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Test method

The oxidative resistance of polymeric geosynthetic barriers (GBR-P) used for road and railway tunnels

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ABSTRACT

The results of autoclave tests according to DIN EN ISO 13438 (method C) and conventional oven tests in line with DIN EN 14575 are presented for the evaluation of the resistance to oxidation of geosynthetic barriers GBR-P based on polyethylene. These GBR-P products are used as a barrier layer against water in road and railway tunnels. The residual mechanical stability of the exposed materials was determined by tensile testing. The remaining activity of the stabiliser was investigated using the OIT method. The results of both accelerating test methods are discussed and compared in detail. Based on autoclave tests at three temperatures (e.g. 60, 70 and 80 °C) and 50 bar oxygen pressure, and additionally two more measurements at 80 °C and 10 and 20 bar oxygen pressure, a modified Arrhenius extrapolation is used for an assessment of the expected service time of one of the GBR-P products.

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1. Introduction

Geosynthetic barriers, mostly based on polyolefins, such as polyethylene (PE) or polypropylene (PP), ethylene-propylene copolymer (EPM) or polyethylene-ethylene-vinyl acetate copolymer (PE/EVA), are innovative products used in several civil engineering applications for about 40 years. Their possible functions [1] are barrier to liquids and gases, protection, reinforcement and separation. Several areas of application, such as landfill sealing, water-collecting basins or tunnel waterproofing, require service times up to 100 years [2–4], since repair or exchange is nearly impossible and consequences of failure are severe. The long term durability of these geosynthetic barriers is governed by their resistance to thermal oxidation and the loss of stabilizer by extraction due to special natural environments (e.g. mountain water in tunnels or leachate in landfills) and oxidative consumption of the stabilizer.

In the BAM German Federal Institute for Materials Research and Testing, a new type of accelerated ageing test method, the so-called "autoclave test", was developed some years ago for geotextiles by Schröder et al. [5,6] and Böhning et al. [7] to investigate the oxidative resistance of polymers. The autoclave test is performed at 80 °C, a significant lower temperature than often used in conventional oven tests (80–130 °C) [8,9], under high oxygen pressure with the specimen exposed to a liquid alkaline media. Based on this concept, an alternative screening test has been proposed and standardized for geosynthetic products (DIN EN ISO 13438, method C1 and C2). With this test method, a service time of at least 25 years can be proven. For a more detailed assessment of much longer functional service times of geosynthetic products, an extended approach is suggested. The durability is measured at least at three temperatures (60, 70 and 80 °C) and 50 bar oxygen pressure, and additionally at 80 °C at two more oxygen pressures (10 and 20 bar) to include the dependence of the oxidation on oxygen pressure. Based on these data, an Arrhenius extrapolation for temperature and pressure dependence is used to estimate service life under application conditions.

Testing long term oxidation resistance is difficult because of the complexity of the autoxidation chain

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reaction mechanism [10–12] and the simultaneous physical transport processes (e.g. diffusion) of reactive species (e.g. oxidation products or additives). In addition, the loss of antioxidants, e.g. by the extracting capacity of an aqueous medium with its additives, may have a strong influence.

At least for polyolefins, the application of temperatures above 80 °C has to be regarded as critical, since a distinct change in the mechanism of oxidation may lead to distinct deviations from a simple Arrhenius behaviour, e.g. the effective activation energies of the oxidative degradation might change significantly in certain temperature regions [13–16]. However, the oxidation proceeds very slowly working at or below 80 °C. Since extrapolation of the usual Arrhenius plots needs data for at least 3 temperatures which should differ by about 10 K [17], the lowest testing temperature would be 60 °C.

The advantage of our methodology is that (a) lower testing temperatures can be realized by accelerating the degradation with elevated oxygen pressure at moderate temperature ≤80 °C and (b) the impact on stabilizers by various aqueous mediums, e.g. extraction or migration, can be considered. The autoclave test is a possibility to measure the oxidative resistance in comparison to the oven test. One disadvantage might be that the application of higher oxygen pressures includes the possibility of promoting some reactions, just as too high temperatures may achieve this for oven tests.

The German Federal Ministry of Transport, Building and Urban Affairs (BMVBS), represented by Federal Highway Research Institute (BASt), commissioned the BAM to investigate the applicability of the autoclave test method for the assessment of the oxidative stability of polyolefin-based GBR-P products applied in road tunnels. This publication gives an overview of the methodology of the autoclave test and describes the main findings of the research.

2. Materials

After surveying the current market of polyolefin-based GBR-P products for tunnel liners, four commercially available GBR-P geomembranes were selected and provided by four manufacturers. These GBR-P products are based on polyethylene, and the thicknesses of the geosynthetic barriers varies between 2 and 3.2 mm. GBR-P products consist of two co-extruded layers. The primary layer is black due to the added carbon black master-batch, and the thin secondary layer (≤0.2 mm), the so-called signal layer, is brightly coloured by use of some other additives (e.g. TiO_2).

Table 1 shows material properties such as the geomembrane thickness, the density, the OIT values (as delivered) and the tensile properties. Basic information about the products such as the type and amount of stabilizer was not disclosed by the manufacturers. However, products 1, 2 and 3 are made from VLDPE (Very Low Density Polyethylene), while product 4 is a blend of polyethylene-ethylene vinyl acetate copolymer. These products are stabilized to some extent by phenolic (e.g. IRGANOX 1010) and phosphitic antioxidants (e.g. IRGAFOS 168).

3. Experimental

3.1. Accelerated ageing methods

For the accelerated testing of the oxidative degradation, two different methods were used. First, the autoclave tests according to DIN EN ISO 13438 (method C) was applied to all products and, second, the conventional oven test according to DIN EN 14575 was applied to products 1 and 2 for comparison. The accelerated ageing test methods are described in detailed in the following sections 3.1.1 and 3.1.2.

3.1.1. Autoclave test (DIN EN ISO 13438)

Autoclave tests were performed in a pure oxygen atmosphere with pressures of 10, 20 and 50 bar at a temperature of 80 °C, and additionally measurements with a pressure of 50 bar at a temperature of 60 and 70 °C. The test specimens were immersed in a test medium of 0.01 M sodium bicarbonate ($NaHCO_3$). The pH value was adjusted at pH 10 by adding a solution of 1 M sodium hydroxide (NaOH). All autoclave exposures were conducted in order to follow the time-dependent degradation of the mechanical properties of the polymeric material. Therefore, at least 5 specimens, or sometimes even more, were removed after different exposure times and the tensile properties and the OIT value were determined.

Fig. 1 shows a schematic view of the autoclave test equipment including all instruments and monitoring devices. The temperature and the pressure were monitored and recorded at least every 15 min by means of an electronic data recorder (Eurotherm 6100). The temperature of the autoclave was controlled by an external heating jacket with a separate PT100 temperature sensor connected to a PID-temperature controller (Eurotherm 2216E). The heating power line was equipped with an electrical contactor controlled by the internal temperature monitoring to prevent overheating of the system. Safe and reliable operation of the autoclaves requires control and monitoring of the relevant parameters, especially for long-term experiments. All the relevant instruments and transducers were calibrated in order to get reliable and reproducible results. The

Table 1
Properties of the GBR-P products: sample thickness measured over the entire width of the geomembrane, density, OIT values and the tensile properties.

Product	Thickness/mm	Density/g/cm^3	OIT value		Tensile properties	
			180 °C/min	200 °C/min	Tensile strength at break/N/mm^2	Elongation at break/%
1	2.19 ± 0.01	0.9184	476	119	253 ± 8	1116 ± 29
2	3.24 ± 0.01	0.9129	118	9	267 ± 10	1198 ± 14
3	2.22 ± 0.01	0.9145	138	23	252 ± 5	1130 ± 12
4	2.09 ± 0.01	0.9465	679	321	189 ± 8	1037 ± 34

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