Novel Findings on Elastomer Exposure Effects in Palm-Based Biodiesel Blends

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Abstract: Several studies on the use of elastomers in biodiesel have primarily focused on how the characteristics of elastomers change when exposed to biodiesel. However, information on the changes in biodiesel characteristics remains limited. This study examined the characteristics of B30 and B40 palm-based biodiesel blends during elastomer exposure. The elastomers were submerged in static B30 and B40 for 1007 hours at ambient temperature. Following 71, 167, 671, and 1007 immersion hours, the characteristics of the biodiesel blends, such as total acid number, water content, color, and oxidation stability, were analyzed. The results show that NBR has the most significant effect on the decline in the quality of B30 and B40, followed by EPDM. In contrast, fluoroelastomer has almost no significant effect. The total acid number of the biodiesel blends increased during NBR immersion. However, fuel immersed in EPDM and fluoroelastomer exhibited opposite trends. The water content characteristic generally increased over time during the 1007 immersion hours of the elastomers; the biodiesel blends observed a significant color change from 0.8 to >2.8 for NBR, 0.8 to 3.50 for EPDM during 71 immersion hours, then had a constant tendency up to 1007 immersion hours. The biodiesel's oxidation stability significantly decreased by 96% for NBR and 97% for EPDM during 71 hours of immersion. However, after 1007 immersion hours, oxidation stability increases, probably due to the absorption of biodiesel oxidation by-product by elastomers.

Keywords: palm-based biodiesel; characteristics; elastomers; immersion

1. Introduction

Nowadays, the decline of fossil fuel reserves will drive the use of sustainable biofuels. The advantages of using biofuels are renewable supply, reduced air pollution or greenhouse gas emissions, and may be able to reduce dependence on imported fuel. One alternative fuel that can replace fossil fuel is biodiesel, which is created when any natural oil or fat is trans-esterified with an alcohol, such as ethyl or methyl alcohol[1)], to form fatty acid methyl ester (FAME). Biodiesel can be generated from renewable resources available in each region which may improve energy resilience from imported fuel. Biodiesel is classified based on the origin of its raw materials; one of them is palm-based biodiesel. When palm biodiesel blend was utilized in diesel engines, unburned hydrocarbon emissions were reduced by 73%, and carbon monoxide emissions were decreased by 46%[2)]. While carbon monoxide and hydrocarbon emissions decrease, NOx emissions increase in line with the volume of biodiesel in the mixture[3–8)].

Among those advantages, biodiesel also has disadvantages that may complicate its use. Even though biodiesel has similar physical properties to diesel fuel, at some points, it also has different characteristics compared to petro-diesel fuel, for example as ester compound biodiesel has solvency characteristics. Since biodiesel acts as a stronger solvent than petroleum diesel, it has a different impact on elastomers[9)]. When transitioning to biodiesel, it can remove carbon deposits and cause fuel filter clogging[10)]. Part of diesel engine components, contact with biodiesel may be impacted, and the degrading process of elastomers like seals, gaskets, and hoses, as well as the corrosion of metallic sections of filters, pumps, nozzles, and other components, may be observed. When the oxidation is followed by material swelling, the defect caused by biodiesel to rubbers may be substantially more severe. In addition, rubber compounds include stabilizers,

plasticizers, processing aids, and curing agents that may migrate to the fuel and immediately affect the rubber's characteristics[11–13]. Biofuel may also be impacted by the additive's migration from the rubber[14]. Those problems limit biodiesel use because of how biodiesel's unsaturated molecules and compositional effects cause metal and rubber components in automobile fuel systems to degrade Click or tap here to enter text.Click or tap here to enter text.[15–22].

Indonesia has been blending biodiesel with Petrol-diesel as a fuel in various sectors since 2006. At the beginning of 2020, the government implemented the highest volume of biodiesel in blends by 30% (B30) and may continue until 40% and 50%[23]. Due to unsaturated biodiesel molecules, oxidation stability is one of the essential parameters that characterize biodiesel quality. Biodiesel oxidation leads to oxidative product formation, such as carboxylic acids[24–26]. Upon exposure to air, biodiesel is prone to oxidation, which can occur in a few hours, depending on storage conditions and the degree of fatty acid unsaturation. As biodiesel is stored, its acid number tends to increase over time. At the fatty acid unsaturation sites, the first oxidation products are hydroperoxides. These decomposed into ketones, aldehydes, and shorter-chain carboxylic acids [27]. The longer the fuel is stored, the lower its oxidation value. A considerable oxidation value means a more stable fuel condition. The effect of fuel with low oxidation stability is that it can cause the fuel to become acidic and form insoluble gums and sediments, which can clog fuel filters[28].

According to the results of several studies[1,9,14,16,29–38] investigating how biodiesel and its blends affect elastomeric materials, such as nitrile butadiene rubber (NBR)[35,39–42], ethylene propylene diene monomer (EPDM)[30,33,35,43], and chloroprene rubber[35,44]. Biodiesel increases mass and volume change, elongation at break, and lowers tensile strength, regardless of the feedstock's origin. The degradation of elastomers in the static immersion of B30 palm-based biodiesel for 1007 hours was observed [33], which results in fluoroelastomer having a less noticeable degrading impact than EPDM.

Although many researchers have studied the compatibility of elastomers with biodiesel, few studies focus on the characteristics of biodiesel itself, especially palm oil-based biodiesel, due to elastomeric contacts. This study reports the static immersion effects of NBR, EPDM, and fluoroelastomer commonly utilized in gasket, fuel hose, and o–ring on changes in the characteristics of B30 and B40 palm-based biodiesel blends. In contact with biodiesel, EPDM exhibits the most significant changes in characteristics in terms of mass swelling, followed by NBR, while fluoroelastomer shows no significant changes [30,45]. Whether the changes in the characteristics of the elastomers are directly proportional to the changes in the characteristics of biodiesel as the soaking medium remains questionable and will be clarified in this research. The characteristics of biodiesel blends were analyzed by total acid number, water content, color, and oxidation stability.

2. Experimental

2.1 Materials and Methods

B100 was obtained from PT. LDC Indonesia, a local biodiesel producer in Sumatra, Indonesia, while B30 fuel was purchased from a fueling station owned by PT. Pertamina (Persero) in South Tangerang, Indonesia. Before mixing, the exact concentration of biodiesel in B30 (% FAME) was determined using Agilent Cary FTIR. B40 fuel was obtained by adding biodiesel (B100) into palm-based B30 fuel in a certain volume ratio according to Eq.1. B40 fuel was obtained by mixing palm-based B30 and biodiesel (B100) in a certain volume ratio. Fuels were tested for crucial parameters listed in Table 1, which refers to Indonesian Decree of Directorate General of Oil and Gas No. 0234.K/10/DJM.S/2019.

$$V_{B100} = \frac{V_{B30}\,(40\% - \%\,FAME_{B30})}{60\%} \qquad \text{(Eq.1)}$$

V_{B100} = Volume B100 added to B30 (mL)
V_{B30} = Initial volume of B30 (mL)
$\%\,FAME_{B30}$ = Exact biodiesel content in B30 (%)

Table 1. Fuel Testing Parameters

Parameters	Method	Specifications*		
		Unit	Min.	Max.
Density 15°C	ASTM D4052	kg/m^3	815	880
Water Content	ASTM D6304	ppm		425
Total Acid Number	ASTM D664	mg KOH/g		0.6
FAME content	ASTM D7371	%vol	30	
Viscosity 40°C	ASTM D445	cSt	2.0	5.0
Color	ASTM D1500-07	Color No.		3
Oxidation Stability	EN 15751	hours	35	

The elastomers used in this study are ethylene propylene diene monomer (EPDM), fluoroelastomer, and nitrile butadiene rubber (NBR). Elastomers were prepared using commercial grades of those. Elastomers are widely used in fuel storage systems in tanks. They are used for gaskets, such as pipe joints (flanges), seals in filters, flanges in tank manholes, seals in fuel pumps, valve connections, and other pipe or instrument connections. Today's most commonly used elastomers, such as EPDM and NBR, are inexpensive and readily available.

2.2 Immersion of elastomers

The immersion test was carried out following the guidelines outlined in ASTM D471. The elastomers were hung from a plug at the top of a glass tube 3.8 cm (about 1.5 inches) in diameter and 30 cm (about 11.81 inches) in length filled with fuels and submerged for 1007 hours between 27°C and 30°C. Those operation temperatures represent actual conditions in the storage tank or pump.

2.3 Analysis of B30 and B40 characteristics

60 ml of each fuel was sampled at 0, 71, 167, 671, and 1007 immersion hours according to ASTM D471-16a[33]. The water content, total acid number, color, and oxidation stability were analyzed. Especially for oxidation stability parameters, fuel sampling was carried out only at 0, 71, and 1007 immersion hours. The test results were compared to Standard Spec Source (Indonesian Decree of Directorate General of Oil and Gas No. 0234.K/10/DJM.S/2019).

2.4 Determination of fuels total acid number

The total acid number of fuels was analyzed using Metrohm 848 Titrino plus following the procedure specified in ASTM D664-18, expressed in mg KOH per gram of biodiesel blends. It requires 10 grams of samples solved with 60 ml of solvent (made from >99.8% anhydrous toluene anhydrous, >99.5% anhydrous isopropyl alcohol, and distilled water with a volume ratio 100: 99: 1) titrated with potassium hydroxide solution in anhydrous isopropyl alcohol, to a specified endpoint.

2.5 Determination of fuels water content

The water content of fuels was analyzed using Mitsubishi Chemical Karl Fischer CA-310 with a coulometric technique following the procedure specified in ASTM D6304-07.

2.6 Determination of fuels color

The color of fuels was analyzed using Lovibond PFXi - Series following the procedure specified in ASTM D1500-07.

2.7 Determination of fuels oxidation stability

The oxidation stability of fuels was analyzed using Metrohm 892 Professional Rancimat following the procedure specified in EN 15751, 7.5 grams of samples in a reaction vessel with a sealing cap were connected to the instrument. The 10 liters/hour of purified air is passed through the samples, which have been heated to 110 °C. During the oxidation process, volatile compounds are generated and delivered with the air into a flask of distilled water fitted with a conductivity electrode. Measurement and recording equipment is connected to the electrode. The sudden increase in conductivity occurs when the water absorbs volatile carboxylic acid due to the oxidation process, signaling the end of the induction period.

3. Results and Discussion

Table 2 shows the results of the tests conducted on the immersion fuels. According to that table, B30 met the requirements of the Indonesian fuel specification, which is stated in the regulation. Since the standard specification for B40 is unavailable, the B40 characteristic was analyzed using the B30 specification, and the result is within the allowable limit.

Table 2. Test Results of Fuels

Parameter	Method	Unit	B30 Result	B40 Result
FAME Content	ASTM D7371	%vol	28.39	40.58
Water Content	ASTM D6304	ppm	199.14	305.46
Total Acid Number	ASTM D664	mg KOH/g	0.14	0.15
Density 15°C	ASTM D4052	kg/m^3	849.6	853.2
Viscosity 40°C	ASTM D445	cSt	3.01	3.19
Color	ASTM D1500-07	Color No.	0.8	0.8
Oxidation Stability	EN 15751	hours	63.28	37.48

3.1 Effect of elastomers on changes in fuel's total acid number

Figure 1 shows the influence of immersion hours on changes in fuel's total acid number. Both fluoroelastomer and EPDM moderately decreased total acid number during 71 immersion hours, then had a constant tendency for EPDM while decreasing gradually for fluoroelastomer. This phenomenon may be affected by curing agents such as diamines in those polymers[46]. In contrast to other elastomers, NBR caused a gradual increase in the total acid number of B30 and B40 up to 50% during 1007 immersion hours but still met the specification required by regulation from the Indonesian's fuel specification. The oxidation of biodiesel generates carboxylic acids such as formic acid, propionic acid, caproic acid, and acetic acid[47]. It is important to note that the water can hydrolyze the ester groups of the methyl esters of fatty acids, creating acid groups[39].

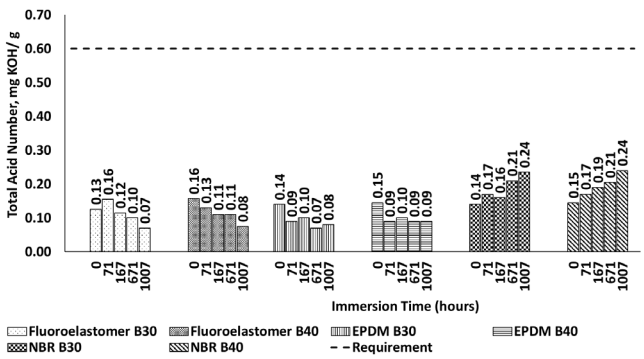

Fig 1. Changes in Total Acid Number of Biodiesel Blends by Different Elastomers for 1007 hours

3.2 Effect of elastomers on changes in fuel's water content

Figure 2 shows how changes in immersion hours affect the water content of fuels. In B30, water content is not affected by the fluoroelastomer but increases moderately during the initial exposure period (71 immersion hours) for EPDM and NBR. However, at B40, all elastomers caused a moderate increase in water content during the initial exposure period (71 immersion hours), but then slightly increased up to 1007 immersion hours. However, up to 1007 immersion hours, the moisture content still met the specifications, remaining below 425 ppm. Since B40 contains more B100, it is more hygroscopic, increasing the water content over time.

Furthermore, the increased rate at which acids and water are formed in biodiesel is probably related to the concentration of hydroperoxides present as storage time increases, which promotes the reaction to form acids and water[14]. As oxidation advanced, the reaction pathways altered due to the rising number of hydroperoxide molecules. These molecules attack the fatty acid substrate and lead to the production of water.

Although B40 is more hygroscopic, the rate of water content increase is higher in B30. After 1007 hours of immersion, the water content increased by 45% and 37% for NBR and EPDM, respectively, while for B40 it was only 27% for EPDM and 31% for NBR. This finding is likely due to the initial water content (0 hours). In B40, the initial water content is higher than in B30, thus closer to equilibrium.

3.3 Effect of elastomers on changes in fuel's color

The ASTM Color Scale is a one-dimensional color scale number with sixteen levels that range from a light straw to a deep red (0.5 to 8.0 units in steps of 0.5 units). Figure 3 shows the effect of immersion time in both B30 and B40 color changes. Fluoroelastomer has no impact on color changes due to the CH_3-F link's strong dissociation energy bond and excellent resistance compared to other elastomers[9,31,33]. The dissociation energy bond and material resistance are related to each other. Those factors affect the degree of elastomer cross-linking degradation[31,48].

Otherwise, a substantial color change happened during EPDM and NBR immersion. The highest increase occurred after 1007 hours of NBR immersion, namely 33.75% and 37.5% for B30 and B40, respectively. For EPDM, the color changed enormously during the initial exposure period (71 immersion hours). It tended to change slightly during the mid-period of exposure and finally reached a stable at 1007 immersion hours. However, the increase in color level did not exceed the specification, which is a maximum of 3. There are no significant differences in color changes between B30 and B40 during exposure. NBR causes the most significant color changes, which exceed the maximum specification of 3. The color changed enormously during the initial exposure period (71 immersion hours), then slightly increased during the mid-period of exposure and finally decreased starting at 671 immersion hours in B30. However, at B40, after the color strongly changes at the initial exposure period (71 immersion hours), it tends to reach stability. This phenomenon's likely cause is stabilizer migration from the elastomer to the biodiesel[14]. The loss of plasticizer from the rubber in biodiesel is another cause of this [14]. With the migration of plasticizer and stabilizer, there was no protective mechanism against oxidation in NBR[14].

Consequently, the migration of carbon black as a filler caused the fuel to become darker in color. The color parameter is crucial in determining fuel quality. Color differences in diesel fuel signify impurities, contamination, reduced combustion efficiency, and decreased engine performance.

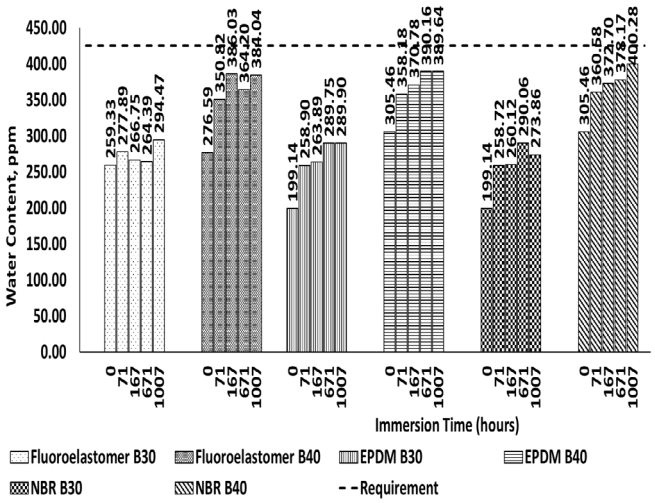

Fig. 2. Changes in Fuel Water Content of Biodiesel Blends by Different Elastomers for 1007 hours

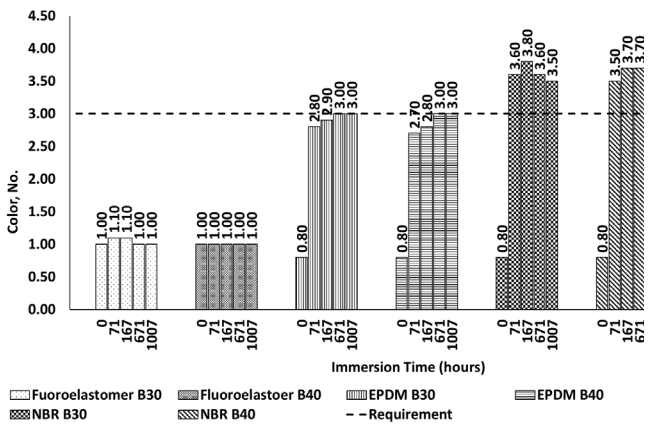

Fig. 3. Changes in Fuel Color of Biodiesel Blends by Different Elastomers for 1007 hours

3.4 Effect of elastomers on changes in fuel's oxidation stability

Fluoroelastomers have better resistance than other elastomers because of the strong cross-linking bond between CH_3 and F. A higher fluorine content in fluoroelastomers enhances their resistance to fuel permeation and degradation. So far, the least resistant fluoroelastomer tested in biodiesel contains 64% fluorine by weight. Figure 4 shows how immersion hours affect a fuel's oxidation stability. Initially, both B30 and B40 significantly decreased oxidation stability during 71 immersion hours of NBR and EPDM, then slightly increased at 1007 h, which resulted in the biodiesel blends being below the specified standards, lower than 35 hours.

Compared to EPDM, NBR causes a more significant decrease in the oxidation stability of both B30 and B40. After 71 immersion hours, NBR results in 98.35% and 98.56% degradation at B30 and B40, respectively, while EPDM causes 98.14% and 97.76% degradation at B30 and B40, respectively. Akhlaghi et al.[14] discovered that after immersion in biodiesel, the intensity of nitrile groups in NBR decreases. This occurrence may be due to the degradation of elastomer structures, causing the constituent monomers of elastomers to migrate into the fuel. The triple bonds in NBR are more susceptible to oxidation than the double bonds in EPDM, resulting in a more significant decrease in oxidation stability. Compared to NBR, EPDM causes a more significant decrease in the oxidation stability of B30 for 1007 immersion hours, but at B40, it was almost the same. EPDM causes 97.88% degradation at B30 and 95.58% at B40, while NBR causes 96.88% degradation at B30 and 96.70% at B40.

From a fuel point of view, B40 possesses lower oxidation stability than that of B30. B100 contains poly-unsaturated fatty acid, which makes it more hygroscopic, thermally unstable, and readily oxidized than petro-diesel[31,49]. B100 can be exposed to air, light, and high temperatures during storage, generating peroxides and hydroperoxides as the first oxidizing products. This oxidation produces alcohols, carboxylic acids, ketones, and aldehydes[50]. An interesting result can be observed in Fig. 4., increasing the immersion period from 71 to 1007 hours leads to a slight increase of oxidation stability. The probable reason is that the elastomer absorbed some of the biodiesel's oxidation byproducts, mainly its main oxidation byproducts like alkyl and peroxyl radicals. As soon as they are formed during the initial oxidation phases, these reactive radicals rapidly attack adjacent unsaturated moieties, starting a chain reaction[39]. Therefore, further oxidation can be avoided as the elastomer absorbs these free radicals and the other oxidation products[14].

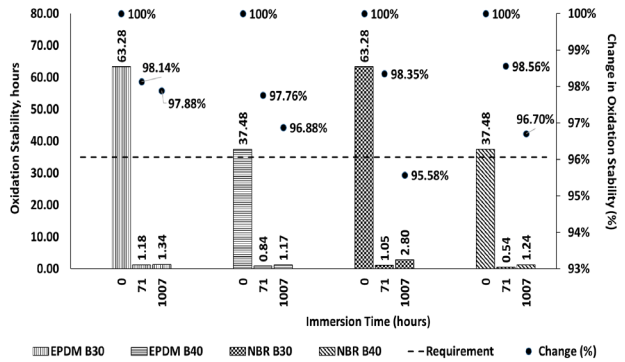

Fig. 4. Changes in Fuel Oxidation Stability of Biodiesel Blends by Different Elastomers for 1007 hours

3.5 Characteristics of Elastomers Affect in Biodiesel Quality

Among the three elastomers, NBR has the most significant effect on the decline in the quality of B30 and B40, followed by EPDM. In contrast, fluoroelastomer has almost no significant effect. The decrease in fuel quality occurs due to the degradation of elastomers, which causes the migration of compounds within the elastomer to the fuel.

Elastomer degradation is partly due to the elastomer's lack of resistance to fuel, particularly those containing biodiesel. In its production, besides the rubber material itself, elastomers also contain other additives, one of which is a curing agent. The curing process causes the long polymer chains of rubber to become crosslinked. The concentration of the curing agent affects the polymer crosslink density. However, crosslink density alone does not directly contribute to the elastomer's resistance to biodiesel. Besides the curing agent, an activator is also needed in the curing system. The selection of the type and concentration of this activator plays a more significant role in the elastomer's resistance to biodiesel[51].

In addition to mechanical properties, the similarity in polarity between the fuel and the elastomer also affects elastomer degradation. A general principle states that polar substances are more likely to dissolve in polar solvents, while non-polar substances are more likely to dissolve in non-polar solvents. It is noted that EPDM has a lower degree of polarity compared to NBR [49].

Compared to the other two, fluoroelastomer has the most negligible impact on the degradation of biodiesel quality. It is known as a biodiesel-resistant material with a cross-linking bond between CH_3 and F. Therefore, fluoroelastomer has minimal impact on the fuel. However, fluoroelastomer also experiences degradation. The type of fluoroelastomer least resistant to degradation contains high unsaturation and metal oxide/hydroxide particles[9].

Based on those results, fluoroelastomer has the most minor degradation effect on biodiesel blends. The use of EPDM and NBR in the degradation of biodiesel blends quality which will affect the fuel's life. Degraded quality fuel will affect the engine performance, so its use should be avoided. Hence, it is essential to carefully choose the elastomers in fuel systems that contact high-ratio biodiesel blends to prevent degradation in biofuel characteristics, such as the use of fluoroelastomer.

4. Conclusions

This paper reports that different elastomers result in different characteristic changes in B30 and B40 palm-based biodiesel blends. The effect of elastomers shifts gradually over time after the initial immersion stage, indicating a negative impact on biodiesel blends. NBR has the worst changes to biodiesel blends in total acid number, color, and oxidation stability. It causes an increase in total acid number, contrast with EPDM and fluoroelastomer, and the most remarkable color change in biodiesel blends. After 71 immersion hours, both B30 and B40 colors have changed drastically and then tend to be constant up to 1007 hours, with ≥3 of ASTM color scale resulting in Off specification according to fuel specification regulations in Indonesia. Water content characteristic was generally increased over time during elastomer immersion over 1007 immersion hours, with B40's water content greater than B30's due to its hygroscopic characteristic. At 71 immersion hours NBR causes more tremendous changes in oxidation stability than EPDM. However, after 1007 immersion hours, the oxidation stability of both elastomers slightly increased, resulting in higher oxidation stability in NBR. During 1007 immersion hours, EPDM causes greater changes in oxidation stability than NBR in B30, while almost the same in B40. Based on those results, fluoroelastomer has the most negligible degradation effect on biodiesel blends. Hence, it is crucial to carefully choose the elastomers in fuel systems that come into contact with high-ratio biodiesel blends to prevent degradation in biofuel characteristics.

It is necessary to conduct research on elastomers for biodiesel blend ratios above 40% and to include temperature variations to determine their effects on both the elastomer and the fuel. The weakness of elastomers when in contact with fuel lies in their impermeability. The impermeability of an elastomer is derived from the size of its free volume, which is determined by the quality and quantity of cross-linking. Therefore, it is essential to deepen the characterization of elastomer cross-linking before and after fuel immersion, as well as the changes in the fuel quality.

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