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Test Report

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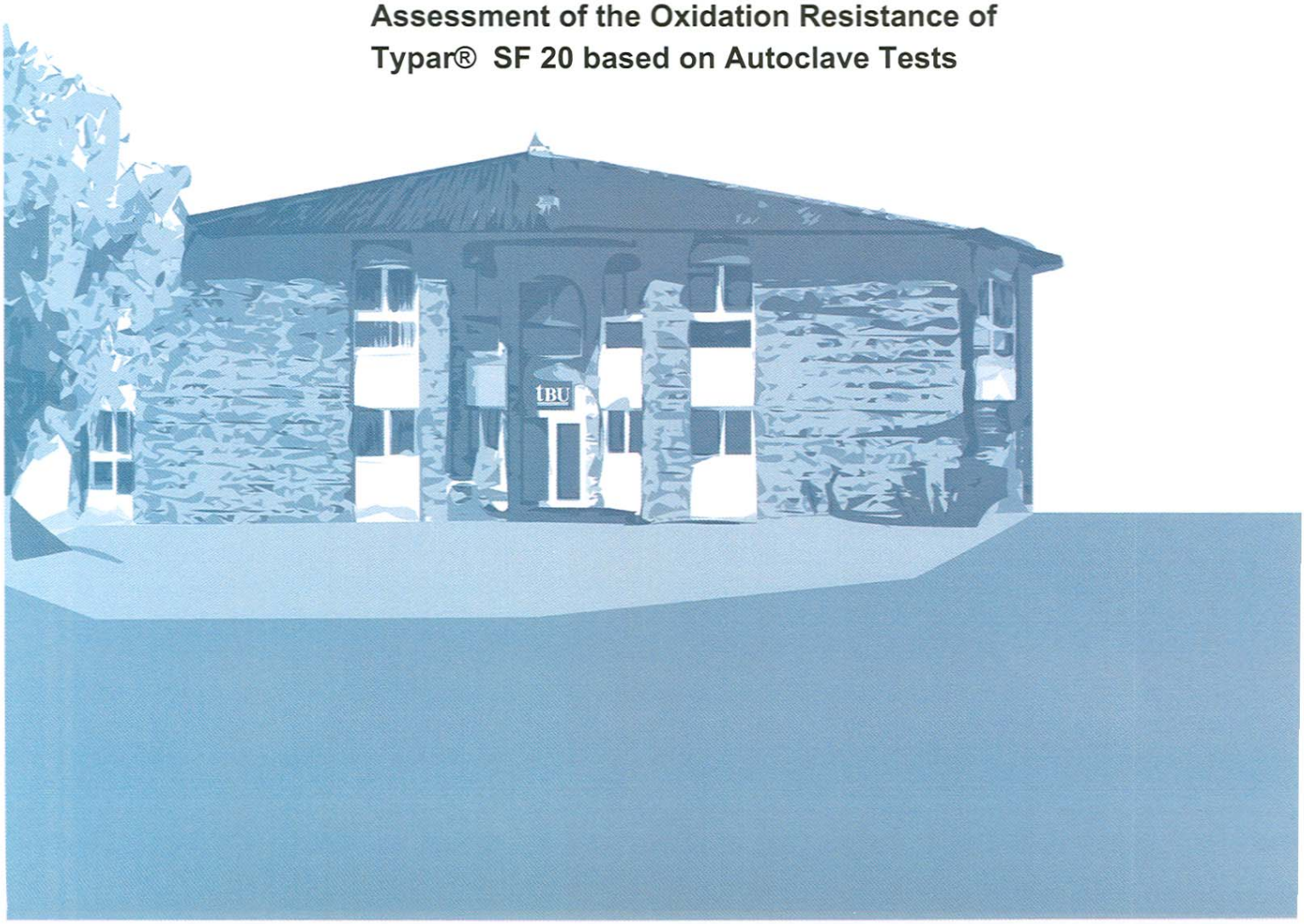
Assessment of the Oxidation Resistance of Typar® SF 20 based on Autoclave Tests



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1 Order

We have been requested to assess the durability for a minimum service life of 100 years in terms of aging of Typar® SF – nonwovens by DuPont de Nemours (Luxembourg) S.à.r.l. Typar® SF 20 has been provided as the lightest member of the Typar® SF – product family.

2 References

2.1 Procedure documentation

The chosen procedure of assess the aging of polyolefines is referenced to various standards and publications, which are listed below.

[DuPont-2006] DuPont: Typar SF – Geotextile. Product description. L11810 Luxembourg: DuPont, 07/2006

2.2 Literature

[BOEHNING-2008] Boehning, M., Robertson, D., Schroeder, H. F.: Autoclave testing a new Approach for the Evaluation of Oxidative Long-Term Resistance of Geosynthetics. Proceedings EuroGeo4, Edinburgh: 2008

[ISO-10319] Norm DIN EN ISO 10319: Geosynthetics – Wide-width tensile test

[ISO-13434] Technical Specification ISO/TS 13434: Geosynthetics – Guidelines for the assessment of durability

[ISO-13438] Norm DIN EN ISO 13438: Geotextiles and geotextile-related products: Screening test method for determining the resistance to oxidation

[ISO-20432] Technical Report ISO/TR 20432: Guidelines to the determination of long-term strength of geosynthetics for soil reinforcement

[RETZLAFF-2008] Retzlaff, J; Bronstein, Z.; Müller-Rochholz, J.; Böhning, M.; Robertson, D.; Schröder, H.: Oxidative Resistance of Geosynthetics: Practical Aspects and Developments of Testing at Elevated Temperature and Oxygen Pressure. Proceedings EuroGeo4, Edinburgh: 2008

[tBU-2008] tBU: The Use of Autoclaves to Assess the Oxidation Resistance of Building Materials made from Polyolefins. tBU Newsletter 17. www.tbu-gmbh.de/en

[EN-29073] Norm DIN EN 29073: Textiles, Text methods for nonwovens. Part 3: Determination of tensile strength and elongation

3 General

Polyolefins are a group of polymers which members polypropylene and polyethylene are commonly used to manufacture geosynthetics. The exposure to environmental conditions will lead to a chemical degradation of the material which is accompanied by a loss of their mechanical properties. Because of this environmental decomposition the service life of geosynthetics made from polyolefins is limited. Depending on the final application a service life of more than 100 years may be required. The degradation of the polymers during their service life is caused by reactions with air oxygen. The degradation rate depends on various parameters as there is the polymeric structure, the environmental conditions and in general, the type and the amount of stabilizers. The environmental parameters are UV-irradiation, oxygen, temperature and humidity which lead to photo-oxidation respectively thermo-oxidation.

The resistance of polyolefins to oxidation can be extended by the addition of antioxidant stabilizers. Because these stabilizers are consumed by oxidation, the amount and the type of stabilizers have an essential effect on the practical service life of geosynthetics. [ISO-20432]

Because the oxidation of polyolefins at the temperature of their end use may not result in a noticeable degradation within a reasonable time, the chemical decomposition process is accelerated by an increased temperature and sufficient oxygen concentration. Such a test environment can be provided by using autoclaves. [ISO-13434]

The methodology of the autoclave test consists of the material exposure for accelerated ageing and subsequent characterization of the state of degradation reached after a certain exposure time. The latter is based on mechanical testing, but also other material properties may be considered in this respect. For the results presented here tensile testing was employed.

4 Laboratory Test

4.1 Product description

Typar® SF is product family consisting of a range of thermally bounded nonwovens made from continuous polypropylene (PP) fibres. A Typar® SF20 has been investigated as the lightest type to be representative for the whole Typar® SF range. [DuPont-2006]

4.2 Test procedure

The autoclave test for the evaluation of the oxidative durability of polymeric materials for geotechnical applications - currently also established as index test in [ISO 13438], method C, is based on the exposure of test specimens to higher oxygen pressures up to 5.1 MPa at elevated temperatures up to 80 °C. At the same time the specimens are immersed in a well defined aqueous medium in order to simulate the possible extraction of antioxidants in aqueous or moist environments in addition to the oxidative degradation.

Standard conditions of the index test for oxidation resistance are defined in [ISO 13438] as 80 °C and 5.1 MPa oxygen pressure. The test specimens are immersed in 0.01 mol/l $NaHCO_3$ aqueous solution, the pH adjusted using 1 mol/l NaOH to 10 at 20 °C.

In order to record the time-dependent degradation behaviour all samples were inserted together at the beginning and batches of samples were consecutively removed according to their intended duration of exposure and later subjected to subsequent characterizations, e.g. tensile testing. A summary of results is presented in Table 1.

For the assessment of the lifetime parameters (i.e. the approach to obtain a parameter to characterize the expected lifetime using the testing conditions mentioned above), this procedure had to be carried out at several temperatures following the approach of [BOEHNING-2008] and [RETZLAFF-2008]. In this case temperatures of 70 °C, 77.5 °C,

80 °C and 85 °C at 5.1 MPa oxygen pressure and additionally at 80 °C at 1.1 MPa and 2.1 MPa.

The autoclave exposure was performed in stirred stainless steel pressure-vessels (autoclaves). The total volume of the autoclave is 8.6 litres with a headspace above the liquid phase of 20 % gas phase. The temperature of the aqueous medium was measured using an immersed PT100 temperature sensor. A temperature constancy better than 0.5 °C was achieved. The pressure of the gaseous phase was measured using a piezo-resistive transducer [tBU-2008].

A schematic view of the autoclave apparatus is shown in Figure 1.

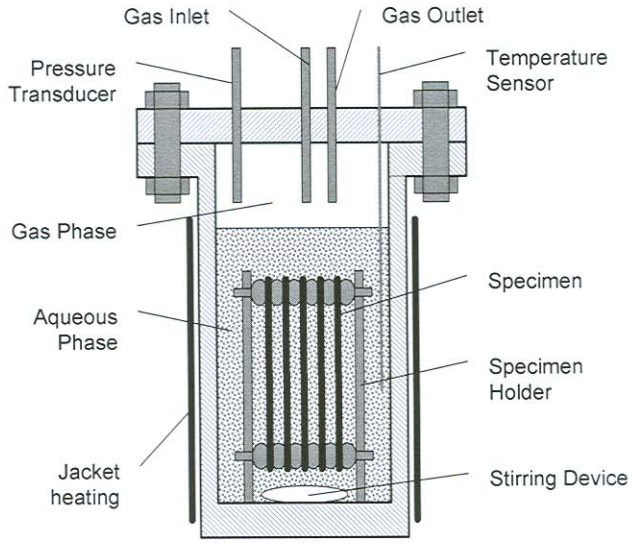

Figure 1: Scheme of the autoclave

4.3 Tensile Strength Measurements

All tensile test measurements were performed with a Zwick Model Z050 in an air conditioned environment at 20 °C and a relative humidity of 65 % in line with [ISO 10319]. The essential tensile tests of the specimens and the specimen preparations were performed according to [EN 29073]. The material was tested in strips, 25 mm wide and approximately 100 mm long. A constant velocity of the drawing clamp of 50 mm/min was applied.

5 Evaluation

The results of the autoclave tests at four temperatures (70 °C, 77.5 °C, 80 °C and 85 °C) and three pressures (1.1, 2.1 and 5.1 MPa) were used for an estimate of the expected lifetime of a geosynthetic product resulting from oxidative durability investigations based on extrapolation of [BOEHNING-2008] employing a modified Arrhenius equation (1):

$$t = A * \exp\left[\frac{(B + C * p)}{T}\right] \quad (1)$$

With t being the time to failure resulting from the tensile test ($t_{[50\%]}$) regarding a corresponding failure criterion of 50 % retained tensile strength at a temperature T,
p denotes the pressure,
parameters A and B correspond to the pre-exponential factor and activation energy and C is the additional parameter introducing the pressure dependence.

Table 1 gives a detailed overview of the achieved results.

Figure 2 shows the 3-D-fitting surface of the modified Arrhenius model representing the test results for the investigated geosynthetic nonwoven Typar® SF 20. The basis for the calculation of the lifetime parameter is the exposure time for 50 % retained tensile strength ($t_{[50\%]}$) of the material which was determined from the time dependent curves obtained for six different autoclave test conditions.
For Typar® SF 20 at application conditions of 25°C (298 K) and 0.21 bar (21 kPa) O_2 – pressure, a lifetime parameter of $\Theta_{50\%}^{298\,K}$ = 358 years is determined and an apparent activation energy of 113 kJ/mol is calculated from equation (2).

$$E_A = \frac{B}{R} \quad (2)$$

E_A is the apparent activation energy,
R is the universal gas constant.

Table 1: Retained tensile strength in dependence of the duration of oxidation exposure

Temperature	Pressure	Test duration	Tensile strength Fm [N]			Retained tensile strength	$t_{50\%}$, interpolated
		[d]	x	s	v / %	[%]	[d]
80.0 °C (353 K)	11 bar (1.1 MPa)	1*	67.67	9.62	14.21	100.0	97.7
		14	68.67	10.84	15.78	101.5	
		28	76.61	7.86	10.26	113.2	
		56	73.56	10.27	13.96	108.7	
		77	58.49	11.77	20.12	86.4	
		91	45.53	9.85	21.64	67.3	
		99	30.46	6.86	22.53	45.0	
		104	26.20	7.47	28.5	38.7	
80.0 °C (353 K)	21 bar (2.1 MPa)	1*	67.67	9.62	14.21	100.0	60.0
		28	86.79	7.40	8.53	128.3	
		63	28.81	10.47	36.34	42.6	
80.0 °C (353 K)	51 bar (5.1 MPa)	1*	67.67	9.62	14.21	100.0	45.7
		21	70.82	14.55	20.54	104.7	
		33	58.61	16.02	27.34	86.6	
		35	52.38	10.16	19.40	77.4	
		36	51.92	8.09	15.58	76.7	
		40	44.77	8.09	18.08	66.2	
		49	27.76	8.35	30.06	41.0	
85.0 °C (358 K)	51 bar (5.1 MPa)	1*	67.67	9.62	14.21	100.0	28.0
		28	33.68	6.50	19.30	49.8	
		33	12.15	8.02	66.01	18.0	
77.5 °C (350.5 K)	51 bar (5.1 MPa)	1*	67.67	9.62	14.21	100.0	53.8
		47	48.59	13.93	28.67	71.8	
		54	34.50	6.93	20.09	51.0	
		61	17.20	6.99	40.64	25.4	
70.0 °C (343 K)	51 bar (5.1 MPa)	1*	70.73	14.35	20.29	100.0	142.2
		14	78.61	16.13	0.21	111.1	
		28	68.95	11.67	0.17	97.5	
		42	80.44	15.53	0.19	113.7	
		56	75.39	9.89	0.13	106.6	
		77	69.58	11.54	0.17	98.4	
		104	62.69	8.62	0.14	88.6	
		119	52.43	2.49	0.05	74.1	
		143	34.66	6.42	0.19	49.0	

* Reference material has been stored for 24 hours in $NaHCO_3$ @ relevant temperature but without overpressure of oxygen

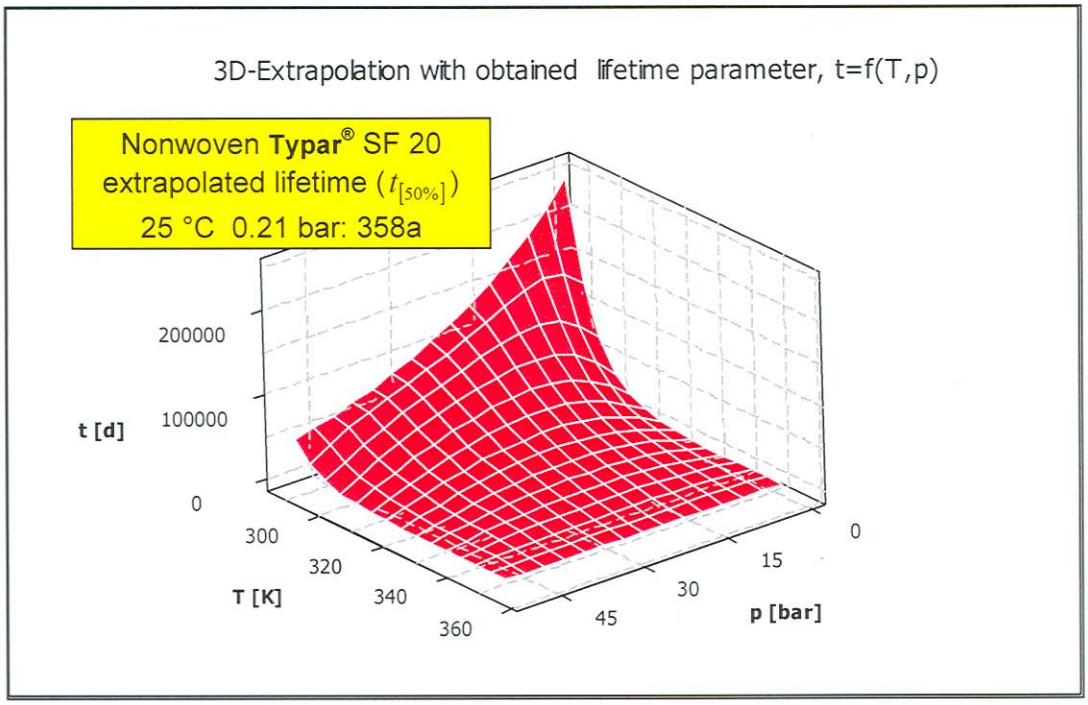

Figure 2: 3D-extrapolation with the obtained a lifetime parameters at 25 °C and 0.21 bar oxygen pressure of the Typar® SF 20

6 Conclusions

The achieved results of the described test series have shown the satisfactory durability of Typar® SF 20 to serve more than 100 years in civil applications providing a sufficient mechanical strength. This behaviour can be expected for the whole product family of Typar® SF - nonwovens.

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