OXIDATIVE RESISTANCE OF GEOSYNTHETICS: PRACTICAL ASPECTS AND DEVELOPMENTS OF TESTING AT ELEVATED TEMPERATURE AND OXYGEN PRESSURE

Jan Retzlaff[1], Zori Bronstein[2], Jochen Müller-Rochholz[3], Martin Böhning[4], Daniela Robertson[5] & Hartmut Schröder[6]

[1] *tBU - Institut für textile Bau- und Umwelttechnik GmbH. (e-mail: jretzlaff@tbu-gmbh.de)*
[2] *tBU - Institut für textile Bau- und Umwelttechnik GmbH. (e-mail: zbronstein@tbu-gmbh.de)*
[3] *University of Applied Sciences Muenster. (e-mail: muero@fh-muenster.de)*
[4] *Federal Institute for Materials Research and Testing (BAM). (e-mail: martin.boehning@bam.de)*
[5] *Federal Institute for Materials Research and Testing (BAM). (e-mail: daniela.robertson@bam.de)*
[6] *Federal Institute for Materials Research and Testing (BAM). (e-mail: hartmut.schroeder@bam.de)*

Abstract: Service life of geosynthetics made of polyolefin materials is mainly limited by oxidative degradation. Durability of polyolefin products depend on formulation, morphology resulting from manufacturing, design and particularly on the antioxidants used. Although the fundamental oxidative reactions are known, the complex effect of geosynthetic characteristics and external influences mean that durability assessments require practical tests.

The assessment of oxidative durability involves exposure to accelerating conditions as well as the material characterisation with respect to the state of stabilisation and/or degradation (e.g. by tensile testing and/or oxidation induction time).

The autoclave test developed by BAM for durability assessments of polyolefin geosynthetics is based on the simultaneous application of moderately elevated temperatures (up to 80 degrees Celsius) and elevated oxygen pressures (up to 5.0 MPa) in combination with a surrounding aqueous medium. Thus this test method provides several advantages in comparison to conventional oven testing, especially with respect to test duration, impact of surrounding environment and the potential to differentiate and rank oxidative resistance between products of very different dimensions (surface/volume) and stabilisation.

In view of the increasing demand for durability assessments of geosynthetic products with lifetimes significantly exceeding 25 years, the contribution is focused on practical aspects concerning implementation and optimisation of test procedures, conditions and evaluation of results. Furthermore, advanced approaches based on multiple exposure conditions will also be included and corresponding calculations of expected service life in typical applications will be evaluated with respect to significance and repeatability. Thereby involved standards and recommendations - such as EN ISO 13438, Part C or EAG-EDT - are considered.

Keywords: Ageing, degradation, HDPE, laboratory test, oxidation, polypropylene

INTRODUCTION

The durability of polymeric materials for the use in civil structures with a scheduled service life of more than 25 years has to be proven by accelerated tests. This is the case for the aging of geosynthetics in particular. Their durability might be essential for the safety of a construction and has to be taken into account for maintenance purposes. A repair of the construction due to an insufficient durability of polymeric parts can cause enormous financial expenses. Based on the recent EOTA Guidelines (Assumption of working life of construction products in Guidelines for European Technical Approvals and Harmonized Standards, 1999) a proof of an expected service life of at least 100 years is requested for relevant building products. This topic is part of the discussions in technical and standardization committees that are working on recommendations and rules for a durability assessment.

TEST PROCEDURE

Practical investigations to assess the durability of polymeric materials and products are done by using defined test conditions (e.g. elevated temperature) that cause an accelerated ageing to affect typical material properties (e.g. mechanical tensile strength). The evaluation can be done either as a simple index test for rough estimates based on minimal requirements or as a more significant result based on the time dependent behaviour of relevant material properties. The second option includes an extrapolation using the stress parameters of the tests and their interdependence. To get realistic results the circumstances must be chosen according to the practical use of the material.

The working group VI.33 of the Federal Institute for Materials Research and Testing (BAM) in Germany has developed a method to assess the oxidation resistance of geosynthetics made of polyolefins, such as polypropylene (PP) and polyethylene (PE), by using autoclaves. The combination of specific temperatures, an increased oxygen pressure and the presence of an aqueous medium allows an acceleration of the auto-oxidation with simultaneous extraction of additives.

These autoclave tests represent a promising method to replace conventional oven tests. The moderate temperatures during the test procedure provide suitable conditions to assess the effect of stabilizers and anti-oxidation-agents at their working temperatures. Due to the high oxygen pressure and the continuous circulation of the aqueous solvent a sufficient amount of oxygen is available for the auto-oxidation of polyolefins.

The time frame for the autoclave tests depends on characteristics and quality of the material and the prevailing application conditions. Generally three temperature levels of 60 °C, 70 °C and 80 °C at 50 bar(5 MPa) oxygen pressure and two additional oxygen pressure levels of 10 and 20 bar at 80 °C have been proven to be suitable for the testing of geosynthetics. In this case the time needed to get reliable results varies from approximately 300 days at the lowest temperature to 30 days at the highest.

That allows both, rather short index tests at higher temperatures and a calibration of these short term tests with long-term tests at lower temperatures. It is a comfortable approach to estimate the residual service life of polyolefins in a construction with accelerated tests. The described standard procedure for geosynthetics can be modified according to specific environmental conditions. This might be interesting for investigations regarding the ageing effect of water that penetrates the mountain rock until it reaches a liner and drainage system of a tunnel. Amongst other components the infiltrating water may contain heavy metal ions that may have a catalytic effect on the oxidation.

Test Facility

The autoclave is the main part of the test equipment. A small gas-proof pressure vessel with a volume of approximate 8.5 litres is commonly used for these tests. This part with the gas connectors and pressure gauge is shown in Figure 1. While the pressure is limited to 50 bar during the tests, the autoclaves have the ability to withstand a pressure of 100 bar. That gives the needed space for further research at higher pressure levels also. Depending on the specimen size and the dimensions of the specimen holder the bottom part of the autoclave will be filled with the test liquid between $^2/_3$ and ¾ of its total volume. The samples must be completely immerged.

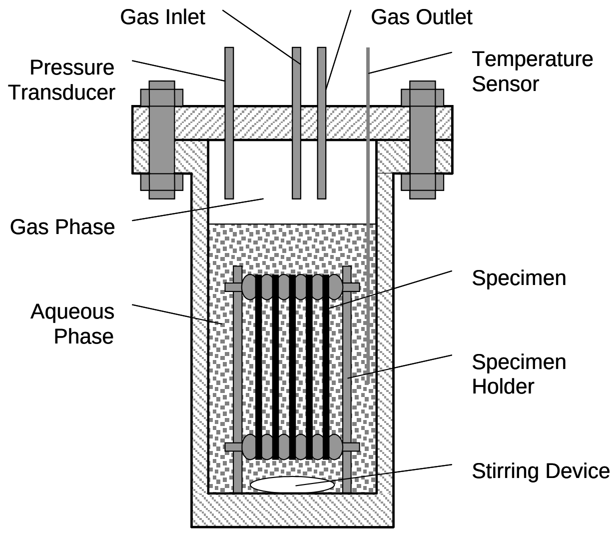

Figure 1. Scheme of an autoclave as used by tBU

The temperature of the liquid will be heated and kept on a constant level by a jacket heating around the body. To force the heating process additional jacket heaters around the flange and/or the cap can be useful. This is important because the accelerated oxidation will be disturbed when extracting specimens out of the autoclaves. It will be done 5 to 6 times during a test series. Though the last extraction defines the end of the test and allows cooling down the test facilities. The temperature is under steady control by a temperature sensor inside the autoclave.

The oxygen is supplied in pressurized gas bottles. To provide the needed pressure in the system the original pressure of the gas bottles is used. The gas bottle is considered to be empty if the bottle pressure is not sufficient to hold 50 bar respectively 5.0 MPa in the system. Even if there is a lack of efficiency in using the total volume of gas, this is a very simple and safe way to provide oxygen for the chemical reaction at the prescribed pressure. A pressure transducer delivers the needed data to check and adjust the pressure.

The aqueous phase has to be under a balanced movement. It has to be ensured that the oxygen saturated liquid moves between the specimens whereas the fluid does not foam. It has been proven to be suitable to activate the stirring device by a magnetic stirrer that is stored underneath the bottom of the autoclave. Figure 2 shows autoclaves in use.

Evaluation

The final evaluation of the tests shall lead to a service life prediction of building materials for more than 100 years and will be done according to (Boehning 2008). After each extraction of the specimen their residual strength will be measured and compared with the original strength before the test. A dependence of the oxidation resistance was found from the temperature which accelerates the chemical reaction in general and the pressure that influences the presence of oxygen. The pressure is an additional dimension for the commonly used Arrhenius plot. (Boehning 2008) is calling this approach a 3 dimensional extrapolation.

THE BENEFITS OF USING THIS METHOD

The defined liquid environment inside the autoclaves leads to a new quality of testing. At first hand the oxidation resistance and the efficiency of anti-oxidation agents can be assessed. Because of the more moderate temperature this can be done closer to the real environmental conditions. That is the main goal of this test procedure. Combining the various test media and conditions with each other is an approach to get a more reliable formulation for

- a fast and efficient procedure with enhanced differentiating ability compared to the conventional oven test, which is based on temperature increase and evaporation of stabilizers only
- a moderate variation of the influencing stresses (temperature ≤ 80 °C)
- a simulation of the most essential characteristics of the installation environment (temperature, oxygen concentration, extraction of additives and anti-oxidants as well as site-specific chemicals) in an specific aqueous surrounding
- modelling an individual stress scenario to evaluate the durability for various application areas of the polymeric materials
- an improved test with a higher efficiency regarding the results and a shorter test duration to provide a durability assessment for the design process of new products.

The steady acceleration of product development cycles and the use of polymeric materials in more demanding applications have caused an urgent need to predict the oxidation resistance of building materials over the whole service life in a reasonable time.

Figure 2. Autoclaves in use

THE PRACTICAL PURPOSE AND FUTURE OPPORTUNITIES

The Autoclaves used for the testing of Geosynthetics have a volume of 8.5 litres. These dimensions are feasible for small samples of Geosynthetics but also for other more massive materials like samples of pipeline materials or machine parts. The high potential of this test method can be used in many parts of the construction and civil industry where polyolefins are used and a total service life prediction or an assessment of the residual serviceability is relevant.

When thinking of the sealing and drainage of tunnels with Geosynthetics and pipes the leachate from the construction site can be used as test liquid to store the polymer materials in the autoclaves. This allows an estimate of the material behaviour under the specific conditions of the local environment. The combination between pressure and elevated temperature is interesting for this type of applications. How the effect of temperature and pressure can be used for future service life assessments has been described by (Boehning 2008)? The same publication explains that there is still an amount of research work to be done to specify the effect of the pressure. The so far used Arrhenius plot is a pure temperature depending procedure which results are not influenced by the applied pressure. The idea is to apply additional factors to remedy this shortcoming.

At this stage further investigations are needed to develop some procedural steps like more practical indicators for the extraction on time of the material samples and to improve the evaluation of the test results.

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Corresponding author: Mr Jan Retzlaff, tBU - Institut für textile Bau- und Umwelttechnik GmbH, Gutenbergstr. 29, Greven, Germany. Tel: +49 2571 98720. Email: jretzlaff@tbu-gmbh.de.

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