AUTOCLAVE TESTING: A NEW APPROACH FOR THE EVALUATION OF OXIDATIVE LONG-TERM RESISTANCE OF GEOSYNTHETICS

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Abstract: Oxidative long time resistance is essential for many polyolefin based products, especially if repair or exchange is not possible as encountered in many building and geotechnical applications. Current regulations and recommendations for geosynthetics cover mainly the needs of a maximum lifetime of 25 years, though many applications require longer lifetimes up to 100 years and more. Reliable testing of the long time oxidation resistance is complicated by the complex interplay of physicochemical processes and reactions in combination with the need of relatively short testing durations, usually not exceeding 12 months. Thus for conventional atmospheric oven testing, the use of temperatures higher than $100 \,^{\circ}$ C is inevitable, making conclusive Arrhenius extrapolations often questionable.

Practical assessments of oxidative durability consist of exposures under accelerated ageing conditions with subsequent characterization of exposed material samples with respect to their residual stabilization and/or their mechanical properties.

The autoclave test combines exposures at elevated temperatures with higher oxygen pressures and is performed with material samples immersed in a well defined aqueous medium representing critical environments in geosynthetic applications and including extraction of additives. Under these conditions meaningful results can be obtained within reasonable testing durations at only moderately elevated temperatures up to 80 °C.

Application of different physicochemical methods, such as tensile testing, OIT /HP-OIT and analysis of residual antioxidant content (ICOT), in order to determine the time dependent degradation behaviour, reveal valuable information about mechanisms and effectiveness of the used antioxidants. Corresponding results for exposures at different temperatures and oxygen pressures are the basis for a 3D-extrapolation in order to estimate the expected service life. Different empirical models are presented and discussed with respect to their practical impact.

Keywords: ageing, degradation, geotextiles, polyolefin, polypropylene, oxidative resistance, lifetime prediction

INTRODUCTION

Geosynthetics, mostly based on polyolefin materials like polypropylene (PP) or polyethylene (PE), are innovative construction products since about 40 years in civil engineering applications, e.g. as barrier to liquids, for filtration, reinforcement, separation and surface erosion protection. The main cause for ageing and degradation of polyolefin materials is oxidation (Jacobson et al. 2001, Commercuc et al. 1997), frequently in combination with both installation damages and mechanical stresses. Sufficient oxidation resistance for geosynthetic and other long-term applications can usually only be achieved by incorporation of antioxidants (AO) into polyolefin raw materials. On this account it is important to have quick and reliable methods for the assessment the oxidative durability of geosynthetic materials. Testing long-term oxidation resistance is complicated due to the complexity of the chain reaction mechanism of oxidation (Gillen et al. 1995, Zweifel 1998) and simultaneous physical transport processes of reactive species. Furthermore there is the exigency of relatively short testing durations, which usually should not exceed 12 months. Essentially the acceleration of the oxidation reaction can only be enforced by use of elevated temperatures and/or higher oxygen concentrations (pressures). Furthermore the loss of antioxidants due to extraction can be taken into account by immersion of the material in a suitable aqueous medium.

Accelerated oxidation tests of polyolefin materials (especially PP) at temperatures higher the 80°C is often critical, because of a significant change in the mechanism of oxidation in this temperature range, e.g. by decomposition of peroxide intermediates or other physical or chemical transitions, like morphological changes or diffusion-limited oxidation (DLO) effects. Achimsky et al. described a transition at 80 °C in polypropylene oxidation kinetics, which was attributed to the coexistence of competitively modes of hydroperoxide decomposition dominating either above or below the transition temperature (Achimsky et al. 1997). This finding may not necessarily be true for all kinds of polypropylene materials, as some kinds may show an Arrhenius relationship over a broad temperature range including 80 °C. The transition temperature may be affected mainly by the nature of the stabilizer and its concentration or the influence of residues of the polymerization catalyst (Gijsman et al. 1993). Furthermore, it must be noted that stabilized polypropylenes stabilized with sterically hindered amine stabilizer (HAS) may exhibit a completely different temperature dependent behavior (Gugumus 1999).

The conventional method for accelerated ageing is the oven test in line with DIN EN ISO 13438, method A, B. These methods are working at temperatures significantly higher than 80 °C at ambient atmospheric conditions.

With the limitations discussed above in mind an accelerated ageing test addressing the oxidative degradation and extraction of stabilizers has been developed. The acceleration is realized by applying moderate temperatures up to 80°C at elevated oxygen pressure up to 5.1 MPa and immersion of the test specimens in a well defined aqueous medium. Our investigations are focused on the assessment of polyolefin materials for geosynthetic applications.

Based on this so-called autoclave test a screening test method is established in EN ISO 13438, method C, covering service life of geosynthetics up to 25 years. This test is implemented using fixed durations of autoclave exposure of 14 and 28 days, respectively, and subsequent mechanical testing. The methodology also allows a more detailed inspection of the oxidative degradation when the time-dependent behavior is considered, i.e. the course of the reference property (e.g. residual tensile strength) is determined ranging to exposure durations where substantial damage of the material is observed. Furthermore a potential approach for an assessment of expected lifetimes for this kind of materials is proposed based on tests at three temperatures (e.g. 60, 70 and 80 °C) at 5.1 MPa oxygen pressure and additionally two more measurements at 80 °C and 1.1 and 2.1 MPa oxygen pressure.

The general advantage of our methodology is that (a) reasonable testing durations can be realized at lower testing temperatures, (b) the rate limiting process is the oxidation reaction and (c) the impact of various aqueous media (e.g. different pH) can be considered, e.g. with respect to extraction of stabilizers. This offers quite new possibilities in comparison to the oven test.

In general the durability of a geosynthetic material is influenced by many factors such as density, morphology, degree of crystallinity, antioxidant composition, polymer/blend composition and molecular mass of the polymer, the effective lifetime furthermore strongly depends on the service environment of the material. The efficiency of the antioxidant stabilizer depends on its solubility in the material as well as its diffusion and migration characteristics which also determine the extent of loss due to extraction by surrounding media.

EXPERIMENTAL

MATERIALS

All materials were donated by manufacturers. The polypropylene (PP) tape yarn materials have been specially prepared for our research by Ciba Specialty Chemicals Inc. (Basel, Switzerland). The isotactic PP raw material, Profax 6501 (MONTELL, USA), was dry-blended with 0.05 % calcium stearate and 0.15 % IRGANOX B 561 (synergistic blend of IRGAFOS 168 (80%) and IRGANOX 1010 (AO I) (20%)) and extruded at 230°C into 50 or 100 μ m thick films. These melt-cast films were cooled in a water bath at 20°C and cut to tape yarns. Data of the three model materials used in this study are summarized in Table 1.

 Table 1. Stabilizer content of the pp type yarn material

	Manufactu	rer informatio	ICOT measurement		
	AO I [wt%]	AO II [wt%]	HAS [wt%]	AO's [wt%]	HAS [wt%]
Sample I	0.03	-	-	0.03	-
Sample II	0.03	0.4	-	0.37	-
Sample III	0.03	-	0.4	0.03	0.46

^{*} Initiated Cumene Oxidation Test

Sample I was prepared using the raw material (basic stabilizer AO I) without further stabilization. Sample II was additionally stabilized by the phenolic antioxidant IRGANOX 1330 (AO II) and sample III is additionally stabilized by the sterically hindered amine stabilizer CHIMASSORB 119 (HAS).

For the detailed assessment of oxidative durability (determination of lifetime parameter) presented in this paper a mechanically bonded nonwoven of isotactic polypropylene was used which contained a common stabilization for geosynthetic applications.

METHODS

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. Mostly 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 and testing of the residual antioxidant activity using HP-OIT and the ICOT method were employed. *Autoclave exposure*

The autoclave test for the evaluation of the oxidative durability of polymeric materials for geosynthetic applications - currently also established as index test in EN ISO 13438, method C (DIN EN ISO 13438 2005) - 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. Besides homogeneous temperature distribution this also ensures a safe operation during the test also for unknown materials which otherwise may exhibit uncontrolled chemical reactions under the oxygen pressures applied.

Standard conditions of the index test for oxidation resistance are defined in EN ISO 13438 as 80 °C and 5.1 MPa oxygen pressure. The test specimens are immersed in 0.01M NaHCO₃ aqueous solutions, the pH adjusted to 10 at 20°C.

In order to record the time-dependent degradation behavior usually all samples are inserted together at the beginning and batches of samples are consecutively removed according to their intended duration of exposure and later subjected to subsequent characterizations, e.g. tensile testing.

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 has to be carried out at three temperatures (60 $^{\circ}$ C, 70 $^{\circ}$ C and 80 $^{\circ}$ C) at 5.1 MPa oxygen pressure and additionally at 80 $^{\circ}$ C at 1.1 MPa and 2.1 MPa.

The autoclave exposure is performed in stirred stainless steel pressure-vessels (autoclaves). The total volume of the autoclave is about 7 litres with a headspace above the liquid phase of at least 20% gas phase. The temperature of the aqueous medium is measured separately using an immersed PT100 temperature sensor. A temperature constancy better than 0.5 °C is achieved. The pressure of the gaseous phase is measured using a piezo-resistive transducer and when equilibrium conditions after specimen installation or removal are reached the constancy is better than ± 5 kPa. Pressure and temperature inside the autoclave are continuously monitored and recorded at least every 10 minutes using an electronic chart recorder.

A schematic view of the autoclave apparatus is shown in Figure 1 (right image). At this moment our testing facility consists of 11 fully equipped autoclaves approved according to German pressure vessel regulations.



Figure 1. View of the autoclave testing facility (left) and a scheme of the autoclave (right)

Depending on the functional groups of the different types of antioxidants used in geosynthetics or on specific requirements or application conditions the composition of the aqueous medium may be adapted, e.g. with respect to pH or/ and metal ion content. It is possible to use different gas phases, e.g. N_2 , O_2 or synthetic air, with defined compositions and therefore a wide range of testing specifications are feasible.

The tape yarns (TY) were exposed in the autoclave wound on a cylindrical tube of about 6 cm diameter, made from fine stainless steel wire mesh. These materials have to be pre-shrunk for 24 hours in an water bath (thermostat) without external load at the intended exposure temperature prior to winding on the wire mesh tube which is performed with a preload of 0.5 N (Schroeder et al. 2002a).

Wovens and nonwovens are tested as strips, 50 mm wide and approximately 200 mm long. The essential tensile tests of the specimens and the specimen preparations are performed according to EN ISO 10319 (1996). According to our experience it is often favorable to remove fabric finishes by washing the woven/ nonwoven specimens in water or other appropriate solvents. The finishes are assumed to be present only temporarily in application. Therefore in our testing procedure the possible influence of the finish is avoided whenever possible.

Oven ageing

In order to compare autoclave tests with conventional oven testing, the materials were also exposed in ovens with forced air circulation in line with DIN 50011-2 at 110 °C. Temperature constancy was ± 1 K with respect to space and time, respectively, in the oven section used. The temperature was recorded at least every 10 minutes. Tape yarns were exposed as strands.

Tensile testing

Tensile testing of tape yarns was executed in line with DIN EN ISO 2062 (1995), usual gauge length: 100 mm, with a constant velocity of the drawing clamp of 50 mm/min.

Tensile testing of nonwovens was performed according to DIN EN ISO 10319: Gauge length: 200 mm; usual width of specimens: 50 mm; constant velocity of the drawing clamp of 100 mm/min.

All tensile test measurements were performed with a Zwick Model 1445 in an air conditioned environment at 20°C and a relative humidity of 65% in line with DIN EN ISO 139:2005 (2005).

ICOT (Initiated Cumene Oxidation Test)

The content of phenolic and HAS stabilizers in polypropylene can be determined by their action on the initiated cumene oxidation, a well-known model reaction (Schroeder et al. 2002b, Zeynalov et al. 2000). The reaction is initiated by 2,2-azobisisobutyronitrile (AIBN) at a temperature of 60° C ($\pm 0.02^{\circ}$ C) and monitored by recording the oxygen consumption volumetrically. The finely ground material under investigation is added to the cumene prior to the test in order to extract contained antioxidants. Phenolic stabilizers can be evaluated from the induction period, HAS stabilizers are determined from the steady state rate of oxygen consumption during cumene oxidation (Zeynalov & Allen 2006). Usually the result is expressed in weight by weight percent (% w/w) of a specific antioxidant (e.g. Irganox 1010 or Chimassorb 944). The method only is determining the amount of active functional groups of these both stabilizer classes and not the kind of stabilizer.

OIT (Oxidation Induction Time)

There are two different OIT test methods to determine the antioxidant stability of polypropylene or polyethylene based on differential scanning calorimetry (DSC): the so-called standard OIT (std-OIT) test in, e.g. line with ASTM D 3895 and the high pressure OIT test (HP-OIT, ASTM D 5885).. In this study only HP-OIT measurements are presented which allow a lower maximum testing temperature in order to also cover effects of HAS which are usually not detectable with std-OIT at 200 °C.

For the HP-OIT test about 10 mg materials are taken from the material specimens. The samples are heated from room temperature to 170 °C at a heating rate of 20 K/ min and then kept under isothermal conditions at that temperature, both under pure oxygen atmosphere at a pressure of 3.4 MPa with a continuous gas flow rate maintained at 20 ml/min. The OIT is then taken as the duration of the isothermal time segment to the onset of the exothermic oxidation peak.

RESULTS AND DISCUSSION

Polypropylene tape yarn

The polypropylene tape yarn stabilized by sterically hindered phenol (AO II) and sterically hindered amin (HAS) stabilizers, see Table 1, were aged in the autoclave under standard conditions in line with DIN EN ISO 13438, i.e. 80 °C and 5.1 MPa oxygen. For the evaluation of the oxidative durability based on the time-dependent degradation a failure criterion for the functionality of the geosynthetic product has to be defined. Here the retained tensile strength (R_F) is used. Two times to failure, $\tau(50\%)$ and $\tau(80\%)$ were selected which are obtained from the interpolated curve representing the results of the tensile tests.

Figure 2 (left image) shows the loss of mechanical properties (R_F) in dependence of the exposure duration of the accelerated ageing process. Phenolic stabilized samples display a dramatic degradation in mechanical performance after the end of an induction period (about 15 days for sample II), which depends on the stabilizer content, the sample geometry and the ageing temperature. With HAS a more gradual deterioration of the mechanical performance is observed. With respect to the screening test defined in EN ISO 13438, method C, both materials would meet the criterion of 14 or 28 days autoclave exposure with more than 50% retained tensile strength.

The course of the oxidative degradation shows the difference in the mechanism of antioxidant action between phenolic and HAS stabilized systems. During the oxidative ageing of polypropylene, phenolic stabilizers are able to effectively prevent the degradation completely for a certain time (induction period). As mentioned before the HAS stabilizers used here are not able to totally prevent but slow down the oxidative degradation of polypropylene. Therefore, the degradation starts early without distinct induction period. The HAS stabilizer used is very effective in decreasing the oxidation rate of the polyolefin based product for a long period.



Figure 2. The left image shows the retained tensile strength (RF) of sample I and II during oxidative degradation process in the autoclave at 80 °C and 5.1 MPa oxygen pressure. The graphical illustration of the right figure shows the retained tensile strength, stabilizer content and HP-OIT values of sample I in dependence of the duration of oxidation exposure.

Depending on the failure criterion, HAS stabilizers may even outperform phenolic antioxidants, as illustrated in Figure 2. After 50 days of exposure at autoclave conditions of 80 °C and 5.1 MPa oxygen pressure the HAS stabilized sample III (0.4% HAS) still exhibits 80% retained tensile strength, whereas the phenolic stabilized sample II (0.4% AO II) has already failed completely.

ICOT and OIT measurements can be used to evaluate the degree of degradation (consumption) and/or extraction of stabilizers from the polymer. The right image of Figure 2 shows the retained stabilizer content (ICOT) and the HP-OIT values in dependence of the time of exposure. It is clearly shown that during the induction period up to the 14th day the tensile properties of the phenolic stabilized samples are not changed during the autoclave exposure but the stabilizer content and OIT values are drastically decreased. Down to a critical value of stabilizer content of around 10 wt.-% the tensile properties are not yet affected. When the stabilizer concentration is further reduced a rapid loss of tensile strength is observed.

Hence it is also possible to estimate the lifetime expectancy of polypropylene materials by determining the state of antioxidant stabilizers e.g. by ICOT-determination of the actual effective stabilizer content. For that it is required to establish a reliable correlation between mechanical deterioration and antioxidant depletion in addition to a trusted determination of the residual content of the antioxidant actually used. In order to meet these prerequisites a comprehensive pre-examination might be necessary. Furthermore the complementary use of these methods reveal important information about the antioxidant characteristics as well as the degradation behavior which would not be accessible using tensile testing alone.

Table 2. The τ	$_{[80\%]}$ - and $\tau_{[50\%]}$ -	value of the retained	tensile strength R _F resulting	from the autoclave tes	t and the oven test
	100/01 [20/0]		- U		

Sample	Autoclave test: 80 °C and 5.1 MPa O ₂ retained tensile strength R _F [%]		Oven test: 110 °C retained tensile strength R _F [%]		
	τ _[80%] /d	τ _[50%] /d	τ _[80%] /d	τ _[50%] /d	
Sample I	29.7	38.2	57.1	66.0	
Sample II	47.6	114.2	233.4	351.0	

Comparison of results of the autoclave test and the oven test are summarized in Table 2. With the autoclave test significantly shorter exposure times are observed. Therefore at this stage, i.e. considering the time-dependent degradation by oxidation in the autoclave at one single test condition (e.g. 80 °C 5.1 MPa oxygen pressure), the method allows an efficient and differentiated comparative evaluation of different materials with respect to oxidative durability in view of the specific conditions chosen for the test.

Extrapolation and determination of a lifetime parameter for a geosynthetic product

The results of the autoclave tests at three temperatures (60, 70 and 80 $^{\circ}$ C) and three pressures (10, 20 and 5.1 MPa) can be used for an estimate of the expected lifetime of a geosynthetic product resulting from oxidative durability investigations based on extrapolation employing a modified Arrhenius equation (see eq. (2)). Our approach is based on the determination of a characteristic lifetime parameter which itself allows a comparison of different materials or products (ranking) and can furthermore be rated considering specific conditions or requirements with respect to an estimated lifetime of the respective material.

If only temperature is the accelerating parameter the well known Arrhenius equation can be used to describe the dependence of the time to failure τ on the temperature (Koerner et al. 1992, Wise et al. 1995).

$$\frac{1}{\tau} = A \exp\left(\frac{-E_A}{RT}\right) \quad \text{or} \quad \ln\frac{1}{\tau} = \ln A - \frac{E_A}{RT} \tag{1}$$

With τ being the time to failure resulting from the tensile test ($\tau_{[50\%]}$ and $\tau_{[80\%]}$) regarding a corresponding failure criterion (50% or 80% retained tensile strength) at a temperature T, E_A is the apparent activation energy, R the universal gas constant and A the pre-exponential factor. As long as the Arrhenius relation is reasonably applicable (which may not be the case for a broader temperature range) an extrapolation to service temperature would then yield the corresponding time to failure, i.e. the expected lifetime under these conditions. Deviations from the strict Arrhenius-behavior lead to a more or less significant uncertainty of the extrapolation result.

As in our testing method an additional influencing factor, the oxygen pressure, is used for acceleration, the Arrhenius equation has to be modified in order to describe the data points τ which now depend on both, temperature and pressure. For that a straightforward procedure is not feasible due to the complex mechanism of the oxidation chain reactions. On this account the following empirical approach is used as shown in equation (2):

$$\frac{1}{\tau} = A \cdot \exp\left\{\frac{(B + C\rho)}{T}\right\}$$
(2)

p denotes the pressure, parameters A and B correspond to the pre-exponential factor and activation energy as in eq. (1). and C is the additional parameter introducing the pressure dependence.

$$\frac{1}{\tau} = A \cdot \exp\left\{\frac{(B)}{T}\right\} \cdot \frac{p}{1 + Dp}$$
(2a)

$$\frac{1}{\tau} = A \cdot \exp\left\{\frac{(B + C\rho)}{T}\right\} \cdot \frac{\rho}{1 + D\rho}$$
(2b)

Alternatively also equations (2a) and (2b) can be employed to describe the $\tau(T,p)$ data but based on our present experience equation (2) yield the most robust and reasonable results when used for extrapolation (Schroeder et al. 2008) and is therefore used for evaluation here. The model using p as an exponential term (equation (2)) is a first approximation, which accounts mainly for energetic effects of pressure involved in a rate determining step of the oxidation reactions. This model is described by three parameters in contrast to the usual Arrhenius equation. Hence, we expected that the pressure dependence could be sufficiently and conservatively represented by this simple model.

For the fitting procedure, the variables were transformed as follows:

$$x = \frac{1}{T}, y = p, z = \ln\left(\frac{1}{\tau}\right)$$
(3)

Equation (4) is resulting from equation (2) and (3):

$$z = A + Bx + Cxy \tag{4}$$

Using equation (4) fit calculations were performed with TableCurve 3D V4.0 (Systat Software, Inc., Richmond, USA). The surface plots τ^{-1} (p, T) as well as extrapolated values were calculated using the obtained values of parameters A, B and C.

For further evaluation and comparison a specified extrapolated value Θ should be considered ("lifetime parameter"). A value resulting from a limited extend of extrapolation should be preferred for that, e.g. from about 20 K extrapolation, but requires a scheme including some agreed conventions to derive expected lifetime estimates for distinctly lower temperatures (e.g. by defining an activation energy E_A for lower temperatures and application specific surroundings). A simpler but by far more uncertain approach is to directly extrapolate the resulting parameter surface to service conditions. For comparison both approaches are presented here.

For a first error estimate of this procedure we assume here an error of 5% for the individually determined τ values. As their determination depends on the course of the reduction of mech. strength (i.e. the material and stabilizer characteristics) with increasing exposure in the autoclave and is complicated by the limited number of data points available this is a reasonable range according to our experience up to now. Various combinations of the modified data points are then subjected to the same fit procedure as the original points and the mean value of the maximum are taken as an error estimate for the lifetime parameter.

Figure 3 shows the 3D-fitting curve of the modified Arrhenius model representing the test result for the investigated geosynthetic nonwoven. The basis for the calculation of the lifetime parameter is the exposure time for 50% retained tensile strength ($\tau_{[50\%]}$) of the material which was determined from the time dependent curves obtained for five different autoclave test conditions. The fitting parameters obtained by regression method using TableCurve 3D software.

For this material at application conditions (25 °C and 0.21 bar O₂) a lifetime parameter of $\Theta_{[50\%]}^{298K} = 155$ years (+/-64 years) is determined and an apparent activation energy of 104 kJ/mol is calculated from equation (5) (with R=8.3145J/Kmol).

$$B = E_A / R \tag{5}$$

For 40°C and 0.21 bar oxygen pressure a lifetime parameter $\Theta_{[50\%]}^{313K} = \text{of about 21 years (+/- 6 years) is obtained.}$

From our experiences obtained so far it can be stated, that the presented method offers a new possibility to assess a lifetime parameter of polyolefin materials in regard to their sensitivity to oxidation. The lifetime found with the modified Arrhenius model for the stabilized geosynthetic product seems to be plausible, promising a good alternative method to the conventional oven test.



Figure 3. 3D-extrapolation with the obtained a lifetime parameters at 25 °C as well as 40 °C and 0.21 bar oxygen pressure of a normally stabilized geosynthetic nonwoven

CONCLUSION

With the autoclave test a new method for the assessment of the oxidative durability of polyolefin based products is presented. For typical accelerated ageing procedures at moderately elevated temperatures up to 80 °C relatively short test durations, usually not exceeding 12 month can be realized. In addition to oxidative influences also the extraction of antioxidants as well as effects of aqueous surroundings can be taken into account and the corresponding conditions (e.g. pH) can be adapted according to specific requirements.

Based on multiple autoclave exposures under different conditions (temperature and oxygen pressure) a possible approach for the determination of lifetime parameters is proposed which may be used in order to get estimates for expected service lifetimes under application conditions. It should be noted that therefore a comprehensive evaluation scheme should be established which accounts for uncertainties in the methodology and also includes practical experiences from other test as well as from service on site.

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