TESTING SEALING COMPOUNDS FOR BIOFUELS

SURPRISING RESULTS

Biofuels present some significant challenges when specifying seals, as their chemical make-up can affect the sealing integrity of elastomers. Standard laboratory tests indicate that typical FKM and FVMQ compounds are compatible with biofuels. However, when sealing materials are tested in conditions reproducing those seen in service, it becomes evident that specialized formulations are required to ensure integrity and long service life.

Trelleborg Sealing Solutions has been working with the automotive manufacturers since biofuel technology was in its infancy. Recognizing the issues in finding the right seals for biofueling systems, it conducted in-depth research on the suitability of sealing formulations in biofuel mixtures. The company evaluated the compatibility of typical automotive biofuels for both diesel and gasoline engines, with various fuel system sealing compounds, focusing on hardness, stress-strain and volume swelling.

Standard laboratory tests suggested that typical elastomers of Fluorocarbon (FKM) and Fluorosilicone (FVMQ) were compatible with commonly used biofuels. However, when tests reproduced service conditions such as water contamination and high pressures, this was not the case.

Water contamination in biodiesel is highly probable. This can lead to nonreversible degenerative effects on certain typical FKMs. In high-pressure gasoline applications the risk of rapid decompression failure, where gas captured within the seal can cause the seal to split, is increased by the addition of ethanol. Non-optimized compounds cracked sub-surface, dramatically reduced seal integrity.

Some biofuel sealing research

• Standard test parameters

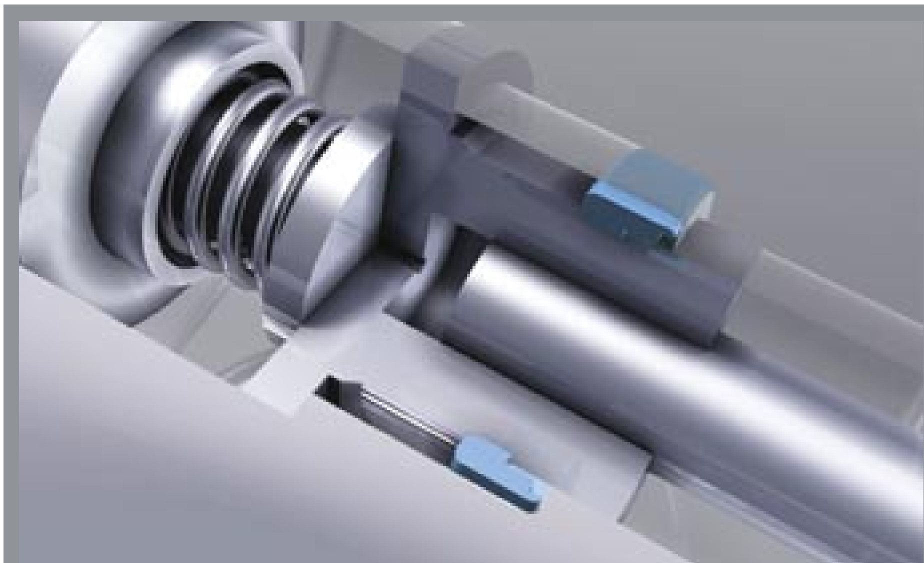
The fuels selected represented typical automotive fuels for both diesel and gasoline engines. They included:

- DIESEL - EN590
- B30 - Diesel with 30% biodiesel (RME)
- E22 - Fuel C (50% toluene and 50% isooctane) with 22% ethanol
- E85 - Fuel C (50% toluene and 50% isooctane) with 85% ethanol
- FAM B - DIN 51604 (50% toluene, 30% isooctane, 15% disobutylene and 5% methanol), 84.5% FAM A, 15% methanol, 0.5% water

A range of primarily fluorocarbon compounds along with one fluorosilicone material, all specially formulated for fuel use by Trelleborg Sealing Solutions, were evaluated. These are listed below with their 10% temperature retraction (TR10) value as an indication of low temperature performance.

• Materials evaluated

1. FVMQ (fluorosilicone), peroxide cure - Preferred application: diesel fuel connectors
2. FKM (fluorocarbon) copolymer,



A practical example of sealing in biofuel applications is this high-pressure pump. In the application, there was engine oil on the drive side and fuel on the pump side. A three-part seal in between them prevents the two from mixing. The seal has sealing lips in both directions made of (Turcon) PTFE based material along with two O-Rings in specially formulated elastomer that act as pre-stressing elements. This solution provides a good wiping effect, high resistance to wear, the ability to function at temperatures down to -40°C and stability in the presence of biofuels.

66%F, bisphenol cure (B) - Preferred application: diesel and gasoline fuel connectors

3. FKM terpolymer, 66%F, bisphenol cure (B) - Preferred application: gasoline fuel injectors
4. FKM terpolymer, 68.6% F, bisphenol cure (B) - Preferred application: diesel pumps
5. FKM terpolymer, 70% F, bisphenol cure (B) - Preferred application: diesel and gasoline fuel connectors
6. FKM tetrapolymer, 68% F, peroxide cure (P) - Preferred application: diesel pumps
7. FKM tetrapolymer, 64% F, peroxide cure (P) - Preferred application: diesel and gasoline, low pressure fuel injectors
8. FKM tetrapolymer, 64% F, peroxide cure (P) - Preferred application: gasoline, high pressure fuel injectors
9. FKM pentapolymer, 65% F, peroxide cure (P) - Preferred application: diesel and gasoline, high pressure, low temperature fuel injectors

All compounds were mixed using standard laboratory mixing equipment. Two millimeter test sheets were prepared by vulcanizing the compound blank for five minutes at 177°C. The test sheets were then post-cured in a laboratory oven, the FVMQ for four hours at 200°C, bisphenol cured FKMs for 16 hours at 250°C and peroxide cured FKMs for 16 hours at 230°C.

The compatibility of the compounds with the fuels was assessed by conducting accelerated aging under laboratory conditions. The test methods used were for hardness to ASTM D 2240, stress-strain to ASTM D 412 and fluid immersion to ASTM D 471.

Results in standard tests

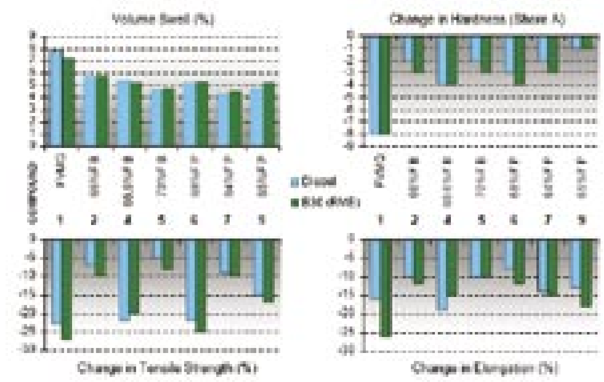
In these standard tests the volume change for all compounds in both conventional diesel and the biodiesel mix B30 (diesel with 30% biodiesel) was well under 10 percent. In addition, mechanical properties and hardness change were typical of suitably performing compounds. This data suggests that all of the elastomers tested are appropriate for use with B30, see graph 1.

The performance of the sealing compounds in gasoline and gasoline biofuel mixes was more variable. Results, shown in graph 2, demonstrate that an ethanol blend ratio of 22% (E22) is more aggressive than 85% (E85) and that Fam B is the most aggressive. Although significantly higher deterioration of properties occurred in gasoline mixes than in the biodiesel mix, most of these sealing compounds have been used in applications with alcohol containing fuels for many years.

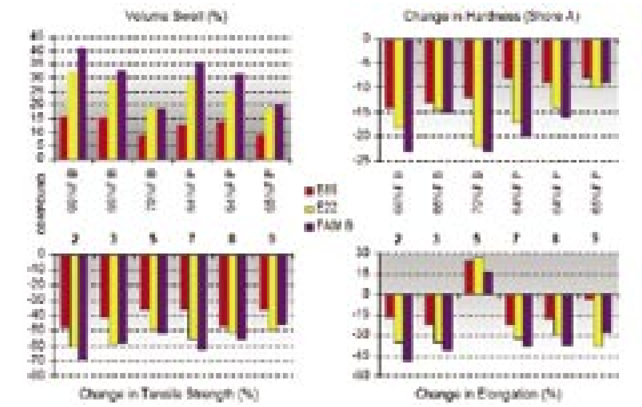
Results in water contaminated diesel

Water is a common contaminant of commercially available diesel and typically exists as a discrete phase at the bottom of storage tanks. Water is significantly more soluble in biodiesel than in conventional diesel, therefore the possibility of dissolved water being present in biodiesel blends increases. Graph 3 compares the effects on sealing compounds after immersion in B30 and B30 contaminated with one percent water.

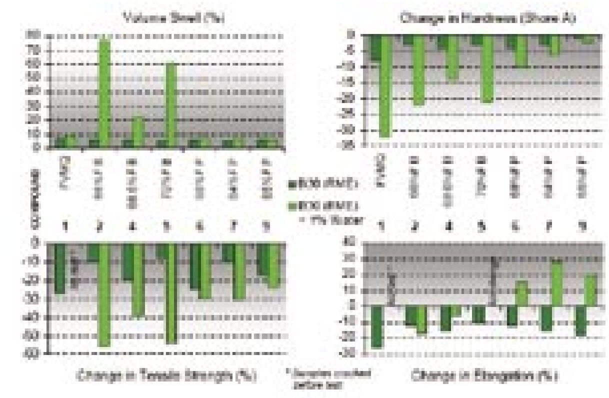
The results show a large deterioration in properties for all bisphenol cured FKMs,



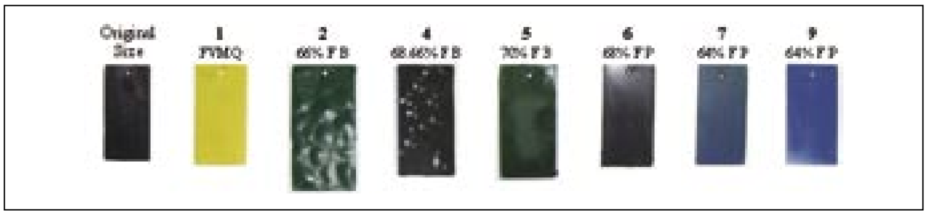
Graph 1: Results of immersion of compounds in conventional diesel and biodiesel mix B30.



Graph 2: Results of immersion of sealing compounds in gasoline based biofuels.

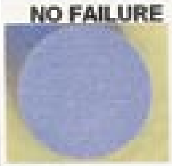


Graph 3: Results of immersion of sealing compounds in B30 and B30 plus one percent water.



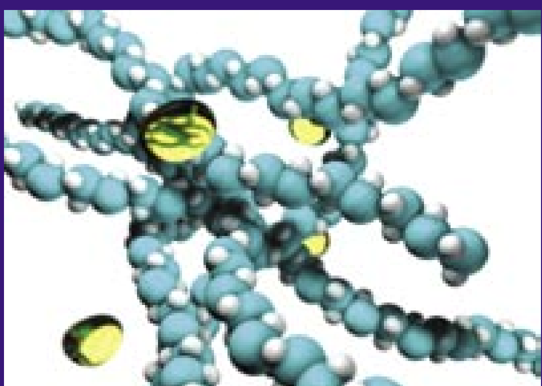
Graph 4: The relative volume change of the test pieces together with blistering of some of the compounds.

compounds 2, 4 and 5. Additionally, graph 4 shows the relative volume change of the test pieces together with blistering of some of the compounds.
The reason for the significant effect on bisphenol cured FKM is their metal oxide content. Water contaminant causes hydrolysis of the esters found in biodiesel and the subsequent formation of carboxylic acids. The carboxylic acids in conjunction with the metal oxides contained in bisphenol cured FKMs cause the deterioration of the polymer via dehydroflourination.
Peroxide-cured FKMs performed best in B30 plus one percent water. Unlike bisphenol cured FKMs, peroxide cured FKMs do not require metal oxides for good vulcanization. The only FVMQ tested showed the highest deterioration in this test with the test pieces disintegrating before a stress-strain reading could be obtained.

	COMPOUND		
	7 (64%F P)	7 (64%F P)	8 (64%F P)
Test Medium	Fuel C	E22 (Fuel C 78% + Ethanol 22%)	
RESULT	NO FAILURE 	FAILURE 	NO FAILURE 
Test Pressure and Medium	120 bar, Nitrogen		
Test Duration and Temperature	168 hours at 60°C/ 140°F		
Decompression Rate	< 1 seconds		
Test Specimen / Sealing Type	O-Ring / piston sealing		
Squeeze / Groove Fill	20% / 80%		

Graph5: results of rapid decompression testing in Fuel C and E22.

Conclusions of the test



• Although standard laboratory tests may suggest that typical FKMs and FVMQs are compatible with biofuels, tests designed to replicate service conditions present a different picture.

• Water contamination in biodiesel is highly likely and the resultant formation of carboxylic acids has a significant degenerative effect on metal oxide containing FKMs, such as the bisphenol vulcanized compounds.

• The addition of ethanol in high-pressure gasoline applications increases the possibility of rapid decompression failure. The resultant cracks dramatically reduce the integrity of the seal and are difficult to detect whilst still sub-surface.

• It is vital that the sealing compound used in biofuel applications is correctly specified for potential operational factors to ensure long and effective seal life. <<

Gasoline high-pressure applications

In high-pressure gasoline applications there is the risk of a sudden reduction of pressure in the system. In these situations gas captured in the seal can cause the seal to split or crack sub-surface, dramatically reducing seal integrity.
To study the effect of rapid decompression in gasoline and biofuel mixes, five O-rings manufactured in compounds 7 and 8 were assembled onto test equipment replicating a high-pressure fuel injector. The O-rings were in contact with pressurized fuel from one side, and aged for 168 hrs at 60°C. The pressure was then reduced to atmospheric pressure within one second and the O-rings were inspected. The fuels tested were Fuel C, representing regular gasoline, and E22. Compound 7 was tested in gasoline with no biofuel added and in E22. Compound 8 was tested in E22 only.
No failures were detected in O-rings made from sealing compound 7 when tested with conventional gasoline. With E22, the O-rings made from compound 7 exhibited internal cracks. These were sub-surface and difficult to detect. This was due to the higher polarity and smaller size of the ethanol in E22 causing increased swell and a large enough reduction in mechanical properties to result in O-ring damage during rapid decompression.
Compound 8 is based on the same polymer as compound 7 but has been specifically formulated to withstand a rapid decompression environment. No decompression failures in the O-rings made from compound 8 were detected when tested in E22. <<

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