Lifetime prediction of geosynthetics based on high-pressure autoclave tests

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ABSTRACT: The high-pressure autoclave test (HPAT) is a standardized oxidation test method in ISO 13438, method C. According to this method, the specimens are immersed in a defined aqueous liquid and they are exposed at elevated temperatures up to 80 °C combined with high oxygen pressure up to 50 bar. The experience shows that for the same lifetime criteria of 25 years, some polyolefin products which withstand the conventional oven test (100 °C, 28 days) don't withstand the autoclave test (80 °C, 14 days, 50 bar oxygen pressure). The HPAT sets the higher requirement concerning the oxidation resistance of geosynthetics. In a HPAT, it is possible to find the duration of immersion when the immersed material begins to oxidize and the mechanical strength begins to decrease as the oxygen pressure in the autoclave starts to decrease. The decrease of oxygen pressure in the high-pressure autoclave indicates that the stabilizer in the test material is consumed and the oxidation of polymer begins. Different types of reduction in mechanical strength values were obtained during the period of immersion in the high-pressure autoclave. The degradation of the materials after a certain immersion time is time dependent. To determine the oxidative degradation, different geosynthetics (woven geotextiles, non-woven geotextiles, geospacers, geogrids, geonets, polymeric geosynthetic barriers) have to be tested at different temperatures and at different oxygen pressures. The life time of geosynthetics must be extrapolated to atmospheric conditions by using a model with a modified Arrhenius equation.

1 INTRODUCTION

Many applications of polyolefin geosynthetics like e.g. landfills or tunneling require a long term oxidation resistance of more than 100 years (EAG-EDT 2005). With the conventional oven test at elevated temperature, the test would take unacceptable long durations to obtain reliable test results under acceptable temperatures. For long term tests on polyolefins, the maximum temperature should not exceed 80 °C because of a significant change in the mechanism of oxidation in this temperature range. Also parameters like morphological changes and transitions, oxygen stability and related diffusion limited oxidation (DLO) effects have to be considered for the choice of the highest temperature applied (Boehning et al. 2008). Therefore an oven test with e.g. 100 °C for PE or 110 °C for PP is too high for lifetime prediction. On the other hand, a test should not last longer than one year. The HPAT makes it possible to determine the long term oxidation resistance at relative short test durations even for temperatures of 60 °C to 80 °C (Schröder et al. 2008).

In ISO 13438 two different test methods are described to test the oxidative resistance: the conventional oven test (method B) and the HPAT (method C). These simple screening tests have been established for CE marking of products made of polyolefins (PP and PE). These screening tests give only a rough indication which is acceptable for 25 years lifetime (ISO/TS 13434).

It is not clear yet that the oven test method is suitable for all materials and product types (Schröder et al. 2001) to prove longer lifetime. As the activation energy for each single product is unknown, an extrapolation to longer lifetime is not justified.

The aim of the immersion in a high-pressure autoclave is the generation of an accelerated ageing process of test materials at elevated temperature and at elevated oxygen pressure. Subsequent the change of properties like e.g. mechanical strength will be tested. In contrast to oven tests, the surface-volume-ratio has no influence on the results from HPAT (Schröder et al. 2001). In this paper, results of tests on woven and non-woven geotextiles, polymeric geosynthetic barriers and geospacers will be presented.

2 EXPERIMENTAL

The HPAT facility and the scheme of the autoclave are shown in Figure 1 and Figure 2.

Figure 1. High-pressure autoclave facility in SKZ.

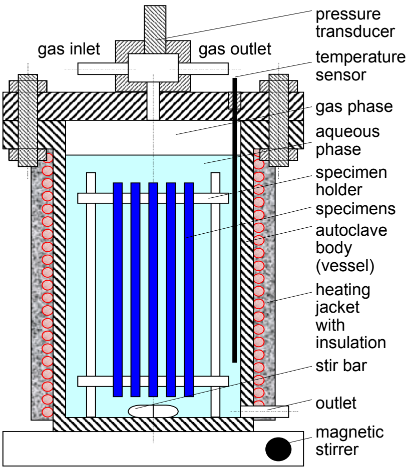

Figure 2. Scheme of the high-pressure autoclave.

The high-pressure autoclave consists a stainless steel body (vessel) with a total volume of 7 litres. The autoclave is filled with a well defined aqueous medium which is stirred by means of a magnetic stirrer. Above the liquid is space for gas phase (O_2) with at least 20 % of the total volume of the autoclave. The temperature of the liquid and the oxygen pressure in the autoclave are continuously measured and recorded. The autoclave is supplied with a heating jacket and with an insulation. The specimens are clamped in the specimen holder made of stainless steel.

According to ISO 13438, method C, the specimens are immersed in 0.01M $NaHCO_3$ aqueous solution at (80±1) °C and at (50±0.5) bar oxygen pressure. The pH value of the solution is adjusted to 10 with 1M NaOH at 20 °C. The purity of oxygen must be at least 99.999 vol%. The duration of immersion takes 14 days or 28 days (materials for reinforcement) for screening tests.

For estimation of lifetime high-pressure autoclave tests were carried out at three different oxygen pressures (10 bar, 20 bar and 50 bar) by varying the temperature (60 °C, 70 °C and 80 °C). The evaluation of standard Arrhenius plots needs results of exposures at no less than 3 different temperatures. To obtain the time dependence of the oxidative degradation, several test specimens were immersed in the same autoclave and were taken out for time to time to test their mechanical properties. Tests are completed when the criteria to lose strength (50 % retained tensile or compressive strength) is fulfilled.

3 TEST RESULTS

Tests were carried out according to ISO 13438 to compare the two test methods: oven test and HPAT. The results are summarized in Table 1. The test material was a geospacer made of high density polyethylene (HDPE). The compressive strength was tested before and after exposure. Additionally the change of compressive strength was tested in high-pressure autoclave after different duration of immersion. The test results are shown in Figure 3.

Table 1. Comparison of oven test and HPAT acc. to ISO 13438 (material: geospacer 1 made of HDPE)

Characteristic	Oven test (100 °C, 28 d)	HPAT (80 °C, 50 bar, 14 d)
Retained compressive strength	>100 %	5 %

Table 1 shows that the residual compressive strength after exposure acc. to ISO 13438 in high-pressure autoclave is 5 % while the specimens after storage in oven at higher temperature don't show any loss of mechanical strength. By the tests for the same lifetime criteria of 25 years, the geospacer 1 withstood the conventional oven test but failed the HPAT and the compressive strength was dropped below 50 % residual strength within few days immersion (Figure 3). Therefore, it can be concluded that the HPAT is the higher requirement concerning the oxidation resistance according to ISO 13438.

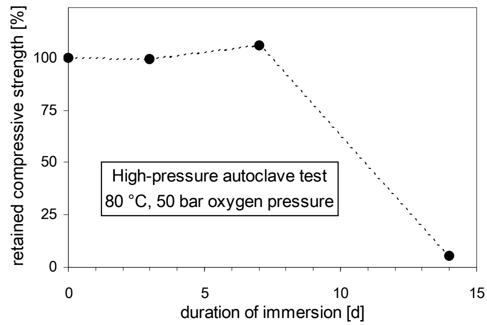

Figure 3. Change of compressive strength during the immersion in high-pressure autoclave at 80 °C and 50 bar oxygen pressure (material: geospacer 1 made of HDPE).

The duration of immersion to begin the decrease of mechanical properties is dependent on the content and type of stabilizer in a geosynthetic. Figure 4 shows the loss of tensile strength in dependence of the duration of immersion in the high-pressure autoclave for a woven (GTX-W) and a nonwoven (GTX-N) geotextile with different type of stabilizers. The rapidly oxidative degradation at the end of the consumption of stabilizer is typical for phenolic stabilized materials. The continuous loss of mechanical strength without distinct induction period is typical for HAS stabilizers (Boehning et al. 2008).

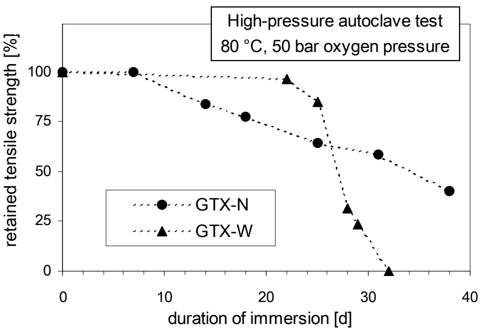

Figure 4. Retained tensile strength depending on duration of immersion in high-pressure autoclave (80 °C, 50 bar oxygen pressure) for two geotextiles with different type of stabilizers.

Figure 5 shows an example for the influence of content of stabilizer in two flexible polyolefin (FPO) polymeric geosynthetic barriers on the tensile properties. The storage in oven for 12 months at 80 °C did not cause significant change of the tensile properties of this materials (SKZ 2005).

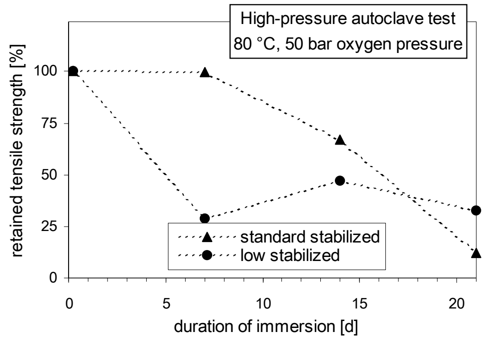

Figure 5. Retained tensile strength depending on duration of immersion in high-pressure autoclave (80 °C, 50 bar oxygen pressure) for different FPO polymeric geosynthetic barriers with different content of stabilizer.

In case of geosynthetics, with rapid loss of mechanical properties, can one observe a decrease of oxygen pressure after certain duration of immersion in the high-pressure autoclave (Figure 6). The decrease of oxygen pressure is small but significant. We observed that the time to decrease of oxygen pressure is the same time when the mechanical properties begin to diminish. The decrease of oxygen pressure indicates that the stabilizer in the test material is consumed and the oxidation of polymer begins.

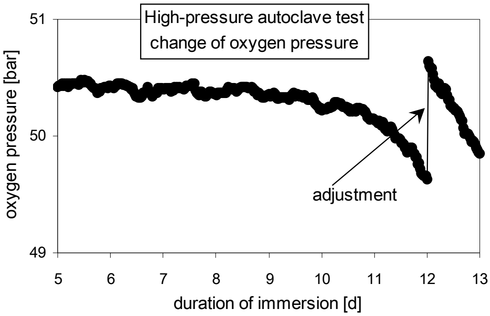

Figure 6. Typical curve for change of oxygen pressure during the immersion in high-pressure autoclave due to oxidative degradation of specimen.

To obtain the estimated lifetime of a geospacer 2, high-pressure autoclave tests were carried out at different oxygen pressures (10 bar, 20 bar and 50 bar) by varying the temperature (60 °C, 70 °C and 80 °C). The criterion of failure was 50 % retained compressive strength. The regression calculation was carried out using the software Table Curve 3D (Systat Software, Inc., Richmond, CA USA). The lifetime was extrapolated to atmospheric condition by using a model with a modified Arrhenius equation (Schröder et al. 2008):

$$1/Y = A \cdot \exp[(B + C \cdot p)/T]$$

Y = lifetime in years
A, B and C = parameters
$B = E_A/R$
E_A = activation energy in J/mol
R = universal gas constant in J/(K · mol)
p = oxygen pressure in bar
T = temperature in K

The 3D-fitting curve of the regression calculation shows Figure 7.

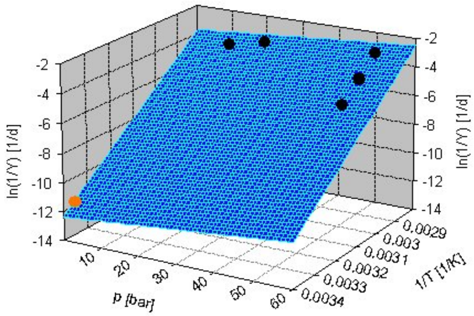

Figure 7. Extrapolation of lifetime to atmospheric condition (25 °C, 0.21 bar O_2 pressure) with a modified Arrhenius equation.

For atmospheric condition (25 °C, 0.21 bar oxygen pressure) an estimated lifetime about 300 years was obtained. The calculated activation energy is E_A = 119 kJ/mol. The conservative evaluation of tests which were carried out at 50 bar by varying the temperature results an estimated lifetime about 100 years (25 °C, 50 bar oxygen pressure) according to Arrhenius.

4 CONCLUSIONS

Tests according to ISO 13438 have shown that for the same lifetime criteria of 25 years, the tested material withstood the conventional oven test but failed the HPAT. It can be concluded that the test of resistance to oxidation with the high-pressure autoclave method makes the higher requirement.

By varying the temperature and the oxygen pressure the HPAT method allowed to estimate the lifetime of geosynthetics in relative short test duration. This fact is a considerable advantage over the conventional oven test.

The decrease of oxygen pressure in the high-pressure autoclave indicates that the degradation of specimen began. This observation is useful to save time because without additional information there is a need to open the autoclaves from time to time and take specimens for mechanical tests to get information about the change of properties. This effect helps to find the onset point when the material begins to lose mechanical strength.

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