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Assessment of Elastomeric Components of a Solar Thermal Collector

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Abstract

Elastomeric components play important functions in solar thermal collectors (STC's) as sealings, fittings or absorbers, being important that they maintain their properties along STC lifetime. In what ageing is concerned, current standards for assessment of elastomeric materials put emphasis on the effect of heating.

The results presented in this paper refer to the evaluation of the effects of exposure to heating as well as to solar radiation by means of accelerated tests on two ethylene propylene diene terpolymer (EPDM) components of a commercial STC both carbon black filled compounds. At the current stage of the study, results showed that the effect of solar radiation may be relevant for the assessment of the components and, for that reason, should be considered along with temperature.

Keywords: *solar thermal collectors, elastomeric components, ageing, accelerated tests*

1. Introduction

Environmental factors cause degradation of polymers (Massey, 2007), in particular UV radiation induce photo-oxidation reactions. Even polymers with low unsaturation as is the case of EPDM, which is extensively used in outdoor applications, are prone to such reactions (Maecker and Priddy, 1991). EPDM formulations for those applications are designed to minimise photodegradation. Nevertheless changes in crosslink density, hardness, tensile properties and appearance are referred (Aimura and Wada, 2006; Zhao et al., 2007).

The work whose results are presented in this paper is part of a study aiming at evaluating the suitability of the accelerated ageing tests specified in standards ISO 9808:1990 and ISO 9553:1997 for the assessment of elastomeric components of STC's operating in high UV radiation environments and contributing to design accelerated ageing tests more capable of reproducing those environments whenever the standards are not adequate. To that end different components of STC's are characterised after the heating test described in those standards and after laboratory exposure to radiation as well as after exposure in two outdoor exposure sites. Detailed characterization of these sites can be found in Diamantino et al. (2017). The results here reported refer to the evaluation of the effects of the laboratory tests – resistance to heating and exposure to xenon-arc and to UV radiation - on two components of a commercial STC, a fitting with the function of isolating inlet and outlet connections and the glazing sealant, both carbon black filled EPDM compounds.

2. Methods

For the tests fittings were used as such while the sealing was cut in segments with a length of approximately 200 mm.

2.1 Resistance to heating test

The test was carried out at 175 ºC for 14 days according to ISO 9808:1990 and ISO 9553:1997 based on the stagnation temperature of the collector (150ºC) in an oven with forced air circulation. Complementary tests were performed at 150 ºC and 125 ºC to investigate the thermal behaviour of the components also at lower

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temperatures.

To evaluate the effects of the heating on the components the relative variations of total mass and dimensions were calculated, thermogravimetric analyses and mechanical tests were done.

The internal diameter of the fittings was determined with a caliper rule, three measures being taken for each sample. The length of the fractions of the sealing was determined with a scale. Relative variation of mass and dimensions was calculated for a minimum of five samples according to equation 1:

$$\text{Relative variation (\%)} = \frac{\text{Final value} - \text{Initial value}}{\text{Initial value}} \times 100 \qquad \text{(eq. 1)}$$

Thermogravimetric analyses were done according to ASTM D6370-99 in a Setaram equipment, model Setsys, with around 10 mg of sample in a platinum crucible. The first heating from room temperature to 560 ºC was carried out in argon atmosphere and the second heating from 300 ºC to 800 ºC was carried out in air atmosphere.

Mechanical tests were done with a Testing Machine Instron 4467 at 100 mm/min for fittings and 500 mm/min for sealing according to ISO 37:2011. The shape of the samples made impossible determining the cross-sectional area, so the maximum force was determined, instead of tensile strength. Elongation at break was determined according to ISO 37:2011. The initial distance between grips in the test of sealing was 10 mm. Five specimens of each component were tested.

2.2 Exposure to radiation sources

Exposure to xenon-arc radiation was done according to ISO 4892-2:2013, cycle nº 4, in an Atlas Weatherometer Ci35A with daylight (borosilicate) filters. Each cycle is composed of a dry period of 1.7 h at a spectral irradiance level of 0.51 W m^{-2} nm^{-1} at 340 nm with black panel temperature of 63±3 ºC, chamber temperature of 38±3 ºC and a relative humidity of 50±10% followed by 0.3 h of water spray at the same spectral irradiance. Samples were collected after 72, 168, 336, 668, 1030, 1436, 1922, 2234 and 3240 h of exposure.

Exposure to UV fluorescent radiation was done according to ISO 4892-3:2013, cycle nº 2, in a chamber Q-Lab, QUV spray with a UVA-340 lamp type. Each cycle is composed of a dry period of 8 h at a spectral irradiance level of 0.76 W m^{-2} nm^{-1} at 340 nm with black panel temperature of 50±3 ºC, followed by 0.25 h water spray and 3.75 h condensation without radiation and black panel temperature of 50±3 ºC during the condensation period. Samples were collected after 84, 180, 348, 732, 1080, 1404, 1752, 2076, 3240 and 4320 h of exposure.

The effect of the radiation on the components was assessed by colour difference evaluation, mechanical tests and glass transition temperature determination.

Colour difference, ΔE^*_{ab}, was determined according to ISO 11664-4:2011 with a spectrocolorimeter X-Rite 948. A minimum of five specimens were used in each determination. With the objective of showing the contribution of the variation of each of the coordinates to ΔE^*_{ab}, average values of ΔL^*, Δa^* and Δb^* were calculated.

Mechanical properties were evaluated as referred above.

Glass transition temperature, T_g, was determined in a TA Instruments DSC Q2000 equipment with a refrigeration system TA Instruments Refrigerated Cooling System 90. A mass of 5 mg was used in each test that was put in an aluminium crucible. Two cycles were carried out between -90 ºC and 100 ºC at a heating rate of 20 ºC/min, the values of the second cycle being taken.

3. Results

3.1 Resistance to heating test

Relative variations of mass and dimensions of the fitting and of the sealing after the resistance to heating test at 125 ºC, 150 ºC and 175 ºC are shown in Figure 1.

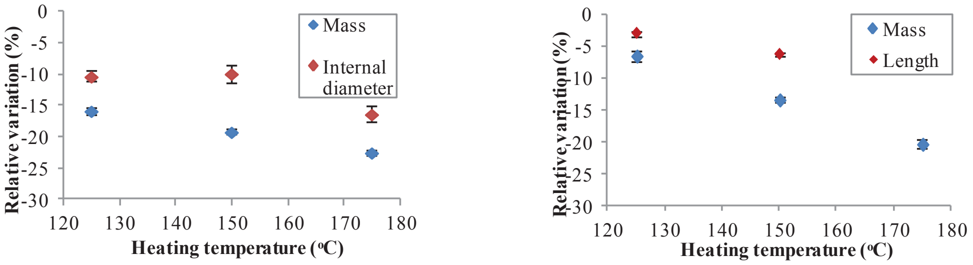

Fig. 1: Relative variations of mass and internal diameter of the fitting (left) and of mass and length of the sealing (right) after resistance to heating tests carried out at 125 ºC, 150 ºC and 175 ºC (bars representing standard deviation).

It was observed mass loss of the fitting in the three tests, variation increasing with the increase of the test temperature. The fitting lost 16% of the mass in the test at 125 ºC, 19 % of the mass at 150 ºC and 22% at 175 ºC. Internal diameter of the sealing decreased 10% after the tests at 125 ºC and 150 ºC and 16% after the test at 175 ºC. ISO 9808:1990 does not indicate an upper limit for volatiles loss.

The sealing lost 6% of the mass in the test at 125 ºC, 13% at 150 ºC and 20% at 175 ºC. The mass loss of the sealing was higher than the sum of maximum of volatiles lost (1%) and volatiles condensable (0.1%) for all the temperatures tested, which is the upper limit value admissible by ISO 9553:1997. The length of the sealing decreased 3% after the test at 125 ºC and 6% at 150 ºC. The length of this component was not determined after the test at 175 ºC as the samples were deformed and brittle.

The results of thermogravimetric analyses in the zone of heating in inert atmosphere of the fitting and of the sealing are shown in Table 1. The thermograms of the unheated components showed two zones of mass loss. The first zone begun in the region typically designated by “highly volatile matter”, referring to compounds of low boiling points (approximately 300 ºC or lower) such as oil, plasticizers, curatives, antioxidants and antiozonants, and ended already in the region designated “medium volatile matter”, referring to medium volatility material such as processing oil and processing aid (Sircar, 1992). The second zone was also included in the region of medium volatile matter where EPDM is found (Paroli et al., 1991; Sircar, 1992).

Tab. 1: Temperature range of mass loss and mass loss of the fitting and of the sealing unheated and after the heating tests at 150 ºC and 175 ºC determined in the zone of heating in argon atmosphere

Component	Heating test temperature (ºC)	Temperature range of mass loss (ºC)	Mass loss (%)
Fitting	Unheated	178 - 421	25
		421 - 500	29
	150	236 - 514	42
	175	243 - 519	36
Sealing	Unheated	171 - 418	23
		419 - 502	24
	150	276 - 501	34
	175	256 - 505	28

The thermograms of the fitting and of the sealing heated at 150 ºC and 175 ºC showed only one zone of mass loss during inert heating beginning in the region of “highly volatile matter” but at significantly higher temperatures than the observed in the first mass loss for the unheated sample indicating that compounds with lower boiling points were removed during the heating tests. In the case of the fitting while above mentioned additives and elastomer corresponded to 54 % of the mass of the unheated sample they corresponded to 42 % and 36 % of the mass of the samples heated at 150 ºC and 175 ºC, respectively. In the case of the sealing additives and elastomer corresponded to 47% of the mass of unheated sample, while they corresponded to 34% and 28% of the samples heated at 150 ºC and 175 ºC, respectively.

Figure 2 shows maximum force and elongation at break of the fitting and of the sealing unheated and after the resistance to heating test. No appreciable variation of maximum force was observed for the fitting after the heating test at 125 ºC, but a decrease of elongation at break occurred. Maximum force clearly decreased after the tests at 150 ºC and 175 ºC. Elongation at break of this component after the heating tests at these higher temperatures was not determined due to brittleness of the samples. Maximum force of the sealing did not also change appreciably after the heating test at 125 ºC but a decrease of elongation at break occurred. Brittleness of the samples after heating at higher temperatures made impossible carrying out mechanical tests.

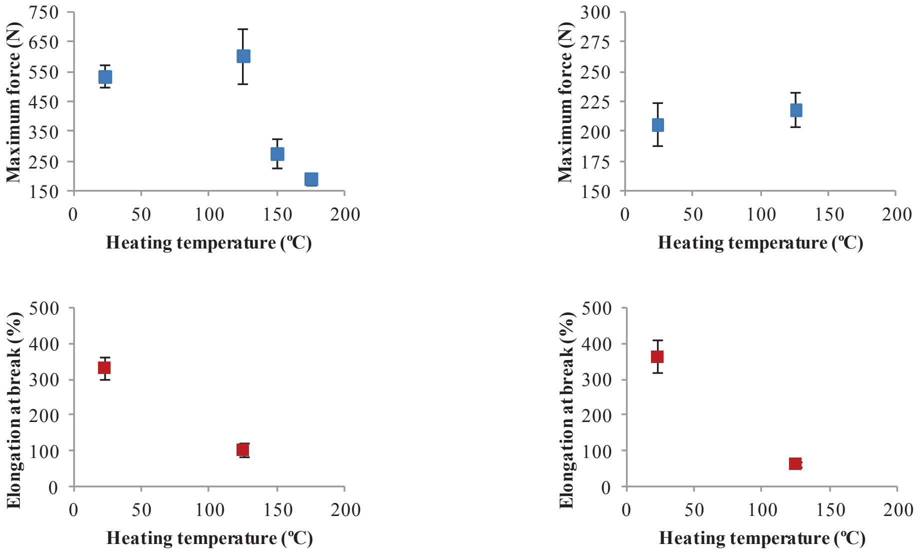

Fig. 2: Maximum force and elongation at break of the fitting (left) and of the sealing (right) unheated and after resistance to heating tests (bars representing standard deviation). Lack of data for 150 ºC and 175 ºC was due to brittleness of the samples.

3.2 Exposure to radiation sources

Colour difference, $\Delta E^*_{ab,}$ and average values of ΔL^*, Δa^* and Δb^* of the fitting and of the sealing after different exposure times to xenon-arc and to UV radiation are shown in Figures 3 and 4, respectively.

Exposure to xenon-arc radiation and water spray caused slight change of samples colour. Regarding the fitting, the final value was practically reached after 336 h of exposure. After this period, the difference in colour was mainly due to an increase of the coordinate L^* meaning the samples were lighter, with a smaller contribution of the b^* coordinate that varied towards the yellow. The colour difference of the sealing was more pronounced the most accentuated difference occurring in the first 72 h of test, after what the colour practically did not change. As observed with the fitting, the difference in colour was mainly due to the increase of coordinate L^*.

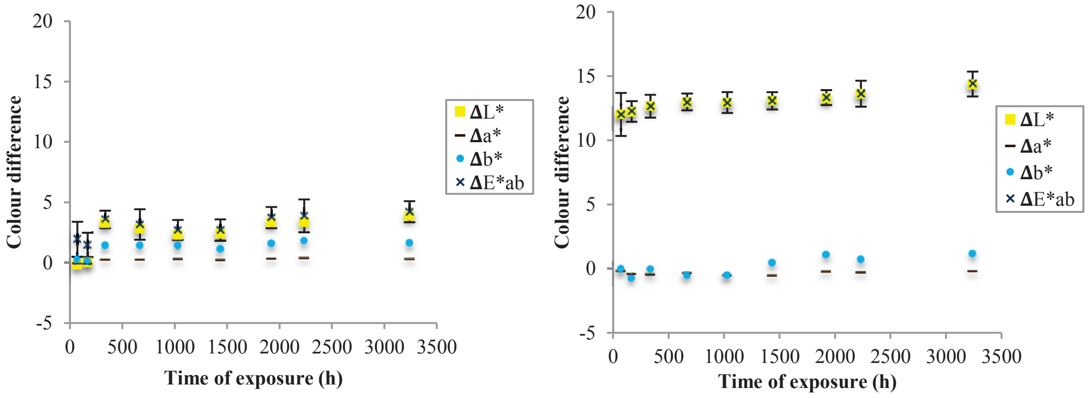

Fig. 3: Colour difference, $\Delta E^*_{ab,}$, and average values of ΔL^*, Δa^* and Δb^* of the fitting (left) and of the sealing (right) after exposure to xenon-arc radiation with water spray(bars representing standard deviation).

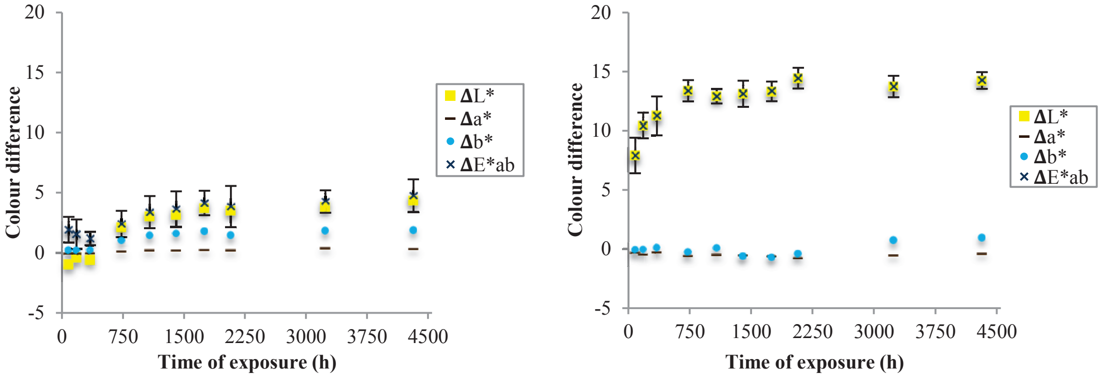

Fig. 4: Colour difference, ΔE^*_{ab}, and average values of ΔL^*, Δa^* and Δb^* of the fitting (left) and of the sealing (right) after exposure to fluorescent UV radiation with condensation and water spray (bars representing standard deviation).

Exposure to UV fluorescent radiation with condensation and water spray caused also slight change of samples colour, the variation in the final of the test being similar to the values determined at the end of the exposure to xenon-arc radiation. The main contribution to the difference of colour of the fitting was from ΔL^*, that until 348 h had negative values, meaning the samples were slightly darker, and after 732 h changed to positive values meaning the samples were lighter. There was also a small variation of the coordinate b^* after 732 h towards yellow. The most pronounced difference of colour of the sealing was observed in the first 72 h but difference of colour still increased, although less sharply until 732 h of test. The difference in colour was mainly due to the increase of L^*.

Maximum force and elongation at break of the fitting and of the sealing after exposure to xenon-arc radiation with water spray are shown in Figure 5.

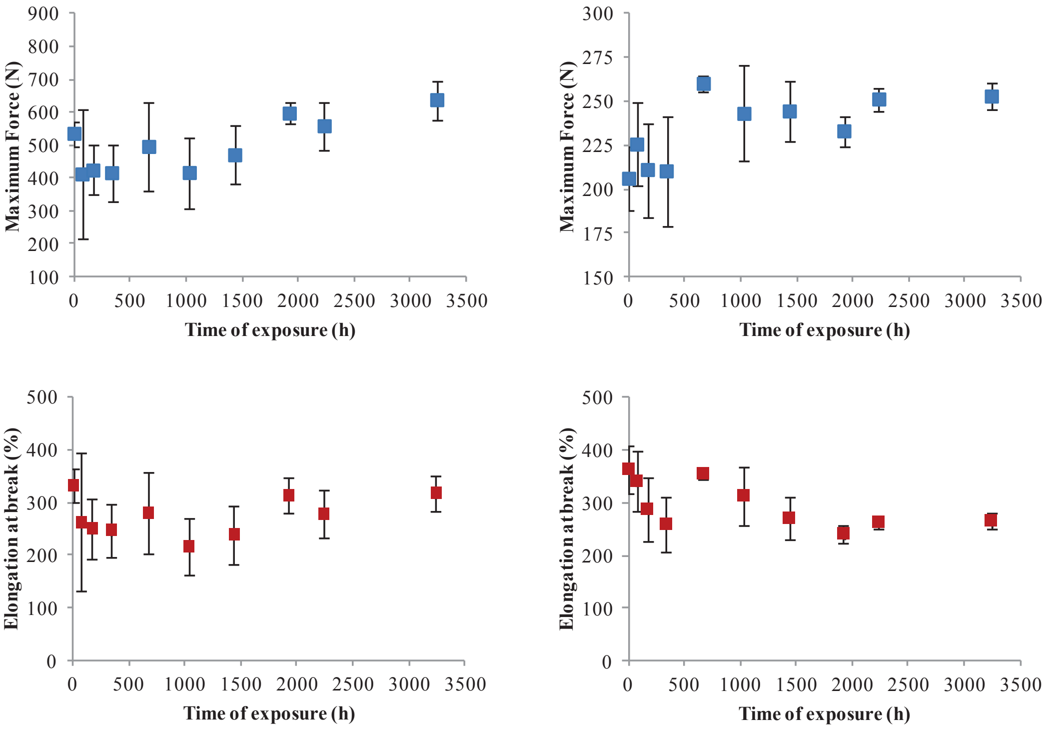

Fig. 5: Maximum force and elongation at break of the fitting (left) and of the sealing (right) after exposure to xenon-arc radiation and water spray (bars representing standard deviation).

Despite the relevant figures of standard deviation mainly due to specimen variability, changes of mechanical properties were noticed. The fitting showed a decrease of maximum force and elongation at break at early stage of exposure followed by an increasing. The maximum force of the sealing increased after an initial period of no defined tendency while the elongation at break decreased at early stage and after a reverse in this tendency decreased again. EPDM rubber undergoes both chain scission and crosslinking upon xenon-arc weathering (Maecker and Priddy, 1991). Although the changes observed may not be straightforwardly related to those polymer changes due to the chemical complexity of compounded EPDM rubber products, the increase of maximum force of the sealing and decrease of elongation at break for longer periods of exposure relatively to the initial values is compatible with formation of crosslinks.

The patterns of maximum force and elongation at break of the fitting after exposure to UV fluorescent radiation with water spray and condensation (Figure 6) resemble in general with those observed after exposure to xenon-arc test. However the more pronounced decrease of maximum force and elongation at break at early stages of exposure seems to indicate that chemical changes were intensified in the UV test. General patterns of maximum force and elongation at break of the sealing are also similar to those observed after exposure to xenon arc radiation. However, at the early stage of exposure a decrease of maximum force was observed.

Glass transition temperatures, *Tg*, of the two components determined along the exposure to xenon-arc radiation with water spray (Figure 7) and to UV fluorescent radiation with water spray and condensation (Figure 8) showed only slight variations. The trend that was observed in the fitting to increasing at longer periods of exposure to xenon-arc radiation means that the system became stiffer. Glass transition temperature of the sealing practically did not change along exposure indicating that this parameter was not sensible to the chemical changes that occurred in the system.

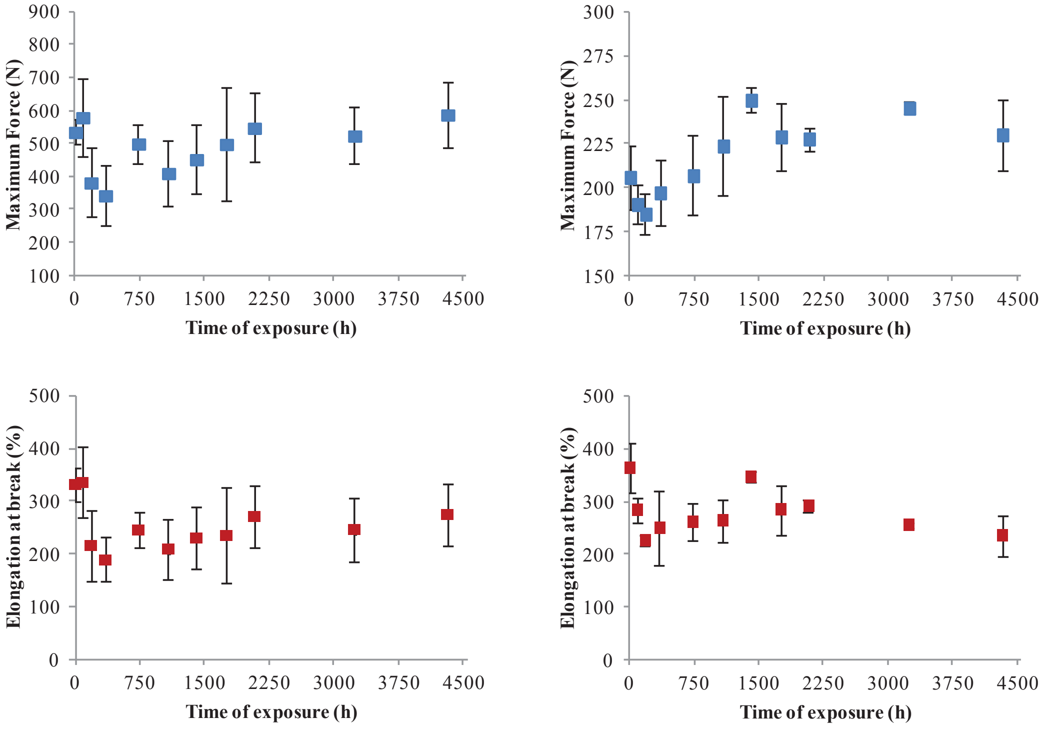

Fig. 6: Maximum force and elongation at break of the fitting (left) and of the sealing (right) after exposure to UV fluorescent radiation with water spray and condensation (bars representing standard deviation).

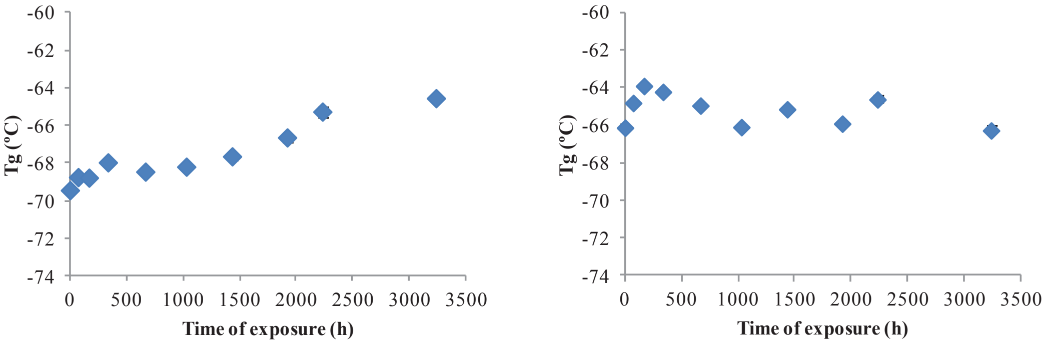

Fig. 7: Glass transition temperatures, T_g, of the fitting (left) and of the sealing (right) after exposure to xenon-arc radiation with water spray(bars representing standard deviation).

Glass transition temperatures of the fitting tended to increase at the beginning of the exposure to UV fluorescent radiation meaning that changes leading to stiffness of the system occurred. This trend was reversed after around 1050 h of test and then recovered at the end of the exposure time. Glass transition of the sealing practically did not change along exposure as observed after xenon-arc radiation.

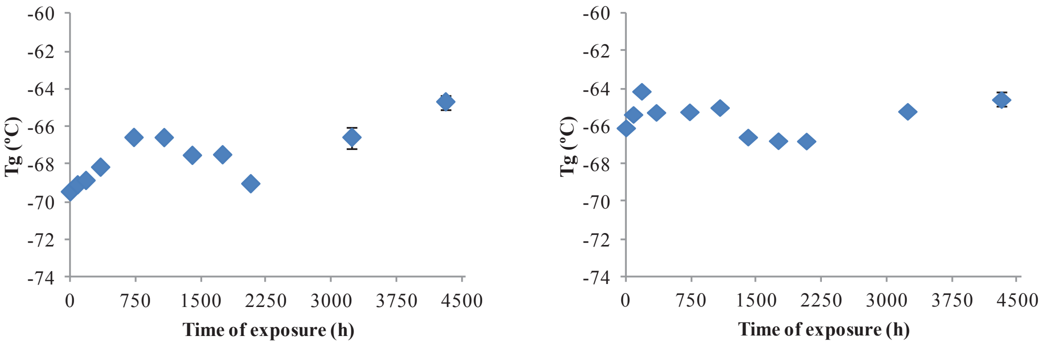

Fig. 8: Glass transition temperatures, T_g, of the fitting (left) and of the sealing (right) after exposure to UV fluorescent radiation with water spray and condensation (bars representing standard deviation).

4. Conclusions

The accelerated ageing tests - resistance to heating, exposure to xenon arc radiation and exposure to UV fluorescent radiation - performed with two elastomeric components of a solar thermal collector, a fitting and a sealing, caused chemical changes indicating that during the operation these components will be affected by high temperature and solar radiation. Resistance to heating tests performed at the temperature indicated by ISO 9808:1990 and ISO 9553:1997 and complementarily at lower temperatures led to significant mass loss and shrinkage of the fitting and the sealing, the variations being more pronounced with the increase of test temperature. Loss of mechanical performance was also clearly observed. Exposure to xenon-arc and to UV fluorescent radiation led to change of mechanical properties, the effects induced by UV fluorescent radiation being more pronounced. It is expected that those changes will be accelerated at the temperature of operation of the collector. The decrease in dimensions and mechanical performance of the fitting and of the sealing will result in poor isolation and higher permeability to atmospheric aggressions, changing the environment inside the collector. Results showed that the effect of solar radiation may be relevant for the assessment of the components and, for that reason, should be considered along with temperature.

5. Acknowledgements

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