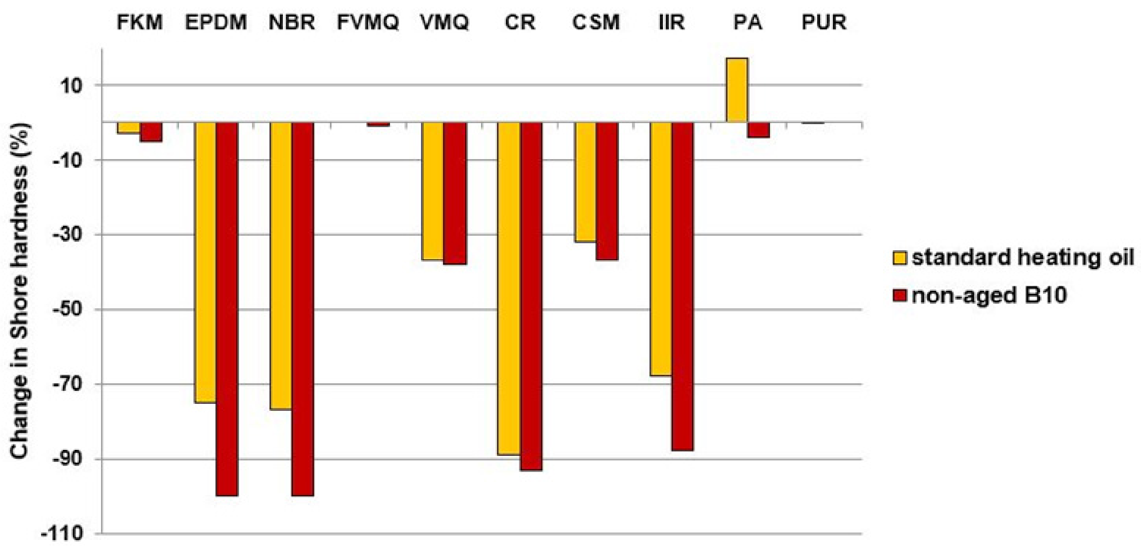


Figure 15: Comparison of the change in Shore hardness of sealing materials after exposure to standard heating oil and non-aged B10 for 42 days at 40°C.

## Conclusion

Measurements of the variations in mass, tensile properties and Shore hardness after exposure of ten different sealing materials - CR, CSM, EPDM, FKM, FVMQ, IIR,NBR, PA, PUR and VMQ- to E10, E85, diesel fuel, non-aged and aged biodiesel and B10 showed clearly that FKM and FVMQ were the most resistant material in all tested fuels up to 70°C. Damage to the materials was lower at 20°C and 40°C than at 70°C.

There is not determined a threshold for the reduction in tensile properties and Shore hardness in the international standards. Therefore, a threshold of 15% was set for the evaluation of the compatibility.

Based on this threshold FKM, FMVQ, CSM, EPDM, NBR, PA and PUR were evaluated as resistant in E10 at 20°C, FKM, FVMQ, PA and PUR 40°C, and none of the tested materials at 70°C.

E85 was less aggressive than biodiesel and B10. Using a threshold of 15% for the reduction in tensile properties the sealing materials FVMQ, VMQ and PA were evaluated as resistant in E85 at 20°C and 40°C. None of the sealing materials were evaluated as resistant in E85 at 70°C, and PA as well as PUR even started to decompose.

Biodiesel fuels are easily oxidized and contain acids and water. NBR, IIR, CR, CSM and EPDM were not resistant at all in biodiesel, independently of the age. FKM and PA showed high compatibility in biodiesel, which was attributed to the absence of polarity. The decrease in tensile properties and Shore hardness increased with the age and the temperature of the biodiesel, but the measured values were still lower than the defined threshold of 15%.

FKM, PA and PUR were evaluated as resistant in diesel fuel with at least 5% biodiesel, only FKM was resistant up to 70°C. CR, CSM, EPDM, IIR, NBR and VMQ were not resistant at all to diesel fuel at all test temperatures.

FKM and FVMQ absorbed much less non-aged and aged B10 and swelled less. CR, CSM, EPDM, IIR, NBR and VMQ were not resistant to B10 at all at 20°C, 40°C and 70°C as the decrease in the tensile properties was significantly over 50%.

FVMQ and PA could be evaluated as resistant in non-aged and aged B10 at 20°C and 40°C, whereas FKM was resistant up to 70°C. The damaging impact of non-aged B10 was higher than that of 4-year aged B10.

## Competing Interests

The author declare that there is no competing interests regarding the publication of this article.

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