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LONG TERM BEHAVIOUR OF CARILON POLYMER AND COMPETETIVE POLYMERS IN AUTOMOTIVE FLUIDS

Part 2: The resistance of CARILON Polymer, PA12 and HDPE to bio-diesel fuel formulations

(March 1996 - April 1997)

by

J.J.M.H. WINTRAECKEN AND A. KRAMER

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Part 2: The resistance of CARILON Polymer, PA12 and HDPE to bio-diesel formulations

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J.J.M.H. Wintraecken and A. Kramer

Approved by: A. Noordam

Summary

To comply with the proposed EURO 2000/2005 legislation aimed at the improvement of air quality, the demands on plastic materials utilised in automotive fuel systems are to be increased substantially. CARILON Polymer shows a natural fit with these demands for future fuel systems which imply: Higher temperature regimes, the use of chemically more aggressive fuels and reduced evaporative emissions of hydrocarbons. To specify CARILON Polymer for fuel system applications the automotive industry not only requires basic mechanical, physical and chemical data but also data on the long term behaviour of this polymer. Especially the long term behaviour data in diesel fuels, including so called 'Bio-diesel', is required.

This report, the second in a series of ageing reports, deals with the long term resistance of CARILON RDP 205 at 80 °C, the competitive Polyamide 12 (PA12) at 80 °C and the competitive high density polyethylene (HDPE) at 60 °C to Bio-diesel fuels: Rapeseed methyl ester (RME) and diesel/RME (85/15 v/v) mixture.

At 60 °C, the envisaged continuous operation temperature of the fuel tank, CARILON Polymer RDP205 exhibits compared with HDPE an excellent retention of properties in RME. CARILON Polymer RDP 205 outperforms HDPE in dimensional stability, stiffness, snappability characteristics (yield performance) and ultimate stress and strain. At 80°C the continuous service temperature level at the sender unit, CARILON Polymer RDP 205 exhibits compared with PA12 a very good retention of properties in diesel/RME and RME. CARILON Polymer RDP 205 outperforms PA 12 in dimensional stability, stiffness and snappability characteristics. At 80 °C the long term ultimate strain behaviour of CARILON Polymer RDP205 in diesel/RME (85/15) v/v is acceptable, but improvement is needed if performance above 80 °C will be required.

The excellent ageing properties of CARILON Polymer in RME and diesel/RME (85/15 v/v) at 60 °C and 80°C also confirm the natural fit of CARILON Polymer in diesel fuel systems.

January, 1998

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LONG TERM BEHAVIOUR OF CARILON POLYMER AND COMPETITIVE POLYMERS IN AUTOMOTIVE FLUIDS

1. Introduction

Long term performance data on CARILON Polymer are needed to support our customers in the automotive industry in their decision making process on material specifications for fuel system parts. At SRTCA a long term ageing program of CARILON Polymer and competitive materials in automotive fluids is being carried out. The 'CARILON ageing program' comprises:

1) The resistance of CARILON Polymer and HDPE to standard diesel fuel (See: CA.97.20523),
2) The resistance of CARILON Polymer, HDPE and PA12 to RME and diesel/RME fuel (subject of this report),
3) The resistance of CARILON Polymer and HDPE to EOLYS catalytic oxidation converter for diesel fuel (The next report in this series).

1.1 The resistance of CARILON Polymer, HDPE and PA12 to RME and diesel/RME

As explained in the first report[1] not only the temperature regime in the fuel system of compression ignition engines (diesel engines) increases, also the aggressiveness of the modern and future diesel fuels increases. One category of these modern fuels is the so called bio-diesel fuel[2]. Characteristic for bio-diesel fuels is that they contain component(s) of vegetable crops.
In general bio-diesels can comprise the following constituents:

1) vegetable oils or blends of oils,
2) methyl esters of vegetable oils and blend thereof, and
3) blends of vegetable oils or methyl esters with conventional (crude oil based) diesel fuel.

For this study we have selected rape seed methyl ester (RME) and a blend of diesel/RME (85/15 v/v). Such type of fuels are currently used, amongst others in France and Germany and are known to be aggressive to some polymers.

The polymeric materials selected for this standard diesel ageing program are:

1. CARILON Polymer RDP 205 (New name: D26HM100)
2. HDPE Lupolen 5021D Q425 ex BASF and
3. PA12 Rilsan A AMNOTLD ex ELF.

HDPE was selected because this material is the current standard material used for the manufacture of blow-moulded fuel tanks for passenger cars. PA12 represents one of the polyamide polymer types used for the manufacture of fuel lines, an other attractive market segment of the fuel system in which CARILON Polymer offers potential.

The primary ageing medium selected is RME, supplied by Novaol (France). In the blend standard diesel fuel CEC-RF-90-A-92 was used[2] at a diesel/RME ratio of 85/15 v/v, which is used in France. As a reference medium air was included in the program. This was included in order to be able to compare the results obtained in this program with data obtained in earlier programs[3].

In RME and diesel/RME the ageing temperatures selected were: 60 and 80 °C:

- 60 °C was selected for HDPE as this temperature is the envisaged maximum operation temperature limit for HDPE diesel fuel tanks. (Earlier work[1] has shown that in diesel above 60 °C the modulus of HDPE decreases to < 50% of its initial value within 100 hours)
- 80 °C was selected for CARILON Polymer and PA12. 80 °C is the envisaged continuous service temperature for fuel lines and sender units of fuel systems of modern direct injection diesel engines.

In air the selected ageing temperatures are 23 °C, 60 °C and 80°C.

- 23 °C was selected as a reference temperature. It simulates the idle time of an empty fuel system. These data allow a quantification of the physical ageing behaviour of the polymers in this test series.
- 60 °C was selected for testing HDPE, and
- 80 °C was selected for testing CARILON Polymer and PA12, giving additional data to the results of the first report[1] of this series.

Test intervals selected initially were: 0, 100, 500, 1000, 2000, 3000 hours. In discussions with the automobile industry on the long term ageing in fuels of CARILON Polymer, it appeared that ageing data up to 5000 hours ageing were required. Therefore we have extended for the CARILON Polymer series and the HDPE series the ageing time from 3000 h to 5000 h by carrying out an additional ageing program. The results of that additional program are included in this report. For PA12 this was not possible as no test specimens of that material were left.

The testing conditions selected for this program are in agreement with ISO 175. Test conditions for determination of the properties: 23 °C ± 2 °C and 50 % ± 10 RH. To avoid confusion the terms test temperature and ageing temperature will be used throughout this report.

2. Experimental

The details on the materials, specimen manufacture and conditioning, ageing media, test conditions and monitoring tests are given in the following sections and are summarised in the Appendices 2 A to C, 3 A and B and 4 A to D.

2.1 Polymer materials used

The polymers selected for this study are given in the table below:

Table 1. Polymers tested.

Nr	Polymer	Identification	Supplier	Batch/lot number	Date Injection Mouldin
1	PK	CARILON Polymer RDP 205	Shell Chemicals	05WMA062	05-07-96
2	PA12	Rilsan A AMNOTLD	ELF	Lot 311337	08-02-96
3	HDPE	Lupolen 5021D Q425	BASF	Lot 463-309043	05-07-96

2.2 Injection moulding

Before injection moulding the CARILON Polymer, PA12 and the HDPE were dried in special gauze trays in a vacuum oven at the following conditions: 400 mbar, 60 °C and 16 hours. Specimens were then moulded using a Battenfeld BA 250/050 with UNILOG 4000 A2 system injection moulding machine. A multi-cavity mould with a turn-key gating system set at the tensile specimen cavity, in accordance with ISO527, has been used. The moulding details of these three series of specimens are given in Appendix 1.

2.3 Specimen identification and conditioning

All specimens have been coded according to the overviews given in the Appendix 2 and Appendix 4 using an engraving device. Thereafter the specimens were conditioned for at least 3 days at 23 °C and 50 % RH.

2.4 Ageing media and ageing conditions

The media used in this study are given in the table below:

Table 2 Media used for the ageing tests.

Nr	Medium	Identification	Supplier	Batch/Lot number
1	Diesel	RF-90-A-92	Haltermann	Batch 6
2	RME	RME Lims ref. 95-05708	Novaol	Batch 5248
3	Air	---	----	---

The conditions of the ageing tests are carried out in accordance with ISO 172 and are summarised in Appendices 2 to 4. At regular intervals during the ageing period the cylinders with the ageing specimens were stirred, but contrary to ISO 172 the ageing media were not refreshed during the ageing testing.

Testing conditions:
In RME and diesel/RME 85/15 %v the ageing temperature is: 80 °C for CARILON RDP 205 and PA12. HDPE is tested in RME only at 60 °C. In air the selected ageing temperatures are 23 °C for all polymers, 60 °C for HDPE and 80°C for CARILON RDP 205 and PA12.

Test intervals of all series can be found in the Appendices 2 to 4.

Specimens aged in RME and diesel/RME were placed in racks ensuring that these specimens were standing vertically and free from each other in stainless steel cylinders. Immediately before the start of the ageing the cylinders containing the specimens were filled with RME or diesel/RME such that the specimens were fully submerged. Then the cylinders were closed and placed in air circulating ovens set at the required ageing temperature. A drawing of the racks and the cylinders is added in Appendix 5.
At each ageing period the required cylinders were withdrawn from the ovens and allowed to cool for 15 to 30 minutes. Then the cylinders were opened carefully and the racks with the specimens lifted from the cylinders. Each specimen was then rinsed 5 to 10 seconds in a 100 ml glass cylinder filled with toluene (99 %) at room temperature, followed by carefull drying with a soft clean cloth. Thereafter these specimens were tested.

Specimens aged in air were placed horizontally in the oven, free from each other, on an open wire rack. After each ageing period the required specimens were taken from the rack. No further treatment was given before the testing. To avoid any cross-contamination each polymer series was aged in a separate oven.

2.5 Monitoring tests

The monitoring tests are given below:

2.5.1 Tensile test

The tensile tests were performed on an Instron 4507 tensiometer according to ISO 527. At least 5 specimens were tested, the tests were carried out at 23 °C ± 1 °C and a relative humidity between 40 and 60%. The machine parameters are given in Table 3:

Table 3. Tensile test parameters

Parameters	
Grip distance (mm)	115
Extensometer	Long travel type, 0 to 500 % extension
Grip length (mm)	50
Crosshead speed (mm/min)	100

The following tensile properties were calculated: secant modulus, yield stress, yield strain, stress at break and strain at break. As the available data acquisition system allows only a limited number of data points, the calculation of Youngs modulus is not possible. However reproducible data on the stiffness of the materials can be obtained using the secant modulus calculated between 0 and 2 % strain.

2.5.2 Mass and dimensions

For the mass change and dimensional change a separate series of specimens were aged and treated under conditions given in section 2.4, however these specimens were replaced after testing. Before the ageing started the specimens were conditioned for minimum 3 days at 23 °C and 50 % RH. At each test period the specimens were taken from the ovens or cylinders and directly after cooling to 23 °C the mass and the dimensions determined. The mass change is calculated as a percentage of the mass change relative tot the initial mass. The dimensional change is calculated as a percentage of the dimensional change relative tot the initial dimension.

2.5.3 Yellowness index

To monitor change in colour the DIN 6167 standard has been selected. The measurement was carried out on a Data Color 3890 using the manufacturer's standard supplied software for the calculation of the yellowness index. The measurements were carried out on at least five tensile specimens immediately before they were tested for their mechanical properties.

3. Results and discussion

All results summarised in the Tables 4 to 9 and all basic data are available in SRTCA CTCAR/2 laboratory book PRP/832/95, appendix 50. The graphical presentation of the results is given in the figures 1 to 24 in the sections 3.1 and 3.2. Data points in the figures are connected with lines for no other purpose than guiding the eye.

First we will discuss the changes in mass and dimensions followed by the changes in tensile behaviour and yellowness. Each of these aspects will be discussed for CARILON RDP 205, PA12 and HDPE. Finally a comparison of the all polymers with respect to ageing is given.

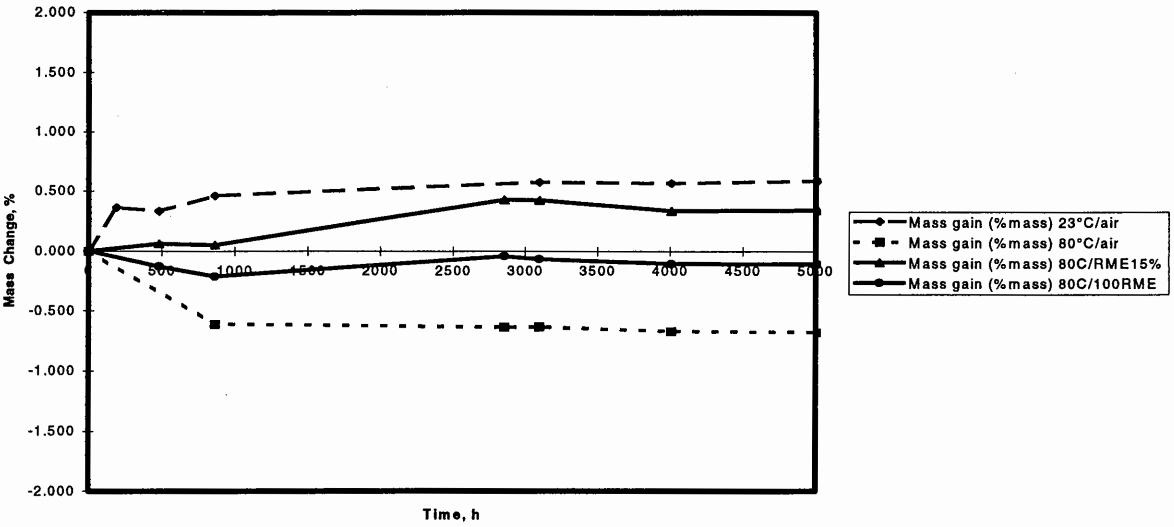
3.1 Effect of ageing on the mass and dimensions of CARILON RDP 205, PA12 and HDPE

All results of the measurements of ageing on the mass and dimensions of CARILON RDP 205, PA12 and HDPE in the Tables 5, 7 and 9.

The changes in mass for CARILON RDP 205 are given in Figure 1. The results show that after 3000 hours a plateau is reached and the changes in mass in diesel/RME are very small (+0.3%), in RME the changes are even smaller and vary between -0.2% and -0.1%.

In air at 23 °C and 50 % RH a plateau in mass increase of 0.5 % is found after 1000 hours. This indicates that the 3 days conditioning before the start of the ageing tests was insufficient to reach an equilibrium. At 80 °C in air a mass change plateau of -0.6 % is found, thus the total volatile matter in the CARILON RDP 205 samples after a long period of conditioning (>3000h) is in the order of 1.1 %. This has not been observed before, a value of 0.6 % to 0.7 % would be more in line with earlier moisture absorption measurements[4]. However these earlier measurements were carried out over a shorter period and at different temperatures.

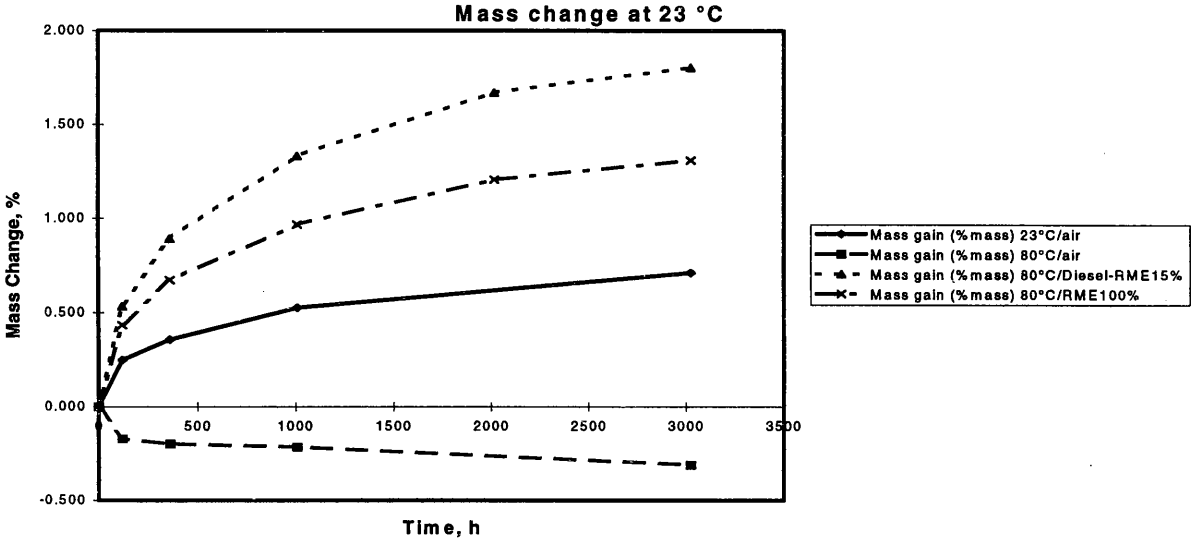
Figure 1. Effect of RME and RME/diesel on CARILON Polymer RDP205, Mass change at 23 °C



For PA12 the mass change in diesel/RME 85/15 is substantial: after 3000 hours a 1.8% mass increase is observed, for RME a 1.2 % mass increase is observed after that period. It is important to realise that at that ageing period the mass change is not yet stabilised, an increasing trend is observed. Thus the further flexibilising effect of the Diesel and RME on the PA12 should be expected.
In air at 23 °C the mass increase after 3000 hours is about 0.6 %, and an increasing trend is noticed at this point in time. Thus the moisture pick-up, a well known phenomenon of polyamides, is not yet stabilised after 3000 hours at 23 °C and 50 % RH.

At 80 °C in air the mass change is -0.3 % after 3000 hours indicating a loss of moisture.

Figure 2.Effect of RME and Diesel/RME 15% on PA12,
Mass change at 23 °C



For HDPE the mass change at 60 °C in RME is very high: 5.5 %. This plateau value is reached after 100 hours exposure to the RME. Therefore a high level of flexibilisation is to be expected for HDPE after 100h at 60°C.
In air at 23 °C and 50 % RH and at 60 °C the mass changes are negligible over the ageing period of 3000 hours.

Figure 3. Effect of RME on HDPE,
Mass change at 23 °C

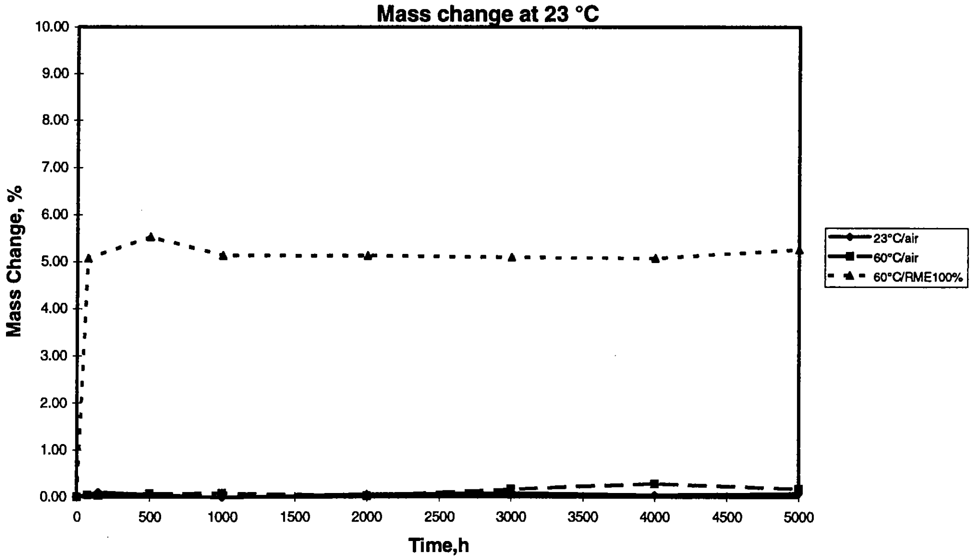
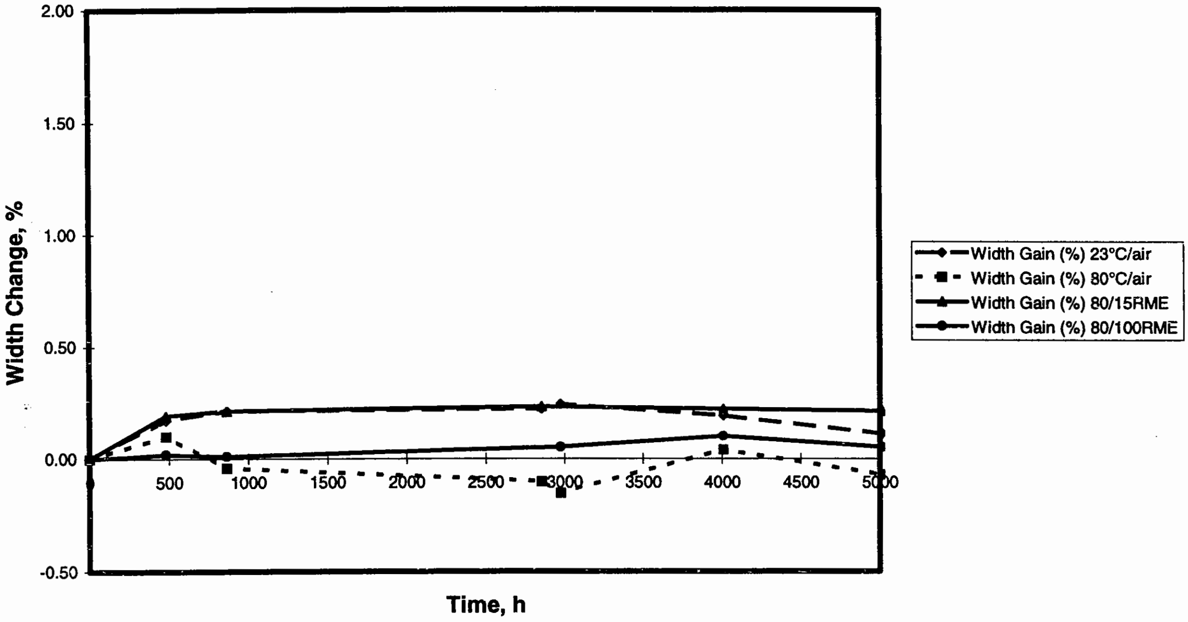
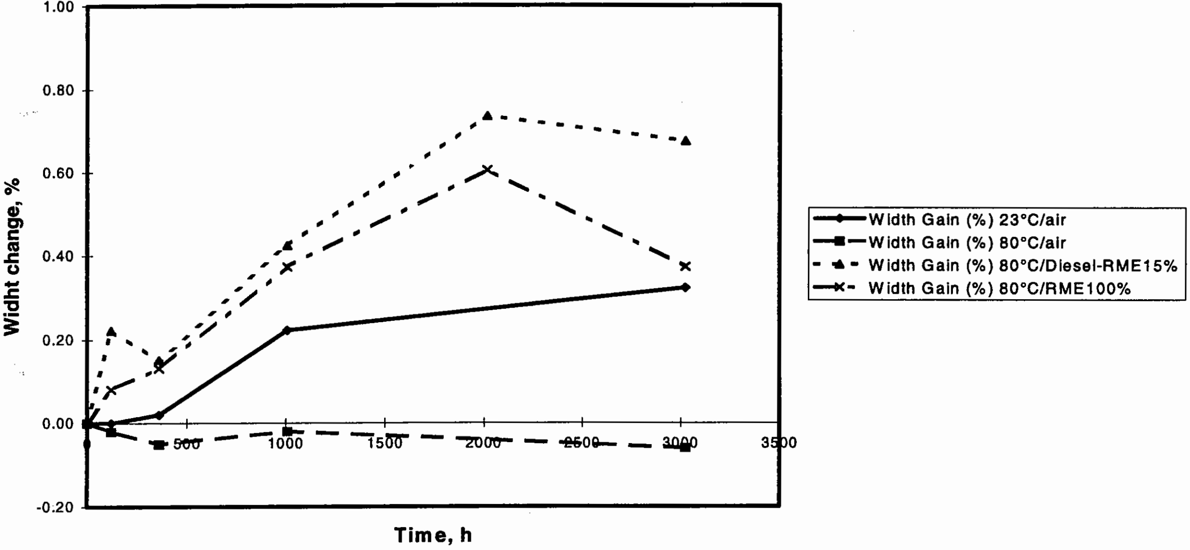


Figure 4. Effect of RME and RME/diesel on CARILON Polymer RDP205, Dimensions, Width Change at 23 °C



The dimensional changes are determined at the width of the specimens and the results are given in the figures 4,5 and 6.
For CARILON Polymer RDP 205 (Figure 4) the changes in width aged at 80 °C in diesel/RME are small; a plateau value of 0.2% is detected after 500 hours. In RME the change is < 0.1%.
In air 23 °C and 50 % RH the change in width is after 500 hours at a plateau value of 0.2 % in air at 80 °C the dimensional changes are very small, < 0.1%.

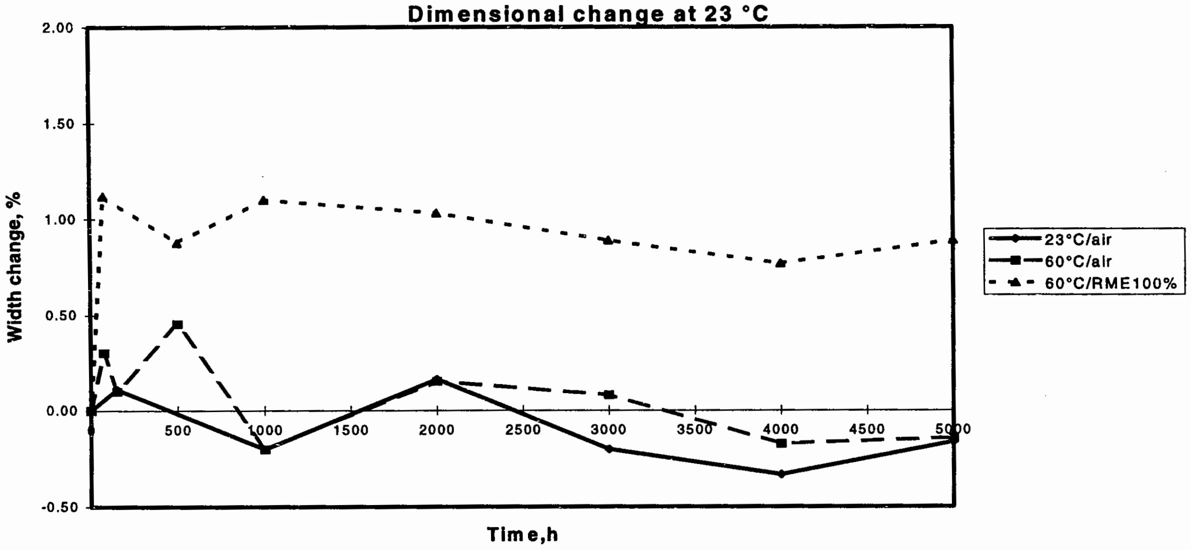
Figure 5. Effect of RME and Diesel/RME 15% on PA12, Dimensional change at 23 °C



For PA 12 the changes in width (dimensions) in diesel/RME and RME show the same trends as the changes in mass, indicating the absorption of these two types of diesel engine fuels. The width change is in diesel/RME aged at 80 °C shows a maximum value after 2000 hours at about 0.8 %, in RME at 80 °C the width change show at 2000 hour also a maximum of about 0.5%.

In both cases the width change shows a decrease, this is unexpected as the mass change still shows an increase after 2000 hours ageing. It is also clear that the dimensional changes for PA 12 are clearly higher than for RDP205. Aged in air at 23 °C and 50 % RH the width change shows a gradual increase to about 0.3% after 3000 hours. Aged in air at 80 °C the change in dimensions over 3000 hours is negligible.

Figure 6. Effect of RME on HDPE, Dimensional change at 23 °C



The change in dimensions of HDPE in RME at 60 °C are as expected high, a plateau value of width change of about 1% is found. The plateau value is reached rapidly: within 100 hours. In air at 23 °C and 50% RH and at 60 °C the changes are negligible.

3.2 Effect of ageing on the tensile properties of CARILON RDP 205, PA12 and HDPE

In this part the ageing effect of diesel/RME and RME on the tensile behaviour and the changes in yellowness of RDP 205, PA12 and HDPE will be discussed. The set-up of this part of the work is given in the Appendix 3, the coding, and arrangement of the specimens in Appendix 4 and the measured data in the Tables 4 to 9.

The changes in secant modulus over the ageing period for CARILON RDP 205 are given in Figure 7. Aged at 80 °C in diesel/RME 85/15 and RME 100% the change is very small, the level of the secant modulus is respectively 1200 and 1400 MPa. This plateau value is reached after 500 hours. In air at 23 °C and 50 % RH first a decrease is observed untill 1000 hours, thereafter up to 3000 hours an increase, indicative for a physical ageing effect. For ageing in air at 80 °C the secant modulus of CARILON RDP 205 first shows a steep increase up to 1500 MPA in the first 500 hours, then the modulus shows a very slow increase. This phase of very slow increase may be caused by a very slow crosslinking reaction[5].

The change in secant modulus of PA12 (Figure 8) is the same in diesel/RME and RME, in both cases a decrease is found after each period of testing, the changes are the highest in the beginning. The loss in modulus is about 25 %. It is caused by the plasticising effect of the diesel/RME or RME. The change in secant modulus of PA 12 aged in air at 23 °C also shows a decreasing trend, in this case the absorbed moisture causes the plasticising effect observed. Aged at 80 °C the secant modulus of PA12 first shows a steep increase to about 1400 MPA in the first 500 hours, then the modulus shows a very slow increase.

Figure 7. Effect of RME and RME/diesel on CARILON Polymer RDP205, Secant modulus at 23 °C

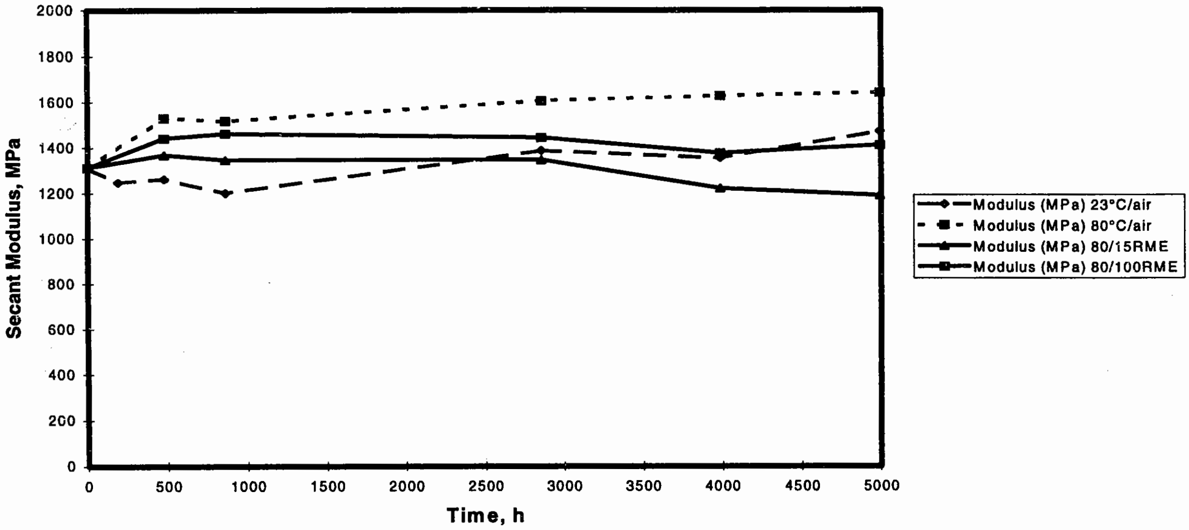
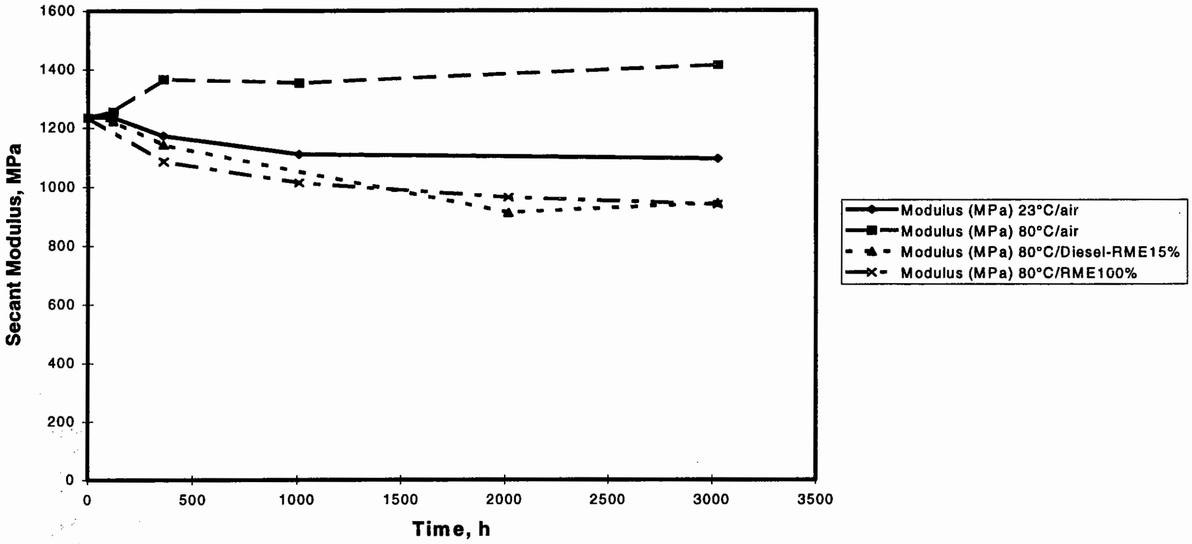
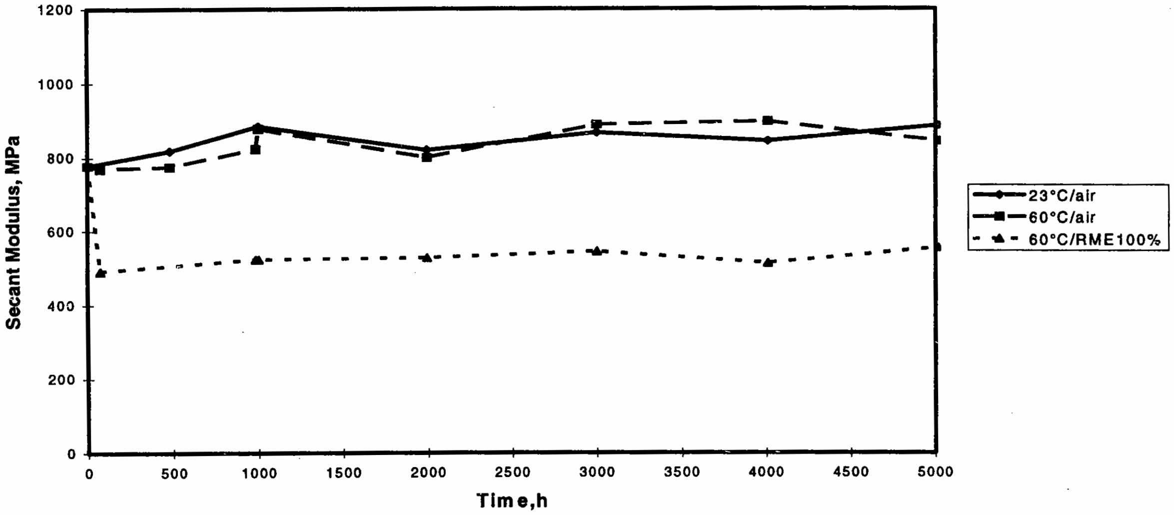


Figure 8. Effect of RME and Diesel/RME 15% on PA12, Secant Modulus at 23 °C



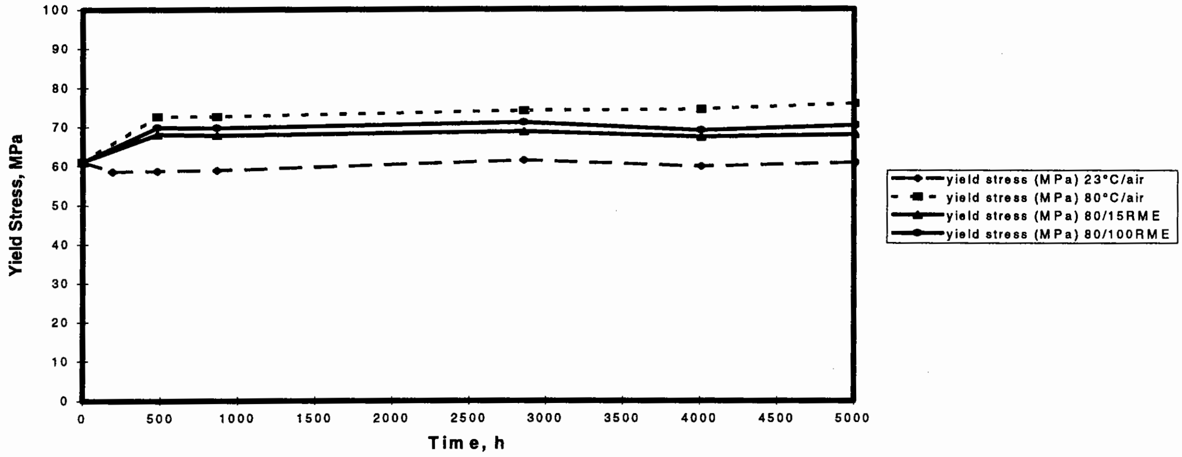
The change of secant modulus of HDPE on ageing in RME (Figure 9) shows a severe decrease in the first 100 hours from about 800 to 500 MPa, at about 500MPa a plateau is reached. Again the explanation is the plasicising effect of the RME. The changes on ageing in air are negligible.

Figure 9. Effect of RME on HDPE, Secant Modulus at 23 °C



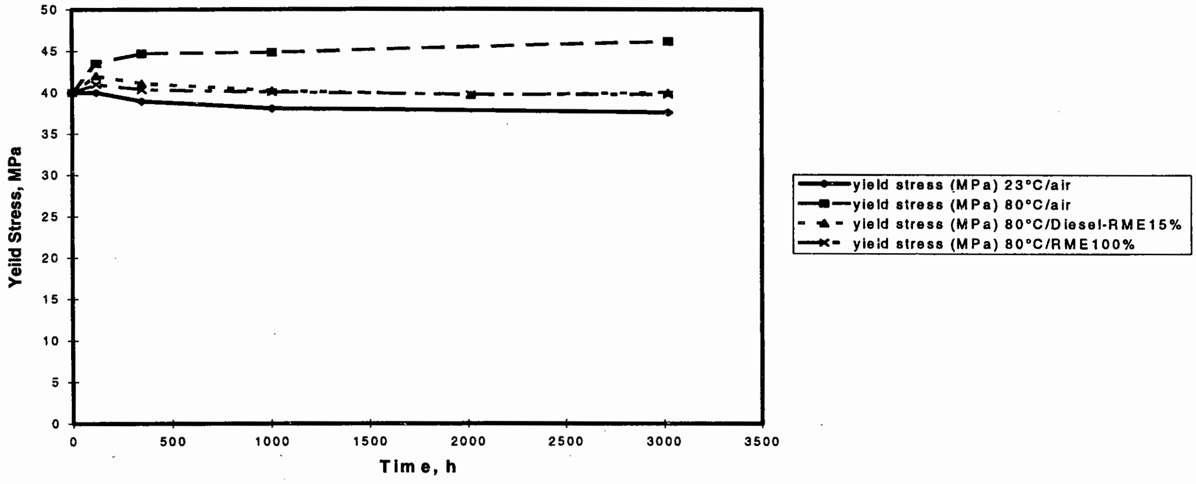
The effect of ageing on the yield stress of CARILON RDP 205 is shown in Figure 10. Ageing in diesel/RME and RME gives comparable results, initially up to 500 hours an increase, thereafter a plateau at a level of agout 70 MPa. In air at 23 °C no changes are observed. In air at 80 °C the results are comparable to diesel/RME but at a slightly higher level.

Figure 10. Effect of RME and RME/diesel on CARILON Polymer RDP205, Yield Stress at 23 °C



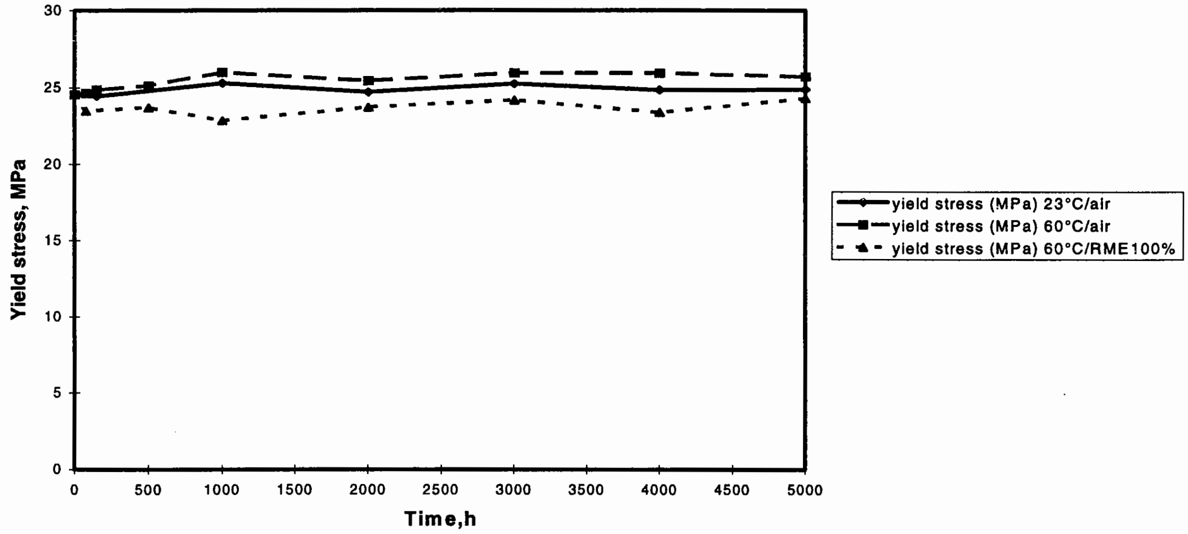
The effect of ageing on the yield stress of PA12 is shown in figure 11. In diesel/RME and RME at 80 °C P12 shows in 3000 hours no change of the yield stress, in air at 23 °C a slight loss is observed. In air at 80 °C the yield stress increases in the first 500 hours from 40 to 45, than a plateau value is reached.

Figure 11. Effect of RME and Diesel/RME 15% on PA12, Yield Stress at 23 °C



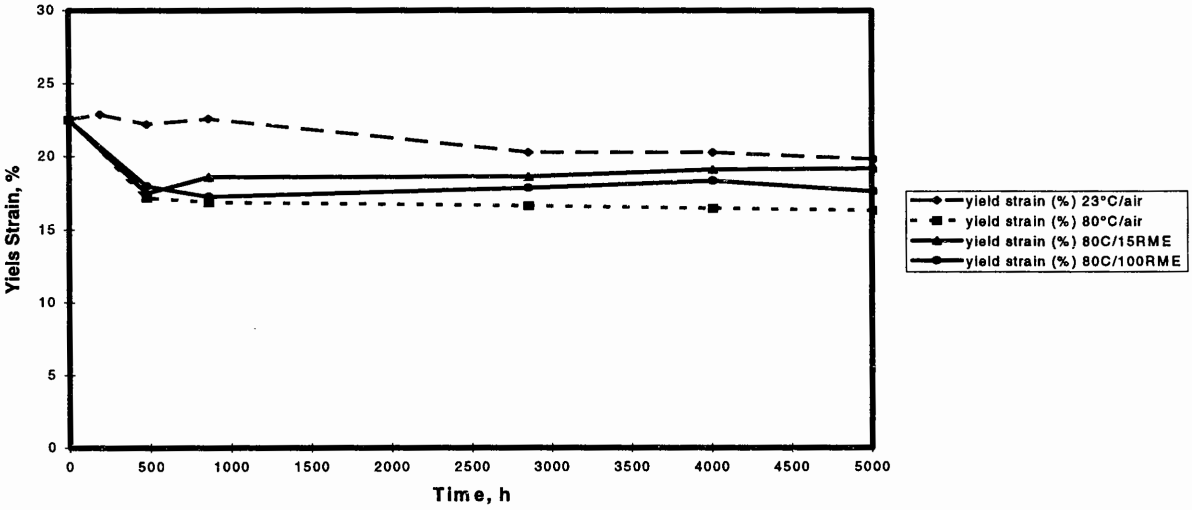
No or only a marginal change in yield stress is found for HDPE aged in RME at 60 °C, aged in air at 23 °C 50% RH and air at 60 °C (Figure 12).

Figure 12. Effect of RME on HDPE, Yield Stress at 23 °C



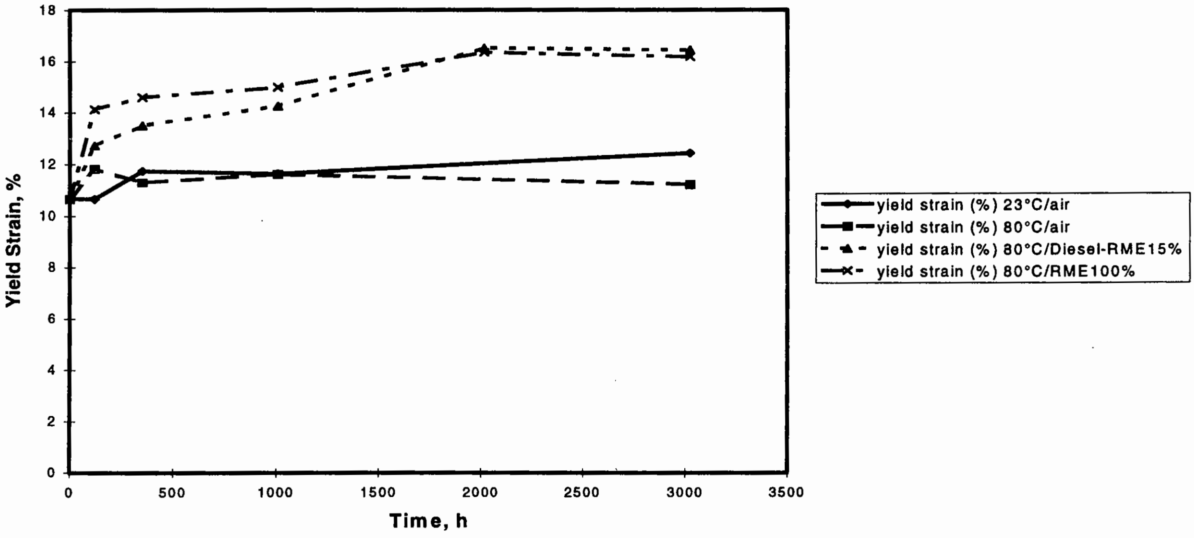
The yield strain of CARILON Polymer RDP 205 aged at 80 °C in diesel/RME and RME decreases in the first 500 hours from 22 % to 18%, then a slight increasing gradient is observed. In air at 23 °C and 50 % RH a very gradual decrease is observed over the 3000 hours test period: form 22 % to about 19 %. In air at 80 °C the yield strain change is comparable to that of the diesel/RME and RME.

Figure 13. Effect of RME and RME/diesel on CARILON Polymer RDP205 Yield Strain at 23 °C



The change of the yield strain of PA12 differs clearly form CARILON RDP 205. Aged at 80 °C in diesel/RME and RME the yield strain increases from initially 11 % to 16 % after 3000 hours. Nevertheless this value is still below the yield strain of CARILON RDP 205. The yield strain in air at 23 °C 50 % RH and in air at 80 °C is more or less unchanged over the 3000 hours of test.

Figure14. Effect of RME and Diesel/RME 15% on PA12, Yield Strain, % at 23 °C



The yield strain of HDPE aged in RME at 60 °C changes in the first 100 hours from 12% to 25 %, thereafter a plateau is reached at 25 %. This can be explained by the plasticising effect of the RME. No change is observed for the ageing results in air at 23 °C 50% RH and in air at 80 °C.

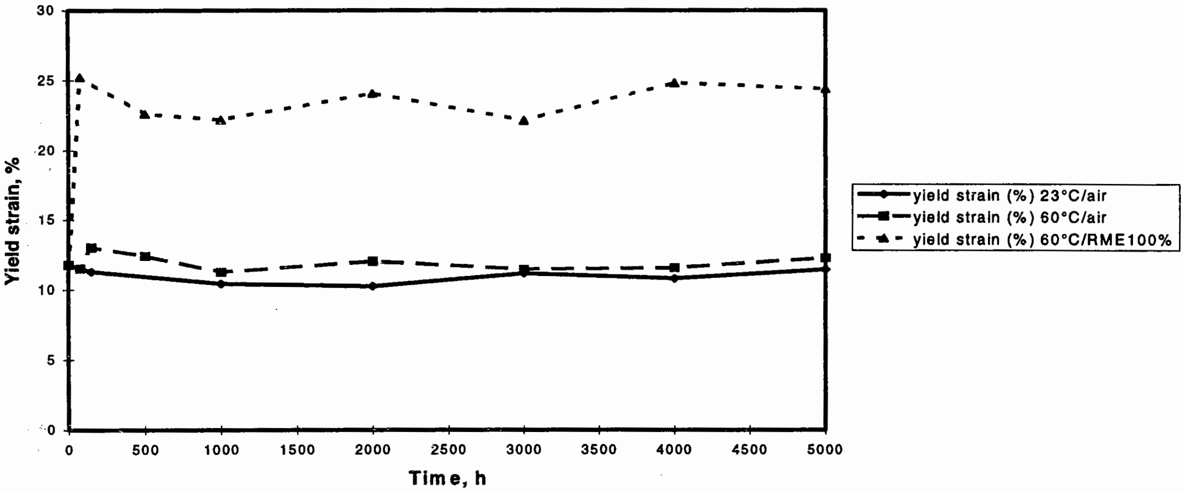
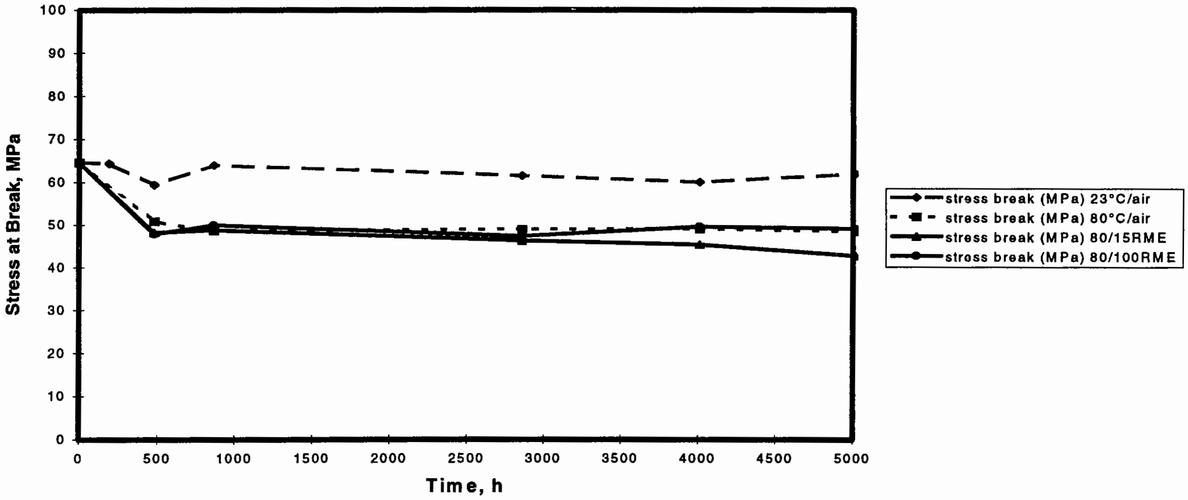


Figure 15. Effect of RME on HDPE, Yield Strain at 23 °C

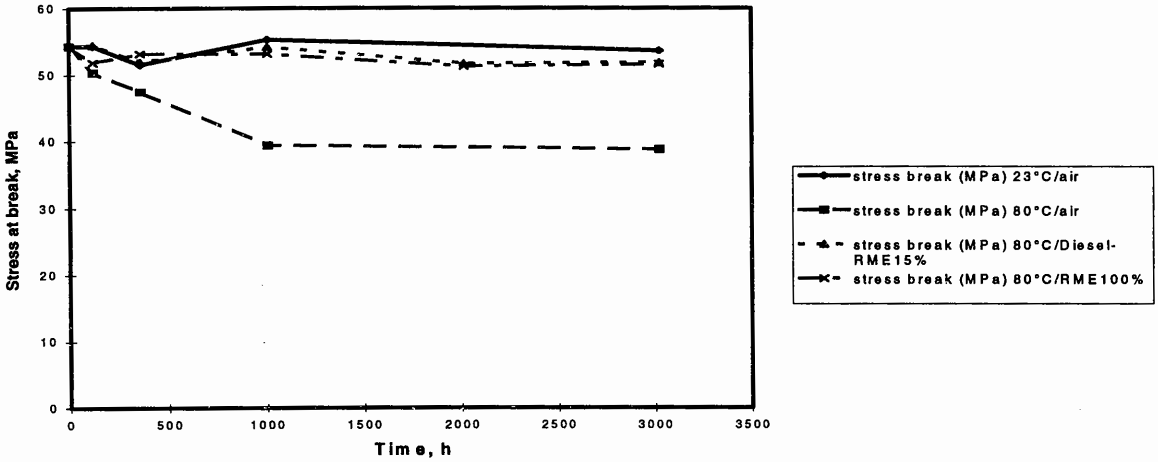
The stress at break data of CARILON RDP 205 (see figure 16) show a clear decrease in the first 500 hours from 64 to about 50 MPa, thereafter a plateau value is reached for diesel/RME, RME and air at 80 °C. No changes are observed are observed in air at 23 °C 50 % RH.



Fugure 16. Effect of RME and RME/diesel on CARILON Polymer RDP205, Stress at Break at 23 °C

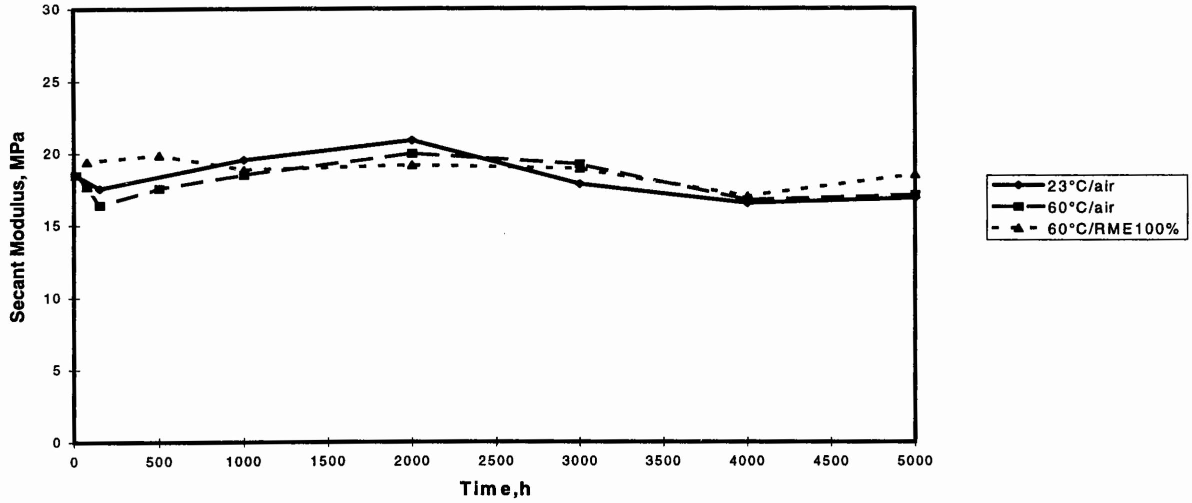
Virtual no change of stress at break of PA12 has been found for diesel/RME, RME and air at 23 °C and the level is about 55 MPa (Figure 17). In air at 80 °C a substantial decrease is found in the first 1000 hours of ageing: from 55 MPa to 38 MPa, thereafter a plateau is observed.

Figure 17. Effect of RME and Diesel/RME 15% on PA12, Stress at break at 23 °C



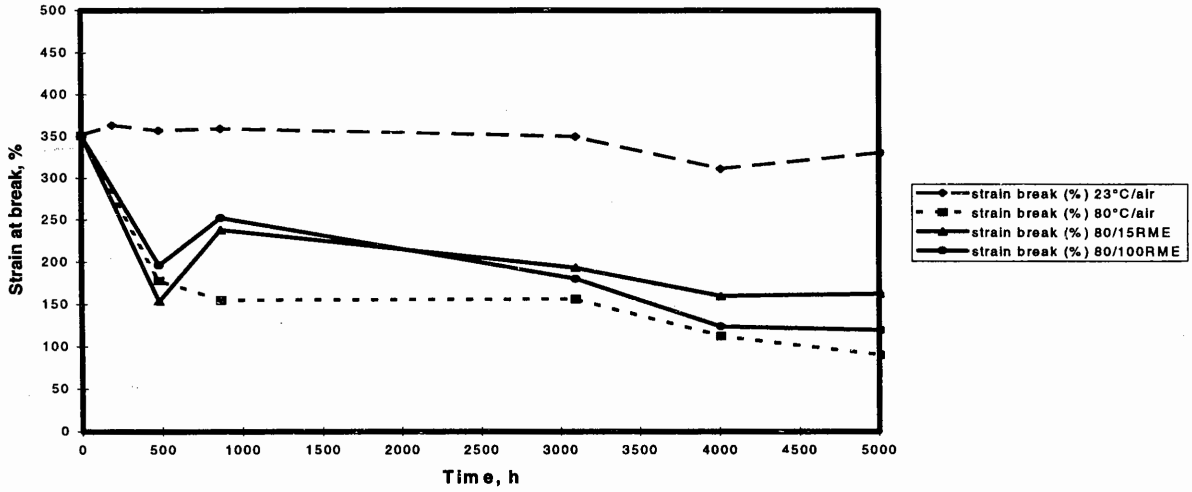
Ageing in air at 23 °C 50 % RH, air at 60 °C and RME at 60 °C has no effect on the break stress of HDPE (Figure 18).

Figure 18. Effect of RME on HDPE, Break stress at 23 °C



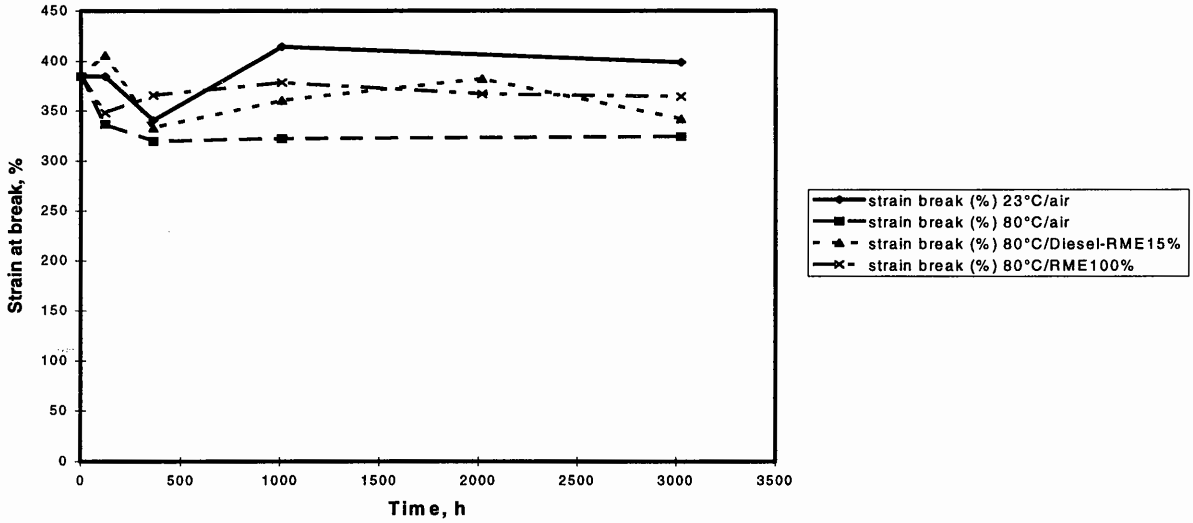
The effect of ageing on the strain at break of CARILON RDP 205 is given in the Figure 19. The results show that ageing at 80 °C in diesel/RME, RME and air have a drastic effect on the strain at break, a loss from about 350 % to about 175 % is found in the first 500 hours. Thereafter a trend of gradual decrease of the strain at break is observed. After 300 hours a strain at break level of about 150 % is found. Also it should realised that the scatter of the results of the strain at break is always higher than for properties like the modulus or the yield stress and strain (see Table 4).

Figure 19. Effect of RME and RME/diesel on CARILON Polymer RDP205, Strain at Break at 23 °C



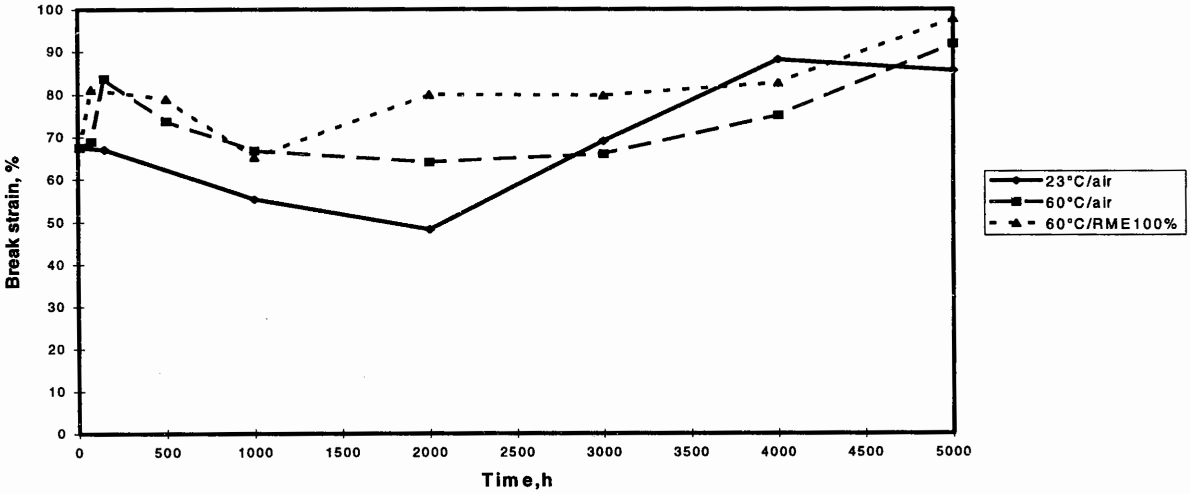
For PA 12 effect of ageing in diesel/RME and RME at 80 °C on the strain at break (Figure 20) is small, the value changes from about 380% to 340%, and most of that change is in the first 500 hours. Ageing in air at 23 °C 50 % RH has no or only a marginal effect on the strain at break. Ageing in air at 80 °C does result in a decrease of the strain at break from about 380 % to about 320 %.

Figure 20. Effect of RME and Diesel/RME 15% on PA12, Strain at break at 23 °C



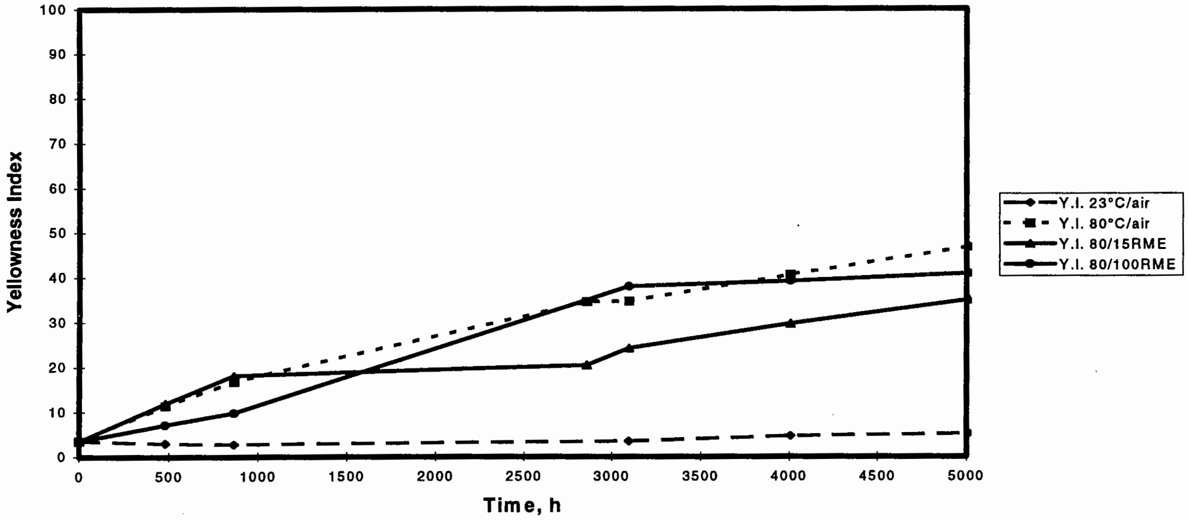
For HDPE the effect of ageing in RME at 60 °C (Figure 21) on the strain at break is rather small if we take in account that HDPE shows a substantial increase in mass over the ageing period. The strain at break increases from about 70 % to about 95%. In air a comparable trend in the change of the strain at break is found.

Figure 21. Effect of RME on HDPE, Break strain at 23 °C



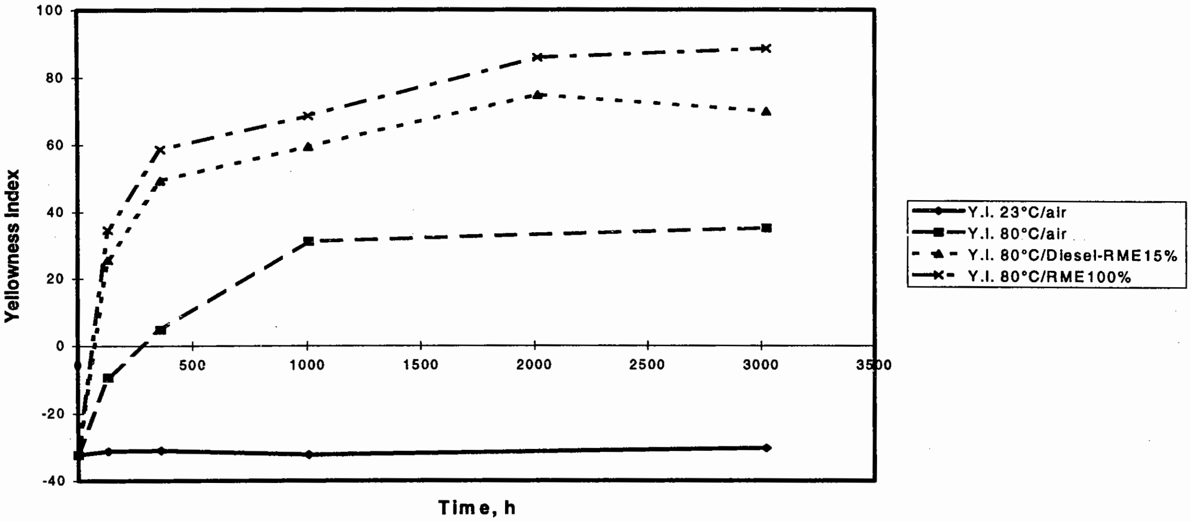
The effect of ageing on the yellowness of CARILON RDP 205 is shown in Figure 22. The yellowness changes in diesel/RME, RME and air are comparable, a steady increase form 4 to about 40 is found. In air at 23 °C 50 % RH no change is found.

Figure 22. Effect of RME and RME/diesel on CARILON Polymer RDP205, Yellowness Index DIN 6167, 23 °C



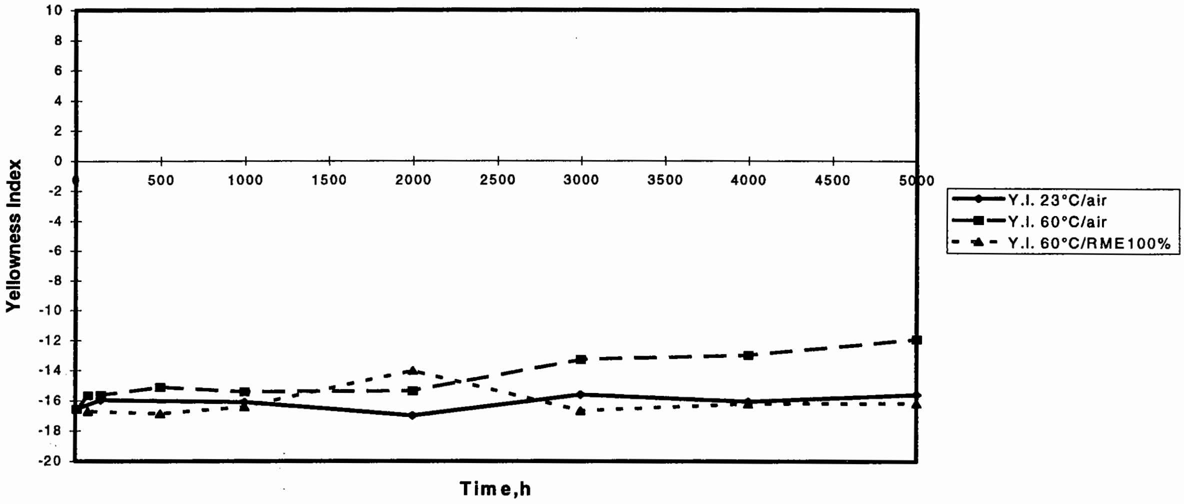
The changes in yellowness due to ageing of PA 12 are given in Figure 23. The changes in diesel/RME and RME are comparable, a fast change in the first 500 hours and thereafter a slow increasing or stabilising trend. The yellowness changes from -30 to 80, which is higher than for CARILON RDP 205. In air at 80 °C the change in yellowness is lower, from -30 to 30. The yellowness does not change when aged in air at 23 °C 50 % RH.

Figure 23. Effect of RME and Diesel/RME 15% on PA12, Yellowness Index at 23 °C



The effect of ageing on the yellowness of HDPE (Figure 24) in RME at 60 °C is very small and negligible in air.

Figure 24. Effect of RME on HDPE, Yellowness Index at 23 °C



3.3 Comparison of ageing behaviour of CARILON RDP 205, PA 12 and HDPE

Comparing the dimensional changes after ageing of CARILON RDP205, PA12 and HDPE in RME (100%) it is clear that CARILON RDP 205 performs substantially better: after 3000 hours the dimensional change of PA 12 is about 4 times higher than CARILON RDP 205, the dimensional change of HDPE is about 10 times higher than CARILON RDP 205.The dimensional changes in diesel/RME are of the same order but only tested for PA12. In air the dimensional changes at the ageing temperatures 80 °C or 60 °C are negligible for all three polymers, at 23 °C and 50 % RH the dimensional stability differs slightly, PA shows as expected the highest change, HDPE the lowest change.

For CARILON RDP 205 aged 3000 hours in RME at 80 °C a change in mass of -0.1% corresponds with a change in dimensions of 0.1%, aged in diesel/RME at 80 °C a change in mass of 0.4% corresponds with a change in dimensions of 0.2%. For PA12 aged 3000 hours in RME at 80 °C a change in mass of 1.2% corresponds with a change in dimensions of 0.4%, aged in diesel/RME at 80 °C a change in mass of 1.7% corresponds with a change in dimensions of 0.7%. From this follows that the diesel fuel has more effect in the mass and dimensional changes than the RME.

Comparing the tensile secant moduli at 23 °C, of the three polymers CARILON Polymer RDP 205 performs best, in all cases it shows the highest secant modulus. If we compare the initial secant moduli of the other polymers tested with CARILON RDP 205, PA12 shows a retention of 95%, HDPE a retention of 60 %. After 3000 hours ageing in diesel/RME or RME, PA12 shows a retention of 70 %, HDPR a retention of 40 %. Thus ageing in diesel/RME or RME is much more aggressive for PA12 and for HDPE than for CARILON RDP205. The advantage in stiffness of CARILON RDP 205 can be exploited in design of components where at equal stiffness a CARILON component requires a lower thickness and thus lower mass.

CARILON RDP 205 maintains after 3000 hours ageing at 80°C in diesel/RME and RME a substantially higher yield stress and strain than PA12 and HDPE. Thus under the conditions of test snapfit joints of CARILON RDP 205 will perform much better than those of PA12 or of HDPE.

The ultimate stress of CARILON RDP 205 and PA12 after ageing 3000 hours in diesel/RME and RME at 80 °C are comparable, however the ultimate strain of PA12 is clearly better. Under these conditions the ultimate strain retention of CARILOM RDP 205 is 53 %, the ultimate strain retention of PA 12 is 88% and the ultimate strain retention of HDPE, after ageing 3000 hours in RME only, at 60 °C, is 117 %. In air at elevated temperatures the retention of the ultimate strain after ageing for CARILON RDP 205 is 45%, for PA12 it is 80%. Moreover the initial ultimate strain is for PA12 400% and for CARILON RDP 205 350 %. This lower retention of ultimate strain is obviously a weakness of CARILON Polymer RDP205 which has been observed in the first part of this program and in earlier programs on the thermal and oxidative stability. Although the minimum retention levels required by our customers are not known it can be expected that the industry will require retention levels of 50 % or above over the full lifetime of a component. Therefor this aspect needs further attention.

As far as the yellowing is concerned HDPE performs substantially better than CARILON RDP 205 and PA12. This observation shows that a dark yellow or brown surface colour of neat fuel system components is not necessarily an indication of loss of performance or degradation of such a component. Moreover this is a strong argument for using black coloured CARILON in this application field.

4. Conclusions

- At 60 °C, the envisaged continuous operation temperature of the fuel tank, CARILON Polymer RDP205 exhibits compared to HDPE an excellent retention of properties in RME. CARILON Polymer RDP 205 outperforms HDPE in dimensional stability, stiffness and snappability characteristics and ultimate stress and strain.

- At 80°C the envisaged service temperature level of fuel lines and sender units, CARILON Polymer RDP 205 exhibits compared to PA12 a very good retention of properties in diesel/RME and RME. CARILON Polymer RDP 205 outperforms PA 12 in dimensional stability, stiffness and snappability characteristics.

- At 80 °C the long term ultimate strain behaviour of CARILON Polymer RDP205 in diesel/RME 85/15 v/v is considered acceptable (confirmed by recent customer feedback), PA12 shows in this respect a better performance. Improvement of the long term ultimate strain behaviour of CARILON Polymer RDP205 in diesel/RME 85/15 v/v is needed if performance above 80 °C will be required.

5. Recommendations and further work

Based on the conclusions above it is recommended to further exploit the excellent performance of CARILON Polymer in modern diesel fuel system applications where diesel/RME and RME are used. CARILON Polymer clearly shows a natural fit in the material palette available to the automotive industry for diesel fuel systems.

Further work and some proposals to further work:

- The execution and reporting of the next part of the CARILON ageing program: the resistance of CARILON Polymer and selected other polymers to EOLYS oxidation catalysts.
- The execution and reporting of the 'CARILON competitive positioning program'.
- Based on the outcome of this study further work will be required to understand the high scatter of the strain at break of CARILON Polymer after ageing in diesel and air. A study of fracture surfaces needs to be a part is this investigation.
- The effect of peak temperatures above 100 °C need to be addressed in a further study.
- The effect of ageing on impact behaviour needs to be addressed.
- Finally to mimic the real live situation of a fuel tank on a car also ageing tests using (small) tanks should be carried out on selected polymeric materials.

Amsterdam, January 1998

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Table 4. Influence of diesel/RME and RME on CARION RDP 205

time (h)	Modulus (MPa) 23°C/air	Modulus SD (MPa) 23°C/air	Modulus (MPa) 80°C/air	Modulus SD (MPa) 80°C/air	Modulus (MPa) 80/15RME	Modulus SD (MPa) 80/15RME	Modulus (MPa) 80/100RME	Modulus SD (MPa) 80/100RME	Modulus (MPa) 23/100RME	Modulus SD (MPa) 23/100RME
0	1312	60	1312	60	1312	60	1312	60	1312	60
192	1252	34								
480	1263	68	1530	70	1368	48	1442	10		
864	1210	81	1517	71	1346	68	1463	92		
2856	1386	54	1605	23	1347	20	1443	35		
2976	1389		1661	88			1503	59	1637	
3096	1457	58	1609	53	1318	105	1339	52		
4008	1354	50	1628	36	1221	64	1376	52		
5016	1471	37	1643	39	1190	56	1412	56		

time (h)	yield stress (MPa) 23°C/air	yield stress SD (MPa) 23°C/air	yield stress (MPa) 80°C/air	yield stress SD (MPa) 80°C/air	yield stress (MPa) 80/15RME	yield stress SD (MPa) 80/15RME	yield stress (MPa) 80/100RME	yield stress SD (MPa) 80/100RME	yield stress (MPa) 23/100RME	yield stress SD (MPa) 23/100RME
0	61.1	0.22	61.1	0.22	61.1	0.22	61.1	0.22	61.1	0.22
192	58.69	0.29								
430	58.75	0.13	72.67	0.82	68.03	0.24	69.86	0.3		
864	58.87	0.22	72.69	0.34	67.87	0.18	69.74	0.27		
2856	61.31	1.32	74.07	0.16	68.75	0.11	71.06	0.29		
2976	59.49	0.28	74.02	0.26			71.5	1.08	62.82	
3096	61.16	0.84	74.99	0.15	67.53	1.39	68.57	1.22		
4008	59.75	0.19	74.39	0.18	67.34	1.09	69.05	0.17		
5016	60.72	0.14	75.86	0.28	67.93	0.11	70.27	0.25		

time (h)	yield strain (%) 23°C/air	yield strain SD (%) 23°C/air	yield strain (%) 80°C/air	yield strain SD (%) 80°C/air	yield strain (%) 80/15RME	yield strain SD (%) 80/15RME	yield strain (%) 80/100RME	yield strain SD (%) 80/100RME	yield strain (%) 23/100RME	yield strain SD (%) 23/100RME
0	22.49	0.78	22.49	0.78	22.49	0.78	22.49	0.78	22.49	
192	22.59	1.34								
480	22.18	1.21	17.16	0.96	17.46	0.49	17.95	0.77		
864	22.56	2.59	16.85	1.28	18.57	1.16	17.21	0.61		
2856	20.24	1.08	16.59	0.34	18.59	0.35	17.81	0.47		
2976	20.45	0.73	15.74	0.46			17.14	0.29	18.35	
3096	19.91	0.35	16.53	0.33	18.58	0.76	18.76	0.39		
4008	20.25	0.53	16.43	0.49	19.08	0.66	18.29	0.8		
5016	19.78	0.48	16.28	0.44	19.14	0.47	17.58	0.41		

time (h)	stress break (MPa) 23°C/air	stress break SD (MPa) 23°C/air	stress break (MPa) 80°C/air	stress break SD (MPa) 80°C/air	stress break (MPa) 80/15RME	stress break SD (MPa) 80/15RME	stress break (MPa) 80/100RME	stress break SD (MPa) 80/100RME	stress break (MPa) 23/100RME	stress break SD (MPa) 23/100RME
0	64.62	11.05	64.62	11.05	64.62	11.05	64.62	11.05	64.62	11.05
192	69.3	5.49								
480	60.67	11.55	50.91	0.93	50.14	0.56	50.59	0.61		
864	64.18	9.99	50.33	1.7	50.63	0.62	50.87	0.69		
2856	62.83	12.55	54.42	9.57	49.64	1.92	49.19	1.87		
2976	58.77	8.92	49.49	2.14			50.78	1.13	57.4	
3096	58.54	12.32	49.72	1.42	49.39	1.56	49.08	2.37		
4008	59.97	112.11	49.56	1.19	46.86	1.66	50.18	8.92		
5016	61.91	11.65	48.72	2.9	47.32	2.58	49.82	0.56		

time (h)	strain break (%) 23°C/air	strain break SD (%) 23°C/air	strain break (%) 80°C/air	strain break SD (%) 80°C/air	strain break (%) 80/15RME	strain break SD (%) 80/15RME	strain break (%) 80/100RME	strain break SD (%) 80/100RME	strain break (%) 23/100RME	strain break SD (%) 23/100RME
0	350.2	13.08	350.2	13.08	350.2	13.08	350.2	13.08	350.2	13.08
192	367	5.6								
480	356.6	9.5	166.61	53.86	145.2	56.22	187.8	80.53		
864	358.3	7.8	154.1	40.2	234.3	83.2	246.9	105.2		
2856	357.42	9.65	175.17	101.04	217.11	66.49	181.61	116		
2976	337.8	18.37	71.79	34.61			125.12	72.88	357.7	
3096	349.1	7.86	156.22	82.81	193.61	103.86	179.8	103.36		
4008	311.22	35.78	112.49	50.26	160.06	84.6	124.04	91.42		
5016	330.9	55.14	89.98	70.77	162.81	92.7	119.7	44.3		

Table 5. Influence of diesel/RME and RME on CARION RDP 205

time	Y.I.	Y.I.	Y.I.	Y.I.	Y.I.
h	23°C/air	80°C/air	80/15RME	80/100RME	23/100RME
0	3.5	3.5	3.5	3.5	3.5
192					
480	3.044	11.38	11.89	7.108	
864	2.797	16.76	18.08	9.76	
2856		34.62	20.43	12.42	
2976	4.312	35.838		10.711	10.17
3096	3.494	34.711	24.23	37.967	
4008	4.627	40.543	29.754	39.116	
5016	5.152	46.817	35.063	40.82	

time	Init Wt 6 pcs	Init Wt 6 pcs	Init Wt 6 pcs	Init Wt 6 pcs	Init Wt 6 pcs	Wt 6 pcs aged	Wt 6 pcs aged	Wt 6 pcs aged	Wt 6 pcs aged	Wt 6 pcs aged
(h)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
	23°C/air	80°C/air	80C/RME15%	80C/100RME	23C/100RME	23°C/air	80°C/air	80C/RME15%	80C/100RME	23C/100RME
0	62.470	62.470	62.470	62.470	62.470	62.470	62.470	62.470	62.470	62.470
192	62.470	62.470	62.470	62.470	62.470	62.696				
480	62.470	62.470	62.470	62.470	62.470	62.6778		62.5083	62.3897	
864	62.470	62.470	62.470	62.470	62.470	62.7593	62.0881	62.5012	62.3372	
2856	62.470	62.470	62.470	62.470	62.470		62.072	62.737	62.444	
2976	62.470				63.2391	62.7954	62.102		62.451	63.514
4152					63.298					63.3929
3096	62.996	63.045	62.944	63.139		63.044	62.645	63.21	63.094	
4008	63.062	62.914	63.173	63.182		63.104	62.493	63.385	63.115	
5016	63.290	63.078	63.249	63.176		63.327	62.65	63.465	63.107	

time	Mass gain	Mass gain	Mass gain	Mass gain	Mass gain
(h)	(%mass)	(%mass)	(%mass)	(%mass)	(%mass)
	23°C/air	80°C/air	80C/RME15%	80C/100RME	23C/100RME
0	0.000	0.000	0.000	0.000	0.000
192	0.362				
480	0.333		0.061	-0.129	
864	0.463	-0.611	0.050	-0.213	
2856		-0.637	0.427	-0.042	
2976					0.435
4152					0.150
3096	0.576	-0.634	0.423	-0.071	
4008	0.567	-0.669	0.336	-0.106	
5016	0.558	-0.679	0.342	-0.109	

time	Width	Width	Width	Width	Width	Width Gain	Width Gain	Width Gain	Width Gain	Width Gain
(h)	(mm)	(mm)	(mm)	(mm)	(mm)	(%)	(%)	(%)	(%)	(%)
	23°C/air	80°C/air	80/15RME	80/100RME	23/100RME	23°C/air	80°C/air	80/15RME	80/100RME	23/100RME
0	9.891	9.891	9.891	9.891	9.891	0.00	0.00	0.00	0.00	0.00
192	9.911									
480	9.908	9.901	9.91	9.893		0.17	0.10	0.19	0.02	
864	9.912	9.887	9.912	9.892		0.21	-0.04	0.21	0.01	
2856	9.913	9.881	9.914	9.915		0.22	-0.10	0.23		
2976	9.915	9.876		9.896	9.9298	0.24	-0.15		0.05	0.39
3096	9.89	9.857	9.931	9.887		-0.01	-0.34	0.40	-0.04	
4008	9.91	9.895	9.913	9.901		0.19	0.04	0.22	0.10	
5016	9.902	9.884	9.912	9.896		0.11	-0.07	0.21	0.05	

time	Thickness	Thickness	Thickness	Thickness	Thickness	Thickness Gain	Thickness Gain	Thickness Gain	Thickness Gain	Thickness Gain
(h)	(mm)	(mm)	(mm)	(mm)	(mm)	(%)	(%)	(%)	(%)	(%)
	23°C/air	80°C/air	80/15RME	80/100RME	23/100RME	23°C/air	80°C/air	80/15RME	80/100RME	23/100RME
0	4.112	4.112	4.112	4.112	4.112	0.00	0.00	0.00	0.00	0.00
192	4.119					0.17				
480	4.117	4.076	4.101	4.099		0.12	-0.88	-0.27	-0.32	
864	4.115	4.096	4.104	4.099		0.07	-0.39	-0.19	-0.32	
2856	4.172	4.094	4.117	4.098		1.46	-0.44	0.12	-0.34	
2976	4.118	4.094		4.071	4.1737	0.15	-0.44		-1.00	1.50
3096	4.108	4.122	4.137	4.139		-0.10	0.24	0.61	0.66	
4008	4.138	4.175	4.139	4.152		0.63	1.53	0.66	0.97	
5016	4.149	4.118	4.149	4.136		0.90	0.15	0.90	0.58	

Table 6. Influence of diesel/RME and RME on PA12

time	Modulus	Modulus	Modulus	Modulus	Modulus	Modulus	Modulus	Modulus
(h)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	80°C/air	80°C/air	80°C/Diesel-RME15%	80°C/Diesel-RME15%	80°C/RME100%	80°C/RME100%
0	1234	45	1234	45	1234	45	1234	45
120	1234	45	1255	67	1222	45		
360	1172	45	1365	20	1142	48	1085	26
1008	1110	85	1353	90			1013	66
2016					911	61	962.6	45
3024	1093	18	1413	119	943	42	938.8	32.5

time	yield stress	yield stress	yield stress	yield stress	yield stress	yield stress	yield stress	yield stress
(h)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	80°C/air	80°C/air	80°C/Diesel-RME15%	80°C/Diesel-RME15%	80°C/RME100%	80°C/RME100%
0	40.02	0.45	40.02	0.45	40.02	0.45	40.02	0.45
120	40.02	0.45	43.54	0.85	42.05	0.26	40.99	0.27
350	38.94	0.21	44.73	0.11	41.1	0.38	40.36	0.48
1008	38.06	0.37	44.81	0.51	40.21	0.27	40.05	0.33
2016					39.65	0.32	39.73	0.27
3024	37.54	0.22	46.15	0.17	39.98	0.12	39.73	0.25

time	yield strain	yield strain	yield strain	yield strain	yield strain	yield strain	yield strain	yield strain
(h)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)
	23°C/air	23°C/air	80°C/air	80°C/air	80°C/Diesel-RME15%	80°C/Diesel-RME15%	80°C/RME100%	80°C/RME100%
0	10.65	0.52	10.65	0.52	10.65	0.52	10.65	0.52
120	10.65	0.52	11.82	0.95	12.72	0.78	14.13	0.71
350	11.73	0.47	11.29	0.22	13.5	0.68	14.59	0.4
1008	11.63	0.5	11.61	0.92	14.26	0.84	15	0.47
2016					16.52	0.89	16.36	0.69
3024	12.43	0.6	11.2	0.55	16.44	0.48	16.17	0.56

time	stress break	stress break	stress break	stress break	stress break	stress break	stress break	stress break
(h)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	80°C/air	80°C/air	80°C/Diesel-RME15%	80°C/Diesel-RME15%	80°C/RME100%	80°C/RME100%
0	54.29	2.46	54.29	2.46	54.29	2.46	54.29	2.46
120	54.2	2.46	50.49	6.09	54.49	2.67	51.26	6.87
360	51.27	0.4	47.48	8.53	51.95	0.78	52.95	2.05
1008	55.19	2.7	42.96	6.06	53.91	1.54	52.94	2.4
2016					51.61	1.41	51.28	1.08
3024	53.34	3.39	38.94	4.72	51.71	1.47	51.4	1.66

time	strain break	strain break	strain break	strain break	strain break	strain break	strain break	strain break
(h)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)
	23°C/air	23°C/air	80°C/air	80°C/air	80°C/Diesel-RME15%	80°C/Diesel-RME15%	80°C/RME100%	80°C/RME100%
0	384.7	52	384.7	52	384.7	52	384.7	52
120	384.3	51.8	336.9	38	394.6	63.8	349.2	45
360	341.4	31.8	320.1	8.2	333.5	7	365.4	40
1008	414.3	13.1	322.1	8.5	350.4	20.9	379.1	42
2016					383.5	63.7	367.1	36
3024	397.95	17.61	324.13	5.52	342.2	30.23	364.6	32

Table 7. Influence of diesel/RME and RME on PA12

time	Y.I.	Y.I.	Y.I.	Y.I.
(h)				
	23°C/air	80°C/air	°C/Diesel-RME1	80°C/RME100%
0	-32.251	-32.251	-32.251	-32.251
130	-31.21	-9.23	25.59	34.55
360	-31.02	4.83	49.38	58.56
1008	-32.251	21.13	59.407	68.476
2016			74.767	85.856
3024	-30.302	35.091	69.86	88.553

time	Mass gain	Mass gain	Mass gain	Mass gain
(h)	(%mass)	(%mass)	(%mass)	(%mass)
	23°C/air	80°C/air	°C/Diesel-RME1	80°C/RME100%
0	0.000	0.000	0.000	0.000
120	0.247	-0.171	0.534	0.434
360	0.356	-0.198	0.894	0.673
1008	0.527	-0.215	1.333	0.967
2015			1.671	1.207
3024	0.713	-0.310	1.806	1.309

time	Width	Width	Width	Width	Width Gain	Width Gain	Width Gain	Width Gain
(h)	(mm)	(mm)	(mm)	(mm)	(%)	(%)	(%)	(%)
	23°C/air	80°C/air	°C/Diesel-RME1	80°C/RME100%	23°C/air	80°C/air	0°C/Diesel-RME15%	80°C/RME100%
0	9.919	9.919	9.919	9.919	0.00	0.00	0.00	0.00
120	9.919	9.917	9.941	9.927	0.00	-0.02	0.22	0.08
360	9.921	9.914	9.934	9.932	0.02	-0.05	0.15	0.13
1008	9.941	9.917	9.961	9.956	0.22	-0.02	0.42	0.37
2016			9.992	9.979			0.74	0.60
3024	9.951	9.913	9.986	9.956	0.32	-0.06	0.68	0.37

time	Thickness	Thickness	Thickness	Thickness	Thickness Gain	Thickness Gain	Thickness Gain	Thickness Gain
(h)	(mm)	(mm)	(mm)	(mm)	(%)	(%)	(%)	(%)
	23°C/air	80°C/air	°C/Diesel-RME1	80°C/RME100%	23°C/air	80°C/air	0°C/Diesel-RME15%	80°C/RME100%
0	4.049	4.049	4.049	4.049	0.00	0.00	0.00	0.00
120	4.049	4.048	4.061	4.057	0.00	-0.02	0.30	0.20
360	4.046	4.041	4.062	4.057	-0.07	-0.20	0.32	0.20
1008	4.064	4.05	4.077	4.069	0.37	0.02	0.69	0.49
2016			4.089	4.071			0.99	0.54
3024	4.063	4.056	4.078	4.073	0.35	0.17	0.72	0.59

Table 8. Influence of diesel/RME and RME on HDPE

time	Modulus	Modulus	Modulus	Modulus	Modulus	Modulus
(h)	(MPa)	SD (MPa)	(MPa)	SD(MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	60°C/air	60°C/air	60°C/RME100%	60°C/RME100%
0	777.8	19.6	777.8	19.6	777.8	
72			769.4	66	491.1	27.2
480	818.4	28.9	775.2	18.63		
984			823.6	26.3	524.3	22.5
1000	884.6	22.4	878.4	22.1	524.1	10
1992	820.4	18.7	799.8	17.4	528.4	17.8
3000	867.2	18.8	888.1	19.6	545.1	22.1
4008	844.1	7.8	897.2	47.3	513.1	18.7
5016	885.2	23.8	844.2	48.3	554.6	12.6

time	yield stress	yield stress	yield stress	yield stress	yield stress	yield stress
(h)	(MPa)	SD (MPa)	(MPa)	SD(MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	60°C/air	60°C/air	60°C/RME100%	60°C/RME100%
0	24.56	0.17	24.56	0.17	24.56	0.17
72			24.63	0.13	23.48	0.69
480	24.44	0.16	24.86	0.12		
984			25.13	0.61	23.7	0.15
1000	25.27	0.08	25.97	0.1	22.83	0.16
1992	24.71	0.15	25.43	0.1	23.72	0.62
3000	25.22	0.09	25.94	0.07	24.18	0.17
4008	24.84	0.23	25.93	0.09	23.36	0.9
5016	24.86	0.22	25.69	0.08	24.29	0.09

time	yield strain	yield strain	yield strain	yield strain	yield strain	yield strain
(h)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)
	23°C/air	23°C/air	60°C/air	60°C/air	60°C/RME100%	60°C/RME100%
0	11.8	0.85	11.8	0.85	11.8	0.85
72			11.53	0.66	25.24	5.58
480	11.29	0.79	13.02	0.83		
984			12.43	0.57	22.62	1.9
1000	10.46	0.65	11.28	0.42	22.2	2.1
1992	10.27	0.11	12.05	0.71	24.07	1.24
3000	11.16	0.37	11.46	0.84	22.13	1.1
4008	10.8	0.41	11.57	0.62	24.83	2.15
5016	11.45	0.93	12.29	0.82	24.41	1.84

time	stress break	stress break	stress break	stress break	stress break	stress break
(h)	(MPa)	SD (MPa)	(MPa)	SD(MPa)	(MPa)	SD (MPa)
	23°C/air	23°C/air	60°C/air	60°C/air	60°C/RME100%	60°C/RME100%
0	18.5	3.13	18.5	3.13	18.5	3.13
72			17.69	2.61	20.44	1.78
480	17.57	2.34	16.41	1.7		
984			17.57	2	20.37	1.57
1000	19.55	2.13	18.49	1.83	19.89	0.92
1992	20.9	0.88	19.98	2.26	20.24	0.89
3000	17.85	2.66	19.23	3.06	19.4	1.86
4008	16.52	1.53	16.74	3.45	18.81	2.72
5016	16.87	3.32	17.1	2.25	18.67	1.66

time	strain break	strain break	strain break	strain break	strain break	strain break
(h)	(%)	SD (%)	(%)	SD (%)	(%)	SD (%)
	23°C/air	23°C/air	60°C/air	60°C/air	60°C/RME100%	60°C/RME100%
0	67.56	12.17	67.56	12.17	67.56	12.17
72			68.97	12.47	78.92	23.43
480	67.09	18.67	83.66	19.44		
984			73.79	12.56	78.45	12.46
1000	55.34	14.36	66.76	7.15	62.95	8.73
1992	48.23	9.87	64.06	19.15	77.91	10.66
3000	68.97	10.35	65.97	19	79.17	8.42
4008	88.07	37.14	75.08	12.93	82.07	18.19
5016	85.5	15.56	91.81	12.94	97.44	22.46

Table 9. Influence of diesel/RME and RME on HDPE

time	Y.I.	Y.I.	Y.I.
(h)			
	23°C/air	60°C/air	60°C/RME100%
0	-16.554	-16.554	-16.554
72		-15.665	-16.71
480	-15.959	-15.639	
984		-15.108	-16.861
1000	-16.095	-15.4	-16.412
1992	-16.99	-15.36	-14.015
3000	-15.601	-13.27	-16.659
4008	-16.034	-12.967	-16.199
5016	-15.576	-11.892	-16.143

time	Weight 6 bars	Weight 6 bars	Weight 6 bars	Mass gain	Mass gain	Mass gain
(h)	(g)	(g)	(g)	(%mass)	(%mass)	(%mass)
	23°C/air	60°C/air	60°C/RME100%	23°C/air	60°C/air	60°C/RME100%
0	43.631	43.631	43.631	0.00	0.00	0.00
72		43.649	45.843		0.04	5.07
480	43.674	43.644		0.10	0.03	
984		43.661	46.044		0.07	5.53
1000	43.624	43.665	45.871	-0.02	0.08	5.13
1992	43.657	43.641	45.868	0.06	0.02	5.13
3000	43.665	43.701	45.852	0.08	0.16	5.09
4008	43.649	43.756	45.847	0.04	0.29	5.08
5016	43.662	43.704	45.927	0.07	0.17	5.26

time	Width	Width	Width	Width Gain	Width Gain	Width Gain
(h)	(mm)	(mm)	(mm)	(%)	(%)	(%)
	23°C/air	60°C/air	60°C/RME100%	23°C/air	60°C/air	60°C/RME100%
0	9.9	9.9	9.9	0.00	0.00	0.00
72		9.93	10.011		0.30	1.12
480	9.911	9.903		0.11	0.03	
984		9.945	9.987		0.45	0.88
1000	9.88	9.88	10.009	-0.20	-0.20	1.10
1992	9.916	9.915	10.002	0.16	0.15	1.03
3000	9.88	9.908	9.988	-0.20	0.08	0.89
4008	9.867	9.883	9.976	-0.33	-0.17	0.77
5016	9.884	9.886	9.988	-0.16	-0.14	0.89

time	Thickness	Thickness	Thickness	Thickness Gain	Thickness Gain	Thickness Gain
(h)	(mm)	(mm)	(mm)	(%)	(%)	(%)
	23°C/air	60°C/air	60°C/RME100%	23°C/air	60°C/air	60°C/RME100%
0	3.851	3.851	3.851	0.00	0.00	0.00
72		3.849	3.914		-0.05	1.64
480	3.867	3.847		0.42	-0.10	
984		3.816	3.916		-0.91	1.69
1000	3.843	3.852	3.911	-0.21	0.03	1.56
1992	3.844	3.845	3.912	-0.18	-0.16	1.58
3000	3.845	3.855	3.92	-0.16	0.10	1.79
4008	3.851	3.852	3.907	0.00	0.03	1.45
5016	3.844	3.857	3.928	-0.18	0.16	2.00

Appendix 1

Moulding details

Polymer	**Unit**	**CARILON RDP 205**	**PA12**	**HDPE Lupolen 5021D Q425**
Date		05-07-96	08-02-96	05-07-96
Barrel Temperatures				
Nose	°C	260	265	255
Zone1	°C	260	270	255
zone2	°C	250	250	245
zone3	°C	220	235	235
Mould temperatures				
Mould 1	°C	70	60	40
Mould 2	°C	70	60	40
Injection speed	%	50	50	70
Holding Pressure		45	38	60
Holding time	s	14	18	35
Cool time	s	5	8	5
Cycle time	s	25	32	47

Appendix 2A

Set up of the ageing programme for RDP 205

AGEING PROGRAMMES FOR POLYMERS

Project name of identification	**Effect of RME 100% and diesel/RME 85/15 % vol/vol on the performance of CARILON Polymer**

MATERIALS

Code	Identification	Batch	Date inj. moulding
P1	RDP205	MDU96/008	22-04-96

MEDIA AND TEMPERATURES

Code	Medium	Temp.	Comments/composition
M1	RME 100%	80	See LJ CAR2/118/96 addendum no 2
M2	RME 15% in diesel	80	See LJ CAR2/118/96 addendum no 2
M3	AIR	80	Effect of temperature on the behaviour of polymer
M4	AIR	23	Reference condition, RH 50%.

AGING TIMES AND DATES OF TESTING

Time Code	Time hours	Days	Hours	Date of testing	Time Code	Time hours	Days	Hours	Date of testing
A0	0	0	0	07-03-96	A0	0	0	0	14-06-96
A1	192	8	0	15-03-96					
A2	480	20	0	27-03-96					
A3	864	36	0	12-04-96					
A4	2856	119	0	04-07-96					
A5	2976	124	0	09-07-96					
A6	4152	173	0	27-08-96					
A7	3000				A7	3096	129	0	21-10-96
A8	3984				A8	4008	167	0	28-11-96
A9	4992				A9	5016	209	0	09-01-97

MONITORING TESTS

Test code	Description	Standard method	Specimen Type	Temp. of test °C	Monitoring properties
T1	Tensile properties	ISO527	Dumbbell	23	Mod, Yld, Brk, (stress/strain)
T2	Dimensional change	LJ11896	Dumbbell	23	Thickness and width change
T3	Mass change	LJ11896	Dumbbell	23	Mass change
T4	Yellowness	DIN6167	Dumbbell	23	Yellowness change
T5	Volatiles		Dumbbell	23	Volatiles change

Appendix 2B

Set up of the ageing programme for RDP 205

Details of the starting of the ageing test on 140696

Details of RME used for testing: Supplier: NOVAOL
Drum, 5L contents according to the label.
Drum code: Ref 5248 datum: 15-04-97

Filling of cylinders with RME

Cylinder code:	Volume	Material	Level, %
113	380	RME	100
114	380	RME/Diesel	15

Mixing of eolys and diesel

	ml	g
RME	75	72.1
Diesel	425	393.9
Ratio, %	15	15.47

Remarks: The liquids are shaked well in an erlenmeier for about 3 minutes at RT

Date: 06-14-96
Time of placement in ovens at 60 °C: 10:30

Oven codes:

material	Oven
MDU96/008	6

Procedure for the removal of specimens from the cylinders

Cylinders were withdrawn from the oven and allowed to cool for about 30 min. before opening.
It was observed that by only wiping of the tensile bars from the RME in the cylinders
the surface was fatty and seemed to have a glossy appearance.
Therefore it was tested whether the specimens could be cleaned better by rinsing with a solvent.
From earlier experiments it was found that toluene is a good solvent for RME.
Therefore all specimens tested were dipped in toluene and stirred
for about 5 seconds in a cylinder with about 300 ml toluene .
Immediately thereafter the specimens were dried with a clean cloth.
Immediately after drying the mass of each set of 6 specimens was determined.
The specimens were tested for the mechanical properties and the yellowness within 3 hours
after removal from the cylinders.

Appendix 2C

Ageing programme for RDP 205, specimens arrangement

AGEING PROGRAMMES FOR POLYMERS

Project: Effect of RME on the performance of CARILON Polymer

Summary of specimen codes

MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	CYL/TRAY	Specimen nrs					
Code	Type	hours			°C	%	Nr	C/T						
P1	P1000	3000	14-06-96	air	23	50			1	2	3	4	5	6
P1	P1000	3000	14-06-96	air	80	--	6	T	7	8	9	10	11	12
P1	P1000	3000	14-06-96	RME 100%	80	--	6	C113	13	14	15	16	17	18
P1	P1000	3000	14-06-96	RME 15%	80	--	6	C114	19	20	21	22	23	24
MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P1	P1000	4000		air	23	50			25	26	27	28	29	30
P1	P1000	4000		air	80	--	6	T	31	32	33	34	35	36
P1	P1000	4000		RME 100%	80	--	6	C113	37	38	39	40	41	42
P1	P1000	4000		RME 15%	80	--	6	C114	43	44	45	46	47	48
MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P1	P1000	5000		air	23	50			49	50	51	52	53	54
P1	P1000	5000		air	80	--	6		55	56	57	58	59	60
P1	P1000	5000		RME 100%	80	--	6	C113	61	62	63	64	65	66
P1	P1000	5000		RME 15%	80	--	6	C114	67	68	69	70	71	72

Appendix 3A

Set up of the ageing programme for PA12

File code **PA12RME2.xls**

MATERIALS

Code	Identification		Batch		Date inj. moulding	
	PA12		Rilsan A AMNOTLD		08-02-96	

MEDIA AND

Code	Medium	Temp. C	Comments/composition
M1	RME 100%	80	See LJ CTCAR2/832/95 Arie Kramer, Appendix 50
M2	RME 15%	80	RME/Diesel 15% mass, See.
			LJ CTCAR2/832/95 Arie Kramer Appendix 50
M3	AIR	80	Effect of temperature on the behaviour of polymer
M4	AIR	23	Reference condition, RH 50%.

AGING TIMES AND DATES OF

Time Code	Time hours	Days	Hours	Date of testing
A0	0	0	0	15-02-96
A1	120	5	0	20-02-96
A2	360	15	0	01-03-96
A3	1008	42	0	28-03-96
A4	2016	84	0	09-05-96
A5	3024	126	0	20-06-96

MONITORING TESTS

Test code	Description	Standard method	Specimen Type	Temp. of test °C	Monitoring properties
T1	Tensile properties	ISO527	Dumbbell	23	Mod, Yld, Brk, (stress/strain)
T2	Dimensional change	LJ11896	Dumbbell	23	Thickness and width change
T3	Mass change	LJ11896	Dumbbell	23	Mass change
T4	Yellowness	DIN6167	Dumbbell	23	Yellowness change
T5	Volatiles		Dumbbell	23	Volatiles change

Appendix 3B

Set up of the ageing programme for PA12

Details of the starting of the ageing test on

Details of RME used for testing: Supplier: NOVAOL
Drum, 5L contents according to the label.
Drum code Ref 5248 datum: 15-04-97

Filling of cylinders with RME

Cylinder code:	Volume,ml	Material	Level, %
A	380	RME	100
B	380	RME/Diesel	15

Mixing of RME and diesel

	ml	g
RME	75	72.1
Diesel	425	393.9
Ratio, %	15	15.4721

Remarks: The liquids are shaked well in an erlenmeier for about 3 minutes at RT

Filling of cylinders with RME
Date: 15-02-96, Arie Kramer and Wilma v. Straaten
Time of placement in ovens at °C: 11:10

Procedure for the removal of specimens from the cylinders

Cylinders were withdrawn from the oven and allowed to cool for about 30 min. before opening. It was observed that by only wiping of the tensile bars from the RME in the cylinders the surface was fatty and seemed to have a glossy appearance. Therefore it was tested whether the specimens could be cleaned better by rinsing with a solvent. From earlier experiments it was found that toluene is a good solvent for RME. Therefore all specimens tested were dipped in toluene and stirred for about 5 seconds in a cylinder with about 300 ml toluene . Immediately thereafter the specimens were dried with a clean cloth. Immediately after drying the mass of each set of 6 specimens was determined. The specimens were tested for the mechanical properties and the yellowness within 3 hours after removal from the cylinders.

Appendix 4A

Set up of the ageing programme for HDPE

MATERIALS

Code	Identification	Batch	Date inj. moulding
P2	HDPE	Lupolen 5021D	25-04-96

MEDIA AND TEMPERATURES

Code	Medium	Temp. C	Comments/composition
M1	RME 100%	60	See LJ CAR2/118/96 addendum no 2
M2	AIR	60	Effect of temperature on the behaviour of polymer
M3	AIR	23	Reference condition, RH 50%.

AGING TIMES AND DATES OF TESTING

Time Code	Time hours	Days	Hours	Date of testing
A0	0	0	0	14-06-96
A1	72	3	0	17-06-96
A2	480	20	0	04-07-96
A3	984	41	0	25-07-96
A4	1000	41	16	25-07-96
A5	1992	83	0	05-09-96
A6	3000	125	0	17-10-96
A7	4008	167	0	28-11-96
A8	5016	209	0	09-01-97

MONITORING TESTS

Test code	Description	Standard method	Specimen Type	Temp. of test °C	Monitoring properties
T1	Tensile properties	ISO527	Dumbbell	23	Mod, Yld, Brk, (stress/strain)
T2	Dimensional change	LJ11896	Dumbbell	23	Thickness and width change
T3	Mass change	LJ11896	Dumbbell	23	Mass change
T4	Yellowness	DIN6167	Dumbbell	23	Yellowness change
T5	Volatiles		Dumbbell	23	Volatiles change

Appendix 4B

Set up of the ageing programme for HDPE

Details of the starting of the ageing test on 140696

Details of RME used for testing: Supplier: NOVAOL
Drum, 5L contents according to the label.
Drum code Ref 5248 datum: 15-04-97

Filling of cylinders with RME

Cylinder code:	Volume,ml	Material	Level, %
110	375	RME	100
111	370	RME	100
112	415	RME	100

Date: 14-06-96
Time of placement in ovens at 60 °C: 10:00

Oven codes:

material	Oven
HDPE	15

Procedure for the removal of specimens from the cylinders

Cylinders were withdrawn from the oven and allowed to cool for about 30 min. before opening.
It was observed that by only wiping of the tensile bars from the RME in the cylinders
the surface was fatty and seemed to have a glossy appearance.
Therefore it was tested whether the specimens could be cleaned better by rinsing with a solvent.
From earlier experiments it was found that toluene is a good solvent for RME.
Therefore all specimens tested were dipped in toluene and stirred
for about 5 seconds in a cylinder with about 300 ml toluene .
Immediately thereafter the specimens were dried with a clean cloth.
Immediately after drying the mass of each set of 6 specimens was determined.
The specimens were tested for the mechanical properties and the yellowness within 3 hours
after removal from the cylinders.

Appendix 4C

Ageing programme for HDPE, specimens arrangement

AGEING PROGRAMMES FOR POLYMERS

Project: **Effect of RME on the performance of HDPE**
Extension of the running RME program

Summary of specimen codes

MATERIALS		**Time**	**Date**	**MEDIA**	**TEMP**	**RH**	**Oven nr**	**Cylinder**	**Specimen nrs**					
Code	**Type**	**hours**			**°C**	**%**	**Nr**	**Nr**						
P2	HDPE	0	Ref 3-6-9	air	23	50								
P2	HDPE	0												
P2	HDPE	0												
MATERIALS		**Time**	**Date**	**MEDIA**	**TEMP**	**RH**	**Oven nr**	**Cylinder**	**Specimen nrs**					
Code	**Type**	**hours**			**°C**	**%**	**Nr**	**Nr**						
P2	HDPE	75		air	23	50			7	8	9	10	11	12
P2	HDPE	75		air	60	--			25	26	27	28	29	30
P2	HDPE	75	17-06-96	100% RM	60	--	15	110	501	502	503	504	505	506
MATERIALS		**Time**	Date	**MEDIA**	**TEMP**	**RH**	**Oven nr**	**Cylinder**	**Specimen nrs**					
Code	**Type**	**hours**			**°C**	**%**	**Nr**	**Nr**						
P2	HDPE	150		air	23	50			61	62	63	64	65	66
P2	HDPE	150		air	60	--			79	80	81	82	83	84
P2	HDPE	150		100% RM	60	--	15	110	507	508	509	510	511	512
MATERIALS		**Time**	**Date**	**MEDIA**	**TEMP**	**RH**	**Oven nr**	**Cylinder**	**Specimen nrs**					
Code	**Type**	**hours**			**°C**	**%**	**Nr**	**Nr**						
P2	HDPE	500		air	23	50			115	116	117	118	119	120
P2	HDPE	500		air	60	--			133	134	135	136	137	138
P2	HDPE	500	04-07-96	100% RM	60	--	15	110	513	514	515	516	517	518
MATERIALS		**Time**	**Date**	**MEDIA**	**TEMP**	**RH**	**Oven nr**	**Cylinder**	**Specimen nrs**					
Code	**Type**	**hours**			**°C**	**%**	**Nr**	**Nr**						
P2	HDPE	1000		air	23	50			169	170	171	172	173	174
P2	HDPE	1000		air	60	--			187	188	189	190	191	192
P2	HDPE	1000	25-07-96	100% RM	60	--	15	111	519	520	521	522	523	524

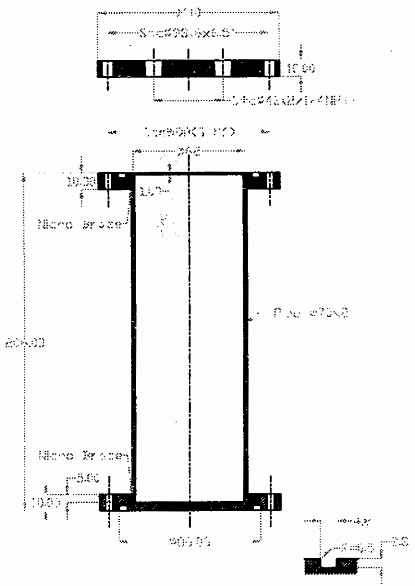
Appendix 4D

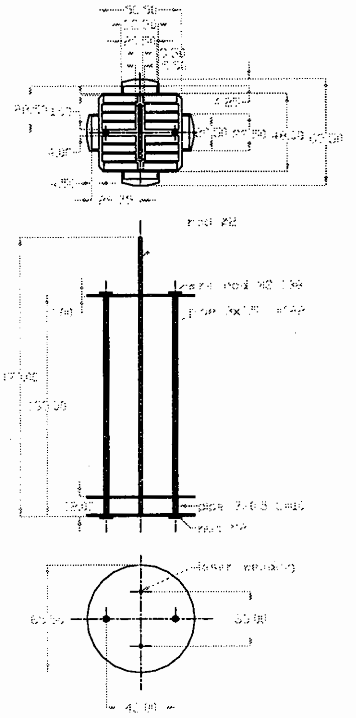
Ageing programme for HDPE, specimens arrangement

MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P2	HDPE	2000		air	23	50			223	224	225	226	227	228
P2	HDPE	2000		air	60	--			241	242	243	244	245	246
P2	HDPE	2000	05-09-96	100% RM	60	--	15	111	525	526	527	528	529	530
MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P2	HDPE	3000		air	23	50			277	278	279	280	281	282
P2	HDPE	3000		air	60	--			295	296	297	298	299	300
P2	HDPE	3000	17-10-96	100% RM	60	--	15	111	531	532	533	534	535	536
MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P2	HDPE	4000		air	23	50			331	332	333	334	335	336
P2	HDPE	4000		air	60	--			349	350	351	352	353	354
P2	HDPE	4000	27-11-96	100% RM	60	--	15	112	537	538	539	540	541	542
MATERIALS		Time	Date	MEDIA	TEMP	RH	Oven nr	Cylinder	Specimen nrs					
Code	Type	hours			°C	%	Nr	Nr						
P2	HDPE	5000		air	23	50			385	386	387	388	389	390
P2	HDPE	5000		air	60	--			403	404	405	406	407	408
P2	HDPE	5000	08-01-97	100% RM	60	--	15	112	543	544	545	546	547	548

Appendix 5:

Drawing of the test cylinders and racks used for ageing tests





List of Abbreviations

EC	**European Commission**
EP	**European Parliament**
OEM	**Original Equipment Manufacturer**
HDPE	**High Density PolyEthylene**
MTBE	**Methyl Tertiary Butyl Ether**
RME	**Rapeseed Methyl Ester**
ISO	**International Standards Organisation**
RT	**Room temperature**
RH	**Relative humidity**
DIN	**Deutsche Industrie Norm**
YI	**Yellowness Index**

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