

# Development and evaluation of nitrile rubbers seals for power transformer application

Mauro Cesar de Avila<sup>1</sup> , Ana Paula Munaro<sup>2</sup>  and Marilda Munaro<sup>1,3\*</sup> 

<sup>1</sup>*Programa de Pós-graduação em Engenharia e Ciência dos Materiais – PIPE, Universidade Federal do Paraná – UFPR, Curitiba, PR, Brasil*

<sup>2</sup>*Solução Adesivos e Selantes, Fazenda Rio Grande, PR, Brasil*

<sup>3</sup>*Centro Universitário Curitiba, Curitiba, PR, Brasil*

\**marilda.munaro94@gmail.com*

## Abstract

Mineral insulating oil (MIO) and natural ester insulating (NEI) oil are used in power transformers as an insulating fluid, while elastomeric seals are usually composed of nitrile rubber (NBR). The proprieties of these seals can change in contact with insulating oil. Variation in sealant properties is undesirable in power transformers. In this work, the variation of some elastomer properties was evaluated before and after accelerated aging in MIO and NEI. The developed elastomeric compositions showed variation in the stress and strain at break after the aging test. It also appeared that MIO penetrated the samples and that dioctylphthalate (DOP) migrated or was extracted into MIO and NEI. The samples vulcanized with peroxide showed better results than those vulcanized with sulfur.

**Keywords:** *compatibility, mineral insulating oil, natural ester insulating, nitrile rubber, power transformers.*

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## 1. Introduction

Electricity is one of the most widely used forms of energy throughout the world. In order for this energy to reach the consumer, it passes through a complex system of generation, transmission, and distribution. The distribution stage is performed using power transformers that raise or lower the voltage to enable distribution over long distances. Electrical power transformers immersed in insulating oil are fundamental for the generation, transmission and distribution of electricity, but they are also very expensive. Cooling and insulation of the electrical system in transformers is achieved by using insulating paper and insulating oils, with mineral insulating oil (MIO) most commonly used. The system is sealed with rings or gaskets composed of elastomers, especially nitrile rubber (NBR)<sup>[1-3]</sup>. Another insulating fluid used is natural ester insulator (NEI) obtained from oilseeds such as soya, canola, sunflower seeds, etc. Natural ester insulators have excellent electrical properties such as flash points greater than 300 °C, which significantly improves the safety level of the power grid, biodegradation rates of more than 95%-97%, and relative dielectric constants about 1.5 times that of mineral insulating oils<sup>[4]</sup>. NEI oils are less flammable compared with MIO oils and they do not contain corrosive sulfur. These oils also offer adequate dielectric strength, making them a feasible alternative for MIO oils in power transformers<sup>[5,6]</sup>. Another important aspect to consider in the use of natural ester as a substitute for mineral oil as a dielectric fluid in transformers is the aging of cellulose

insulating paper. When in contact with fluids of plant origin, this paper normally has a lower speed of aging than that which occurs on the same paper when immersed in MIO under the same experimental conditions<sup>[7]</sup>.

NBR formulated elastomeric materials were first used in the automotive industry because of their resistance to fuels and a variety of oils and other fluids. Currently, NBR is used in a large number of applications, mainly in seals where the material is in permanent contact with the lubricants necessary for the vehicle's moving parts<sup>[8,9]</sup>.

The elastomeric seal allows complete contact between the seal and the joint and settles with slight pressure<sup>[10]</sup>. Rubber products are available in different types of hardness and textures. Harder material can be used in cases of greater stress, where increased sealing power is needed. Most elastomeric seals have excellent flow and compressibility characteristics. The lateral flow increases when the stresses in the seal are high during use. Different fillers and fibers can be used in the fabrication of elastomeric seals in order to improve mechanical strength and maintain seal stability and control<sup>[11]</sup>. Nitrile rubber (NBR) is a copolymer of acrylonitrile and butadiene. The polar acrylonitrile group present in NBR imparts excellent fuel and oil resistance, and hence it is widely used in many important industrial applications like fuel and oil supply hoses, rollers, drive belts, seals, and so on<sup>[12]</sup>.

Material compatibility is a vital element for transformer design. All the materials that are typically used in the manufacturing of mineral oil-immersed transformers have to be compatible with natural ester liquids<sup>[13]</sup>. Incompatibility between the seal material and the insulating oil can result in degradation of the elastomer and contamination of the oil, causing loss of the dielectric properties of the fluid, reduced useful life of the transformer, and environmental contamination due to fluid leakages. Power transformer failures lead to power supply disruption, fines paid by energy utility companies, maintenance costs, and sometimes the loss of the transformer.

Previous studies have investigated the compatibility of elastomers with lubricating oils and fuels such as gasoline, and diesel<sup>[14]</sup>. Studies have been conducted on the compatibility of nitrile rubber (NBR) with different biodiesel compositions<sup>[15]</sup>. A recent review pointed out aspects of using NEI in transformers. However, there have been no evaluations of the rubber seal<sup>[6]</sup>.

The authors are not aware of studies evaluating sealing elastomers aged in insulating fluid, especially NEI. Much less is known concerning the performance of elastomers in contact with MIO and NEI<sup>[16-19]</sup>.

The aim of this work is to evaluate different compositions of nitrile rubber submitted to aging in insulating oil, MIO and NEI. The samples were characterized by determination of Shore A hardness, tensile strength, and elongation, as well as by thermogravimetry analysis (TGA) and dynamic mechanical thermal analysis (DMTA) before and after the aging tests. Only the elastomer samples were evaluated. Possible changes in MIO and NEI are outside the scope of this study and have not been evaluated.

## 2. Materials and Methods

### 2.1 Materials

The compounds were produced using NBR containing 34% acrylonitrile 35 LM® supplied by KUMRO, dioctyl phthalate plasticizer (DOP) supplied by CYA Rubber, Nipol 1312 LV® polymeric plasticizer (low viscosity liquid NBR with 28% acrylonitrile) supplied by Zeon; and carbon black (types N762 and N339) supplied by Cabot.

The other additives used were as follows: antioxidant (quinolone type) and antiozonant (paraphenylenediamine type) from Lanxess; zinc oxide from Brasóidos; stearic acid from SIM Stearin; ventilated sulfur, benzothiazole vulcanization accelerator (MBTS), and tetramethylthiuram disulfide vulcanization accelerator (TMTD) from Auriquímica; 40% dicumyl peroxide in calcium carbonate from Retilox, and hydrocarbon resin from Parabor.

Mineral insulating oil type AV-66 IN® supplied by Petrobras and natural ester insulating soy oil type ENVIROTEMP FR3® supplied by Cargil, both were used in the aging and compatibility assays. Some properties of the MIO and NEI are shown in Tables 1 and 2.

### 2.2 Methods

Preparation of the Mixtures: the formulations were prepared in a laboratory cylinder (Model Lab mil 350,

COPE brand), as shown in Figure 1, according to the ASTM D3182 standard method 8<sup>[20]</sup>. First, the elastomers were added to the cylinder with internal cooling water circulation, followed by fillers and plasticizer until complete mixing. Then, the other additives were added and mixed for a period of 4 minutes, except for the accelerators and sulfur. Finally, accelerators and sulfur were added and mixed for a time of 3 minutes. The compost discharge temperature was 80 °C and the total mixing time was approximately 15 minutes.

As shown in Figure 2, the samples were vulcanized for 4 minutes (for the formulations with sulfur) and 7 minutes (in the presence of peroxide), in a mold (150 mm x 150 mm x 2 mm) at a temperature of 160 °C. For the aging test in MIO and NEI, shore A hardness and tensile strength tests were performed on 5 specimens. Thermogravimetry and

**Table 1.** Properties of the MIO AV-66 IN®, typical value.

	TYPICAL VALUE
Neutralization Index	0.01 mg KOH/g oil
Water	20 ppm
Density at 20 °C	0.887 g/cm <sup>3</sup>
Power factor at 90 °C	0.04%
Flash point	150 °C
Viscosity at 40 °C	9.32 cSt
Viscosity at 100 °C	2.31 cSt
Pour point	-60 °C

**Table 2.** Properties of the NEI ENVIROTEMP FR3®, typical value.

	TYPICAL VALUE
Neutralization Index	0.02 mg KOH/g oil
Water	54 ppm
Density at 20 °C	0.9192 g/cm <sup>3</sup>
Dielectric losses at 20 °C	0.13%
Dielectric losses at 100 °C	3.9%
Flash point	308 °C
Viscosity at 40 °C	35.99 cSt
Viscosity at 100 °C	8.57 cSt
Pour point	-7 °C
Flash point	342 °C



**Figure 1.** Laboratory cylinder used in mixing formulations.

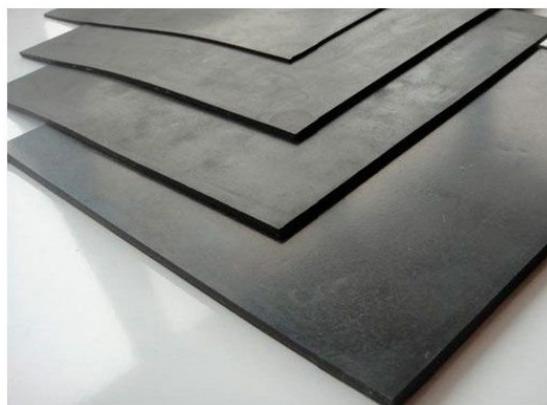


Figure 2. Vulcanized samples.

dynamic mechanical thermal analysis were performed on 3 specimens.

Four different compositions were prepared, as shown in Table 3.

Aging in MIO and NEI: the aging of the vulcanized samples was performed according to the ABNT NBR 14274 standard method<sup>[21]</sup>. The test specimens were conditioned for 16 hours in an oven at  $100 \pm 1$  °C, followed by aging in direct contact with MIO and NEI, also at  $100 \pm 1$  °C, for 7, and 14 days. The samples were aged as shown in Figure 3.

All samples were placed in the oven for the aging test at the same time and under the same conditions, as indicated in Figure 4. The first samples were removed from the oven and tested after 7 days and the others after 14 days.

Shore A Hardness: measurements were made before and after aging, according to the ASTM D2240<sup>[22]</sup>, procedure 10 using an Asker Model DD2 A durometer with a load application time of 5 seconds.

Tensile Strength Test: measurements were made according to ASTM D412<sup>[23]</sup>, before and after aging of the samples, with 5 test specimens being submitted to traction using a universal testing machine (Model 4467, Instron) operated with a 1 kN load cell and a crosshead speed of 100 mm/min.

Thermogravimetry: thermogravimetric measurements were performed according to the ASTM D6370 standard<sup>[24]</sup>, using a Netzsch Model TG-209 instrument. An approximately 20 mg portion of the sample was heated from 20 °C to 550 °C, at a rate of 20 °C/min, in an inert nitrogen atmosphere. In the next step, an oxygen atmosphere was introduced with heating at the same rate up to 800 °C. Measurements were made using samples before and after aging for 7 days.

Dynamic mechanical thermal analysis: DMTA: measurements were performed according to the ASTM E1640<sup>[25]</sup>, using a Netzsch model 242 instrument, in penetration mode. 10 mm x 10 mm plates with a thickness of 3.15 mm were used. The test was carried out in a nitrogen atmosphere in the temperature range of -60 °C to 30 °C, with 1 Hz frequency of dynamic force, and a heating rate of 3 °C/min, and nitrogen flow of 50 mL/min. The measurements were made using samples before and after aging for 7 days. MIO and ENI samples were not evaluated.

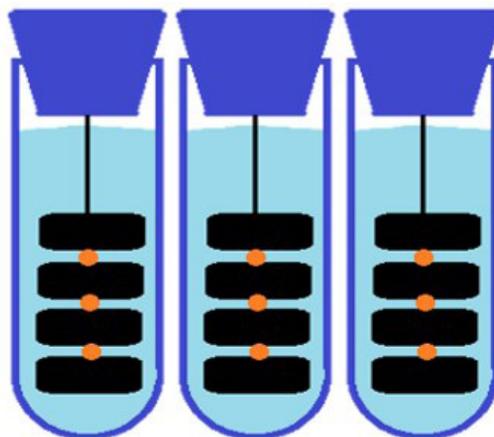


Figure 3. Schematic representation of the system used for aging samples in MIO and ENI.



Figure 4. Conditioning of the samples in the oven during the aging test.

Table 3. Compositions A, B, C and D of the NBR formulations, in PHR.

	A	B	C	D
<b>NBR 35LM</b>	100.0	100.0	100.0	100.0
<b>Polymeric Plasticizer</b>	20.0	-	20.0	-
<b>DOP</b>	-	20.0	-	20.0
<b>N 339</b>	30.0	30.0	30.0	30.0
<b>N 762</b>	40.0	40.0	40.0	40.0
<b>Zinc oxide</b>	4.0	4.0	4.0	4.0
<b>Stearic acid</b>	1.0	1.0	1.0	1.0
<b>Resin A-80</b>	8.0	8.0	8.0	8.0
<b>Antioxidant</b>	1.0	1.0	1.0	1.0
<b>Antiozonant</b>	1.0	1.0	1.0	1.0
<b>Sulfur</b>	1.6	1.6		
<b>MBTS</b>	1.5	1.5		
<b>TMTD</b>	0.2	0.2		
<b>Peroxide 40%</b>	-	-	4.0	4.0

### 3. Results and Discussions

After being prepared and vulcanized, the nitrile rubber samples showed the following properties, as shown in Table 4. The samples vulcanized with sulfur showed greater hardness than those vulcanized with peroxide, indicating greater crosslinking, and therefore the tensile strength was higher for samples A and B.

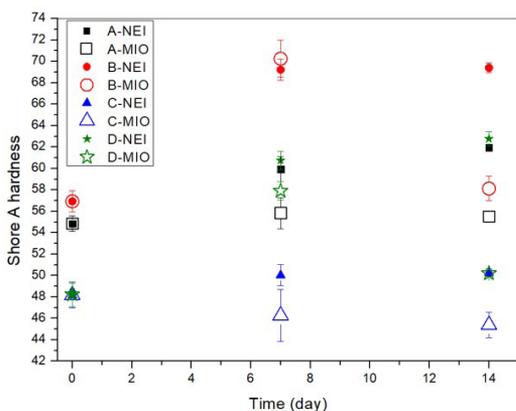
Figure 5 shows the results of the Shore A hardness test applied to the compounds before and after aging in MIO and NEI.

Compounds A and B, which were vulcanized with sulfur, showed a higher initial hardness than that of compounds C and D, which were vulcanized with peroxide. This is probably due to the lower degree of reticulation in samples vulcanized with peroxide.

For samples aged in MIO, a small increase in hardness was observed in the first 7 days, stabilizing for compositions A and C, which were formulated with polymeric plasticizer. This variation, due to being small, makes it impossible to state whether any component was extracted from the formula, even though low molecular weight compounds such as stearic acid and A-80 hydrocarbon resin may have been extracted. It is also impossible to tell whether MIO permeation occurred with consequent breakage of the chain and formation of some free radicals, crosslinking and stiffening. Compounds B and D, formulated with dioctyl phthalate ester plasticizer (DOP) showed a significant increase in hardness after 7 days. This is possibly due to the extraction of the DOP from the sample, stiffening the material. After the first 7 days, there is a reduction in hardness values up to 14 days of aging. It is possible that the MIO permeated

**Table 4.** Original properties of the prepared samples, after vulcanization.

	HARDNESS (Shore A)	STRESS AT BREAK (MPa)	ELONGATION (%)
Sample A	55 ± 1	19.8 ± 0.60	1430 ± 5
Sample B	57 ± 1	22.4 ± 0.67	1230 ± 6
Sample C	48 ± 1	18.8 ± 0.56	1450 ± 6
Sample D	48 ± 1	16.8 ± 0.50	1210 ± 3



**Figure 5.** Hardness variation as a function of aging time in MIO and NEI.

the sample and by repulsion expelled the DOP and, after 7 days, started to act as a plasticizer, resulting in a decrease in hardness as observed in 14 days of aging. In samples B and D, with plasticizer DOP the content of acrylonitrile was lower. The polymeric plasticizer adds acrylonitrile to the formula because it is a nitrile rubber of low viscosity. Thus, the MIO finds a greater non-polar fraction in compounds B and D, which favors the permeation of the MIO in these samples. The best results were observed in sample C, where the hardness variations were smaller throughout the test, indicating less degradation.

In compositions aged in NEI, an increase in hardness was observed in the first 7 days and, afterwards, the hardness practically stabilized until the end of the test. In samples B and D, the increase in hardness was greater compared to samples A and C. It is likely that the NEI in samples A and C, due to having polar groups and a certain affinity with the acrylonitrile fraction of the NBR and with the formation of free radicals due to the degeneration of the elastomer segments, crosslinked and stiffened the compositions with a consequent increase in shore A hardness. In compositions B and D, in addition to what was described for compositions A and C, the DOP must have migrated to the NEI, which resulted in increased hardness. It was also observed that the increase in hardness was lower in samples vulcanized with peroxide than those vulcanized with sulfur after aging. The changes in these properties were smaller for samples C and D, as expected, since peroxide-vulcanized compounds possess C-C crosslinks that are more stable than the C-S bonds obtained in vulcanization using sulfur, and are therefore less susceptible to thermal degradation<sup>[26]</sup>.

Figures 6a and 6b show the results of stress and strain at break for compounds A, B, C and D as a function of aging time in MIO and NEI. The values recorded in the tensile strength test in the compounds before aging showed variation, mainly in the elongation values. Sample C (polymeric plasticizer) and sample D (DOP) have the same curing system (peroxide) and the results are: stress at break of C = 18.8 MPa and of D = 16.8 MPa (C > D). Samples B and D, formulated with DOP, presented values of greater tensile strength and less elongation.

Compounds A and C exhibit similar behavior. In MIO aging, the values of maximum tension and strain at break decreased in the first 7 days and, afterwards, practically stabilized until the end of the test. MIO aging decreases the maximum stress and strain at break values. The maximum stress value is reduced, probably, due to the permeation of the MIO in the sample, which starts to act as a plasticizer and favors the movement of the segments. The reduction in the strain at break measurement value suggests that the degradation processes had started, with a consequent break in bonds. It is likely that MIO permeation accentuates the degradation processes and decreases the maximum stress and strain. The NEI sample did not significantly change the maximum stress values, but it did decrease strain at break. This behavior shows that the permeation of NEI in the samples was lower compared to that of MIO and, therefore, it did not act as a plasticizer, maintaining a maximum tension close to that of the material without aging. However, even with little permeation of NEI in the sample, some degradation

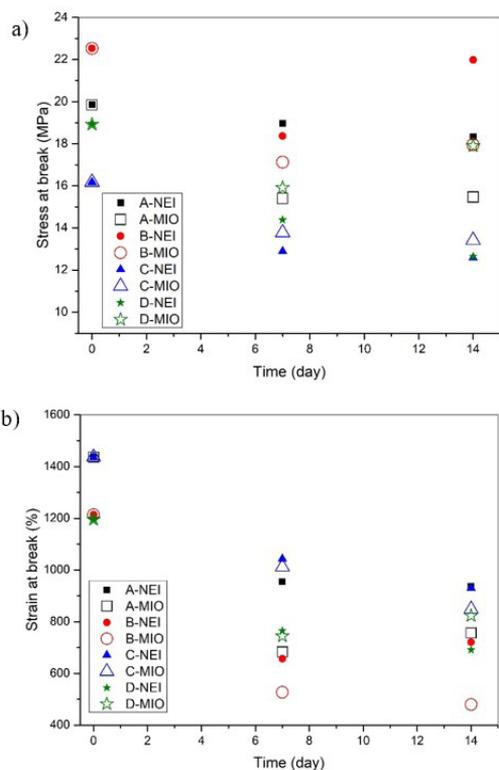
processes must have started, with a consequent chain break and decrease in strain.

Compounds B and D showed similar behavior to A and C, respectively, when aged in MIO, but with higher values in the original material without aging. With aging in NEI, this reduction was more accentuated, which was expected due to the tendency of DOP to migrate to the oil.

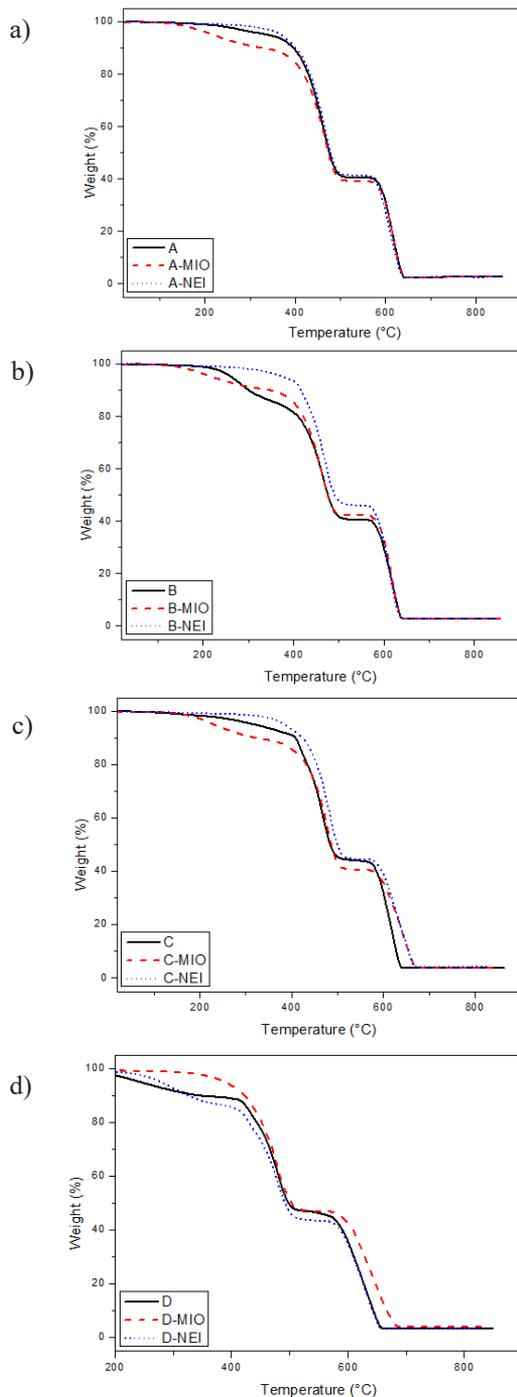
Figure 7 shows the thermogravimetric curves obtained for compounds A, B, C and D, before and after 7 days of aging in MIO and NEI, at 100 °C.

For compounds A and C, similar behavior was observed in the thermogravimetric analysis. Before aging, an initial loss of mass was observed at a temperature of approximately 235 °C, which must be lower molecular weight and non-inorganic components of the formulation that have been volatilized in this temperature range. For the formulations aged in MIO, a loss of mass was observed at temperatures below 200 °C, which can be attributed, in addition to the volatilization of low molecular weight and non-inorganic compounds as in the original sample, to the volatilization of the MIO which may have permeated between chains of the compound, in some quantity, causing some degradation, corroborating with the results obtained in the mechanical

tests. Table 5 shows mass loss of samples before and after aging in MIO and NEI up to 300 °C obtained in thermogravimetric analysis



**Figure 6.** Results of the tensile test as a function of the aging time in MIO and NEI in 7 and 14 days of aging at 100°C: (a) Stress at break; (b) Strain at break.



**Figure 7.** Thermogravimetric curves of samples (a) A; (b) B; (c) C; (d) D before and after 7 days of aging in MIO and NEI.

**Table 5.** Mass loss of samples A, B, C and D before and after 7 days of aging in MIO and NEI up to 300 °C.

	SAMPLES											
	A	A MIO	A NEI	B	B MIO	B NEI	C	C MIO	C NEI	D	D MIO	D NEI
Loss mass (%)	4.18	9.33	1.2	12.30	9.60	1.60	1.22	9.26	1.3	9.00	9.00	0.50

It is possible to observe that mass loss up to the 300 °C was greater after aging in MIO ( $A < A$  MIO and  $C < C$  MIO) showing MIO penetration into a sample. The mass loss curves for formulations A and C, when aged in NEI, showed small loss of mass until 300 °C compared to the original sample ( $A$  NEI  $< A$  and  $C$  NEI  $< C$ ). This may be an indication that low molecular mass and non-inorganic components were extracted by the NEI during aging. Compositions A and C were formulated with polymeric plasticizer which is an elastomer, and therefore the initial mass variations that preceded NBR-related mass loss should not be attributed to the extraction of the plasticizer by NEI. The reverse is expected for B and D, formulated with DOP.

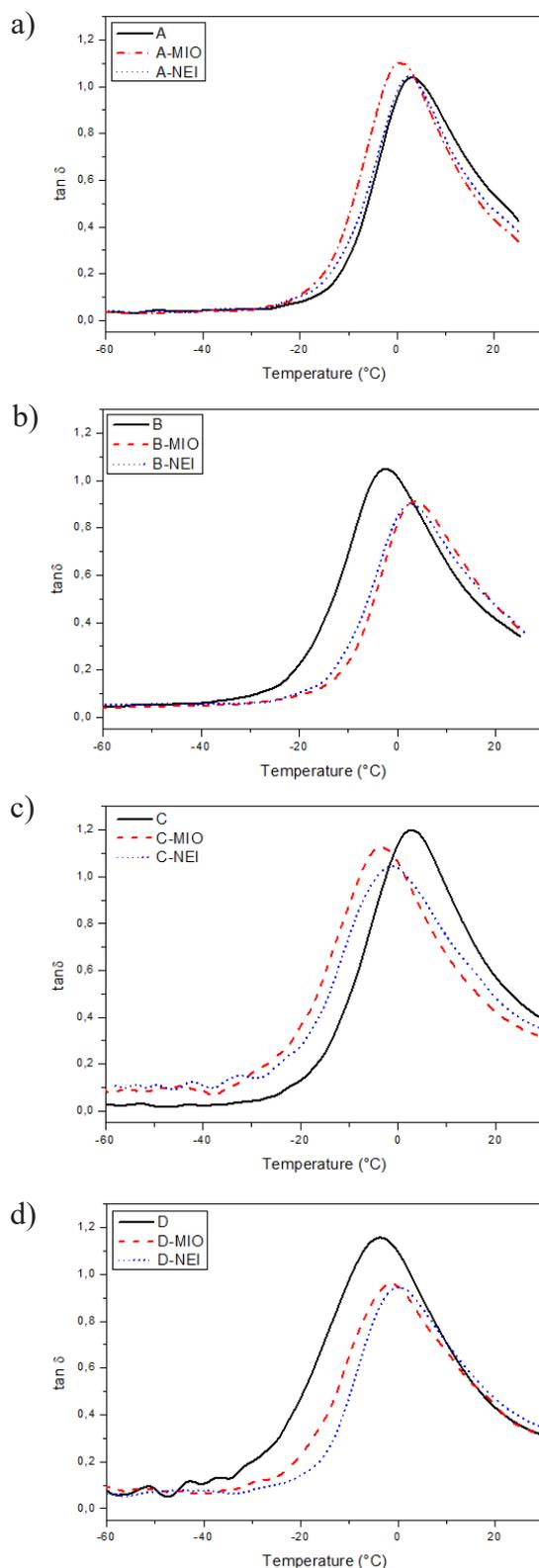
In the thermogravimetric analysis of samples B and D, a greater loss of mass was observed before aging between 230 °C and 300 °C, when compared to samples A and C. This is attributed to the volatilization of the DOP plasticizer and other components of low molecular weight. The curves for samples aged in MIO show that while part of the DOP was extracted from the formulation, part of the MIO permeated the sample, evidenced by the fact that the loss of mass started at a lower temperature and the fraction above 230 °C was lower than in the polymer without aging. For samples aged in NEI, mass loss was observed at the expected temperature range for the elastomer (above 300 °C), which may also indicate that DOP, low molecular and non-inorganic components were extracted by the NEI during the aging test ( $B > B$  NEI and  $D > D$  NEI).

The DMTA test was performed in a penetration modulus and the results are expressed in a mechanical damping modulus  $\tan \delta$ , as a function of temperature.

Figure 8 shows the  $\tan \delta$  curves obtained by the DMTA for compounds A, B, C and D before and after 7 days of aging in MIO and NEI, at 100 °C.

The behavior observed in the  $\tan \delta$  curves of samples A and C are similar and show that the glass transition of the material when aged in MIO shifted to lower temperatures when compared to samples without aging. This can be attributed to the permeation of the MIO in the sample, which starts to act as a plasticizer, thus shifting the glass transition. The intensity of  $\tan \delta$  increased in relation to the sample without aging; that is, the energy loss or viscous modulus increased, and thus it is likely that the material presents greater deformation. The material aged in NEI slightly changed the characteristics of glass transition and  $\tan \delta$ , suggesting that NEI did not permeate in sufficient quantity to affect the mobility of the elastomer chain segments and thus affect its viscous modulus.

For compounds B and D, the variation in the glass transition temperature of the compounds aged in MIO and NEI was shown to be close and shifted in higher temperatures. This can be attributed to the extraction of the sample's DOP during aging. In this case, it was expected that for the material aged in MIO the temperature would be shifted to lower temperatures than when aged in NEI due to its permeation in the sample and the MIO acting as a plasticizer. However, the amount of oil that



**Figure 8.** DMTA curves ( $\tan \delta$ ) of samples (a) A; (b) B; (c) C; (d) D before and after 7 days of aging in MIO and NEI.

permeated was probably not sufficient to change the glass transition temperature of the material, and as the efficiency

of the oil as a plasticizer was lower than that of DOP, the glass transition shifted to a higher temperature. The  $\tan \delta$  intensity of the material decreased in both cases, suggesting degradation processes and cross-linking, thus increasing the elastic modulus of the material aged in MIO and NEI.

#### 4. Conclusions

All compositions evaluated showed degradation when aged in MIO and NEI. The compounds formulated with DOP plasticizer showed significant mass loss in TGA analysis indicating their migration of the composition. The NEI extracted the DOP from the samples more intensively during the compatibility test compared to MIO. Some low molecular and non-inorganic compounds were extracted from the elastomer when aged in NEI and MIO. The samples vulcanized with peroxide showed less increase in the hardness values and less variation of the tensile strength and elongation after aging in MIO and NEI when compared to the variations in the sulfur vulcanized compounds, except for compositions formulated with DOP. Based on the variation of the evaluated properties, the 35% acrylonitrile content did not guarantee the integrity of the elastomeric compositions when aged in MIO and NEI.

#### 5. Author's Contribution

- **Conceptualization** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.
- **Data curation** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.
- **Formal analysis** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.
- **Investigation** – Mauro César de Avila; Marilda Munaro.
- **Methodology** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.
- **Project administration** – Marilda Munaro.
- **Resources** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro..
- **Software** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.
- **Supervision** – Mauro César de Avila.
- **Validation** – Marilda Munaro.
- **Visualization** – Mauro César de Avila
- **Writing – original draft** – Mauro César de Avila; Marilda Munaro.
- **Writing – review & editing** – Mauro César de Avila; Marilda Munaro; Ana Paula Munaro.

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