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ARTICLE

Effect of Acids and Alkalis on the Resistance of a Polypropylene Geotextile Against Thermo-oxidation

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ABSTRACT

The long-term behaviour of geosynthetics is one of the most important topics in the research about these materials. This work studies the effect of some liquids (water, sulphuric acid 0.1 mol.L^{-1} and sodium hydroxide 0.1 mol.L^{-1}) on the resistance of a polypropylene geotextile against thermo-oxidation. For that purpose, the geotextile was (1) exposed in isolation to the liquids (immersion tests) and to thermo-oxidation (oven-ageing tests) and (2) exposed consec-utively to both degradation tests (combined effect). The damage suffered by the geotextile in the degradation tests was evaluated by monitoring changes in its tensile behaviour. Based on the changes occurred in tensile strength, reduction factors were determined. The reduction fac-tors obtained in the successive exposures to liquids and thermo-oxidation were compared with the reduction factors determined by the traditional methodology for the combined effect of those agents. The results, among other findings, showed the existence of an effect of sulphuric acid 0.1 mol.L^{-1} on the resistance of the geotextile against thermo-oxidation. Indeed, the suc-cessive exposure to sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation (two agents that individ-ually did not cause relevant damage) led to some degradation. Due to the interaction occurred between the degradation agents, the traditional methodology was unable to predict correctly (by underestimating) the reduction factor for the combined effect of sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation.

1. Introduction

Geosynthetics are polymeric materials used in the construction of many civil engineering structures, such as waste landfills, roads, railways, hydraulic structures or coastal protection structures. These materials provide an excellent alternative to more traditional construction materials due to their high efficiency, low cost, ease of installation and low environmental impact. There are many types of geosynthetics, being the geotextiles the most used ones due to their ability to perform many different functions: filtration, drainage, protection, separation or reinforcement.

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For being suitable for use in civil engineering structures, the geosynthetics must have a good resistance against many degradation agents. The most common degradation agents of these materials include: liquids (like acids or alkalis), high temperatures, oxygen, ultraviolet (UV) radiation and other weathering agents, creep and abrasion[1,2]. The installation process can also provoke damage to the geosynthetics[3].

The polymers most used for the production of geosynthetics include polyolefins (like polypropylene (PP) or polyethylene), polyesters and polyamides. PP (polymer most used for producing geotextiles) has a good resistance against many chemical substances, like acids or alkalis[4,5]. However, it has a relatively poor resistance against oxidation[6,7]. The oxidation process of PP follows a complex chain reaction mechanism that can be induced by UV radiation (photo-oxidation) or by temperature (thermo-oxidation) (detailed description of the oxidation process in[6,8,9]). The damage caused by photo and thermo-oxidation can be retarded by adding chemical additives (such as antioxidants and/or UV stabilisers) to PP[9][10][11]. In the absence of UV radiation, the oxidation process of PP is relatively slow at ambient temperatures[12]. However, it cannot be neglected when considering products for long-term use, like geosynthetics.

The long-term behaviour of geosynthetics is normally predicted based on data obtained from laboratory tests where the materials are exposed (often under accelerated conditions) to degradation agents[13][14][15]. For that purpose, the organisations for standardisation (like the European Committee for Standardization or the American Society for Testing and Materials) have developed many methods. For example, the resistance of geosynthetics to liquids can be evaluated by EN 14030[16], EN 12447[17] or ASTM D6389[18]. EN ISO 13438[19] and ASTM D5721[20] can be used to evaluate their resistance against thermo-oxidation. The resistance of geosynthetics against degradation can also be evaluated by field tests (degradation under real conditions)[21,22,23]. However, these tests are often very time consuming (months or years), being unsuitable when quick results are needed.

The damage that occurs in geosynthetics during the degradation tests is often evaluated by monitoring changes in their mechanical properties (mainly in their tensile behaviour). In order to take into account the resistance changes that geosynthetics suffer over time, reduction factors (RF) are often used in the design phase[24,25]. For example, for reinforcement applications, the tensile strength of geosynthetics is typically affected by a set of reduction factors accounting for the effects of installation damage, creep, weathering and chemical and biological agents[24,25].

The actual design methods, the standard degradation tests for durability evaluation and most studies found in literature about the durability of geosynthetics consider the isolated action of the degradation agents, not accounting for possible interactions between them[26]. However, the combined effect of the degradation agents (which happens in real cases) can be much different (more severe) from the sum of their isolated actions. For example, Carneiro et al. (2014)[15] and Carneiro et al. (2018)[26] showed the existence of interactions between chemical degradation agents of geosynthetics. The occurrence of interactions between mechanical degradation agents has also been reported in literature[27,28]. The occurrence of the previous interactions can lead to inaccurate global reduction factors, which are traditionally obtained by multiplying relevant partial reduction factors (each determined in isolation)[26]. Therefore, the identification and quantification of interactions between the different degradation agents may lead to a better prediction of the long-term behaviour of the geosynthetics.

This work studies the effect of some liquids in the thermo-oxidation process of a PP geotextile. For that purpose, the geotextile was (1) immersed on liquids (water, sulphuric acid 0.1 mol.L^{-1} and sodium hydroxide 0.1 mol. L^{-1}), (2) exposed to thermo-oxidation and (3) immersed in liquids followed by thermo-oxidation (combined ac-tion). The main goals of the work included: (1) determi-nation of the effect of the degradation tests on the tensile behaviour of the geotextile, (2) evaluation of the effect of the immersion tests on the resistance of the geotextile against thermo-oxidation (identifying possible interactions between the degradation agents) and (3) comparison of the reduction factors obtained by the traditional methodology for the combined effect of liquids and thermo-oxidation (determination of the reduction factors in isolation for each degradation agent and further multiplication) with those obtained in the successive exposure to those agents.

2. Experimental Description

2.1 Geotextile

This work studied a nonwoven needle-punched PP geotextile stabilised with 0.2% (percentage in weight) of the additive Chimassorb 944 (C944). C944 is a UV stabiliser belonging to the HALS (hindered amine light stabilisers) family. Besides acting as UV stabiliser, C944 is also highly effective in protecting PP geotextiles against thermo-oxidation[12]. The main properties of the geotextile can be found in Table 1.

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Table 1. Main Properties of the Geotextile (Undamaged Sample)

Property	Test standard	Mean value
Mass per unit area (g.m^{-2})	EN ISO 9864[29]	272 (±15)**
Thickness at 2 kPa (mm)	EN ISO 9863-1[30]	3.16 (±0.10)**
Tensile strength* (kN.m^{-1})	EN ISO 10319[31]	13.12 (±0.55)**
Elongation at maximum load* (%)	EN ISO 10319[31]	112.5 (±4.3)**

*determined in the machine direction of production

**95% confidence intervals in brackets

The sampling process (for the characterisation and degradation tests) was carried out according to EN ISO 9862[32]. The specimens (machine direction of production) were collected from positions evenly distributed over the full width and length of the geotextile (supplied in a roll), but not closer than 100 mm to the edges. The specimens for the same characterisation or degradation test (total of 5 specimens for each test) were taken from different longitudinal and transverse positions of the roll. The specimens were 200 mm wide and 300 mm long.

2.2 Degradation Tests

First, the geotextile was exposed in isolation (single exposure) to immersion in liquids and thermo-oxidation (description of the tests in the following points). Then, the geotextile was exposed consecutively to the action of liquids and thermo-oxidation (multiple exposures). Table 2 summarizes the degradation tests carried out in this work.

Table 2. Degradation Tests.

Single exposure	Multiple exposure
H_2O	H_2O + TO (28 days)
H_2SO_4	H_2O + TO (56 days)
NaOH	H_2SO_4 + TO (28 days)
TO (28 days)	H_2SO_4 + TO (56 days)
TO (56 days)	NaOH + TO (28 days)
	NaOH + TO (56 days)

(TO – thermo-oxidation)

2.2.1 Immersion Tests

The geotextile was immersed at room temperature (about 20 ºC) in deionised water (H_2O) (pH ≈ 7), in sulphuric acid (H_2SO_4) 0.1 mol.L^{-1} (pH ≈ 1) and in sodium hydroxide (NaOH) 0.1 mol.L^{-1} (pH ≈ 13). These conditions were chosen to represent strong acid and alkaline environments (the neutral medium was intended for comparison). The immersion tests were carried out in the dark and had a duration of 150 days. Sulphuric acid (p.a. grade) and sodium hydroxide (p.a. grade) were obtained from Merck. Water was treated microbiologically and purified by reverse osmosis followed by deionisation on ionic exchange columns.

The specimens immersed in sulphuric acid 0.1 moL.L^{-1} and sodium hydroxide 0.1 mol.L^{-1} that were subsequently exposed to thermo-oxidation (multiple exposures) were not washed after the immersion tests (in order to be contaminated with remains of the acid or alkali) and were dried at room temperature in the absence of light. The subsequent thermo-oxidation tests were carried out in a short period of time (a few days or weeks) after the drying process.

2.2.2 Thermo-oxidation Tests

The thermo-oxidation tests were carried out in an oven (*Heraeus Instruments*, model T6120) and consisted in exposing the geotextile at 110 ºC in a normal oxygen atmosphere (21% O_2) without forced air circulation. The exposure period lasted for 28 and 56 days. The exposure period of 28 days corresponds to method A2 of EN ISO 13438[19]. The increase of the exposure time from 28 to 56 days was intended to harshen the degradation conditions and thereby enhance the effects of thermo-oxidation.

2.3 Evaluation of the Damage Suffered by the Geotextile

The damage suffered by the geotextile during the degradation tests was evaluated by monitoring changes in its tensile behaviour (tensile tests according to EN ISO 10319[31]). The tensile tests (velocity of 20 mm.min^{-1}) were carried out in an equipment from *Lloyd Instruments* (model LR 50K) equipped with a load cell of 5 kN (from *Lloyd Instruments*). Each tensile test included the analysis of 5 specimens (in the machine direction of production) with a length of 100 mm (between grips) and a width of 200 mm.

The mechanical parameters obtained in the tensile tests included tensile strength (T, in kN.m^{-1}) and elongation at maximum load (E_{ML}, in %). Elongation was determined by expressing the relative displacement of the grips as a percentage of the original length (100 mm). The results obtained for tensile strength and elongation at maximum load (mean values of 5 specimens) are presented with 95% confidence intervals determined according to Montgomery and Runger[33] (Equation 1).

$$\mu = x \pm t_{\alpha/2,n-1}\frac{s}{\sqrt{n}} \tag{1}$$

Where μ is the population mean, x is the sample mean, t is Student's t-distribution value for the confidence level α and n-1 degrees of freedom, n is the number of specimens

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tested for each sample and *s* is the sample standard deviation. The changes occurred in tensile strength are also presented in terms of retained tensile strength ($T_{Residual}$, in %), obtained by dividing the tensile strength of the damaged samples ($T_{Damaged}$) by the tensile strength of an undamaged sample ($T_{Undamaged}$) (Equation 2).

$$T_{Residual} = \frac{T_{Damaged}}{T_{Undamaged}} \times 100 \quad (2)$$

2.4 Determination of Reduction Factors

The reduction factors for the effects of immersion in liquids (RF_L), thermo-oxidation (RF_{TO}) and immersion in liq-uids followed by thermo-oxidation (RF_{L+TO}) were obtained by the following equation:

$$RF = \frac{T_{Undamaged}}{T_{Damaged}} \quad (3)$$

Where, $T_{Undamaged}$ and $T_{Damaged}$ represent, respectively, the tensile strength of the geotextile before and after the degradation tests. The reduction factors obtained by the traditional methodology for the combined effect of immersion in liquids and thermo-oxidation ($RF_{L+TO\ Trad}$) were determined by multiplying the reduction factors obtained in isolation for each degradation agent (RF_L and RF_{TO}, re-spectively) (Equation 4).

$$RF_{L+TO\ Trad} = RF_L \text{ x } RF_{TO} \quad (4)$$

The reduction factors presented in this work correspond to particular degradation conditions and cannot be generalized or applied directly in the design. For being used in the design, the reduction factors must be analysed case by case, taking into consideration the particular conditions of each construction.

3. Results and Discussion

3.1 Single Exposures to Liquids and Thermo-oxidation

The tensile properties of the geotextile had no relevant changes after the immersion tests in water, in sulphuric acid 0.1 mol.L^{-1} and in sodium hydroxide 0.1 mol.L^{-1} (Table 3), which is in accordance with the good resistance reported in literature for PP geotextiles against liquids. The minor variations observed in tensile strength and elongation at maximum load can be attributed to the heterogeneity of the geotextile (nonwoven geotextiles typically have some heterogeneity arising from their manufacturing process).

Table 3. Tensile Properties of the Geotextile After the Single Exposures to Liquids and Thermo-oxidation.

Degradation test	T (kN.m^{-1})	E_{ML} (%)	$T_{Residual}$ (%)	RF
H_2O	13.06 (±1.44)	125.6 (±10.3)	99.5	1.00
H_2SO_4	13.10 (±1.60)	110.4 (±14.3)	99.8	1.00
NaOH	13.74 (±1.71)	122.6 (±12.0)	104.7	1.00
TO (28 days)	12.64 (±1.24)	89.2 (±4.7)	96.3	1.04
TO (56 days)	12.76 (±1.82)	83.4 (±4.1)	97.3	1.03

(95% confidence intervals in brackets)

Similarly to the immersion tests, the thermo-oxidation tests also did not cause relevant changes in the tensile strength of the geotextile (retained tensile strengths very close to 100%) (Table 3). However, they caused a reduction in elongation at maximum load (decrease from 112.5% to 89.2% and to 83.4% after, respectively, 28 and 56 days of thermo-oxidation). The decreases in elongation at maximum load may be related with the occurrence of some shrinkage (about 2.5%) in the geotextile during the exposure to thermo-oxidation, which led to a reduction of the deformability of the nonwoven structure.

3.2 Multiple Exposures to Liquids and Thermo-oxidation

The specimens immersed in sulphuric acid 0.1 mol.L^{-1} (originally white) acquired a brown colour during the exposure to thermo-oxidation (Figure 1), which readily indicated the occurrence of some damage in the geotextile. Besides the colour change, no other changes were detected (by the naked eye) in the geotextile. By contrast, the colour of the specimens immersed in water and in sodium hydroxide 0.1 mol.L^{-1} (white in both cases) remained practically unaltered during the thermo-oxidation tests.

Figure 1. Geotextile Immersed in Sulphuric Acid 0.1 mol.L^{-1} Before (Left) and After Thermo-oxidation (Right).

The tensile behaviour of the geotextile after the thermo-oxidation tests was similar for the specimens previously immersed in water (multiple exposure) and for the specimens without immersion (single exposure to

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thermo-oxidation) (Table 4). Indeed, no relevant differences were found in tensile strength and in elongation at maximum load when comparing the single exposure to thermo-oxidation with the multiple exposure to water and thermo-oxidation. Therefore, the immersion in water (during 150 days at about 20 ºC) had no effect on the resis-tance of the geotextile against thermo-oxidation.

Table 4. Tensile Properties of the Geotextile After the Multiple Exposures to Liquids and Thermo-oxidation.

Degradation test	T (kN.m^{-1})	E_{ML} (%)	$T_{Residual}$ (%)	RF
H_2O + TO (28 days)	13.00 (±2.30)	85.7 (±6.2)	99.1	1.01
H_2O + TO (56 days)	13.04 (±1.63)	82.4 (±4.3)	99.4	1.01
H_2SO_4 + TO (28 days)	11.16 (±0.91)	71.2 (±6.6)	85.1	1.18
H_2SO_4 + TO (56 days)	10.20 (±1.99)	62.7 (±7.6)	77.7	1.29
NaOH + TO (28 days)	12.51 (±1.73)	84.6 (±2.5)	95.4	1.05
NaOH + TO (56 days)	12.28 (±1.19)	89.2 (±6.0)	93.6	1.07

(95% confidence intervals in brackets)

Contrarily to the immersion in water, the immersion in sulphuric acid 0.1 mol.L^{-1} led to a reduction in the resistance of the geotextile against thermo-oxidation (the occurrence of some damage had already been indicated by the colour change). Indeed, the specimens immersed in sulphuric acid 0.1 mol.L^{-1} had a retained tensile strength of 85.1% after 28 days of thermo-oxidation (Table 4). The increase of the exposure time led to a further decrease in tensile strength (retained tensile strength of 77.7% after 56 days of thermo-oxidation). Elongation at maximum load (after the thermo-oxidation tests) was also lower for the specimens immersed in sulphuric acid 0.1 mol.L^{-1} (multiple exposure) than for the specimens without immersion (single exposure to thermo-oxidation). This way, two degradation agents that individually did not cause relevant damage (retained tensile strengths between 96.3% and 99.8% in the single exposures), together led to some degradation in the geotextile.

The reduction in the resistance of the geotextile against thermo-oxidation after the immersion in sulphuric acid 0.1 mol.L^{-1} may have some possible explanations: (1) the remains of sulphuric acid catalysed the thermo-oxidation process (the specimens were not washed after the immersion test in order to be contaminated with remains of sulphuric acid), (2) occurrence of losses and/or consumption of the HALS-type UV stabiliser during the immersion in sulphuric acid 0.1 mol.L^{-1}, leaving the geotextile less protected against thermo-oxidation (in the absence of the additive, the geotextile would be totally destroyed after 9 days at 110 ºC[12]) or (3) the protective mechanism of the HALS-type UV stabiliser was affected by the remains of sulphuric acid. Further studies are needed to undoubtedly explain the influence of sulphuric acid in the thermo-oxidation process. These studies are mainly related with polymer chemistry, falling outside the area of civil engineering.

The multiple exposure to sodium hydroxide 0.1 mol.L^{-1} and thermo-oxidation caused a slight reduction in the tensile strength of the geotextile (retained tensile strengths of 95.4% and 93.6% after 28 and 56 days of thermo-oxidation) (Table 4). However, and having into account the 95% confidence intervals, it is not possible to conclude if these small decreases are due to some effect of sodium hydroxide on the thermo-oxidation process or if they only reflect the typical heterogeneity of non-woven geotextiles. Regarding elongation at maximum load, no relevant differences were found when comparing the multiple exposure to sodium hydroxide 0.1 mol.L^{-1} and thermo-oxidation with the single exposure to ther-mo-oxidation. Although not evident in the experimental conditions used in this work, the existence of an effect of sodium hydroxide on the thermo-oxidation process was identified (under different experimental conditions) by Carneiro et al. (2014)[15].

3.3 Comparison of Reduction Factors: Multiple Exposure vs. Traditional Methodology

The reduction factors obtained in the multiple exposures to liquids (water, sulphuric acid 0.1 mol.L^{-1} or sodium hydroxide 0.1 mol.L^{-1}) and thermo-oxidation were compared with the reduction factors determined by the traditional methodology (Equation 4) for the combined effect of both degradation agents (determination of reduction factors in isolation for each degradation agent and further multiplication). The reduction factors obtained directly from the single and multiple exposures to the degradation agents can be found in Tables 3 and 4, respectively.

The reduction factors determined by the traditional methodology for the combined effect of (1) immersion in water and thermo-oxidation and (2) immersion in sodium hydroxide 0.1 mol.L^{-1} and thermo-oxidation were not much different from those obtained in the multiple exposures to both degradation agents (Table 5). By contrast, the multiple exposure to sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation led to slightly higher reduction factors (1.18 and 1.29 after, respectively, 28 and 56 days of thermo-oxidation) than those predicted by the traditional methodology for the combined effect of both agents (1.04 and 1.03 after, respectively, 28 and 56 days of thermo-oxidation) (Table 5).

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Table 5. Reduction Factors for the Combined Effect of Liquids and Thermo-oxidation Obtained by the Traditional Methodology ($RF_{L+TO\ Trad}$) and in the Multiple Exposures to the Degradation Agents (RF_{L+TO}).

Multiple exposure	RF_{L+TO}	$RF_{L+TO\ Trad}$
H_2O + TO (28 days)	1.01	1.04
H_2O + TO (56 days)	1.01	1.03
H_2SO_4 + TO (28 days)	1.18	1.04
H_2SO_4 + TO (56 days)	1.29	1.03
NaOH + TO (28 days)	1.05	1.04
NaOH + TO (56 days)	1.07	1.03

The differences found between the reduction factors for the combined effect of sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation (obtained by the traditional methodology or in the multiple exposure to both degradation agents) can be attributed to the interaction that occurred between the degradation agents (which the traditional methodology was unable to account for). This way, when interactions occur between the degradation agents, the multiplication of reduction factors (each representing the isolated effect of a degradation agent) may not represent correctly (by underestimating) the combined effect of those agents. Other examples of interactions between degradation agents of geosynthetics (which led to inaccurate reduction factors when using the traditional methodology) can be found in Carneiro et al. (2014)[15], Dias et al. (2017)[28] and Carneiro et al. (2018)[26].

4. Conclusion

The isolated exposures to liquids (water, sulphuric acid 0.1 mol.L^{-1} or sodium hydroxide 0.1 mol.L^{-1}) and to thermo-oxidation did not cause relevant changes in the tensile strength of a PP geotextile (retained tensile strengths very close to 100%). By contrast, elongation at maximum load suffered a reduction after thermo-oxidation (no relevant changes occurred after the immersion tests). Globally, the geotextile presented a good resistance against the immersion tests and against thermo-oxidation.

The damage occurred in the geotextile in the multiple exposures to liquids and thermo-oxidation was not always equal to the sum of the damage caused by each agent individually. Indeed, the multiple exposure to sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation (two agents that individually did not cause relevant damage), led to some degradation. Under similar experimental conditions, the effect of sulphuric acid 0.1 mol.L^{-1} (pH≈ 1) on the resistance of the geotextile against thermo-oxidation was higher than the effect of sodium hydroxide 0.1 mol.L^{-1} (pH ≈ 13). However, under different experimental conditions (higher immersion temperature), the effect of sodium hydroxide 0.1 mol.L^{-1} can be higher than the effect of sulphuric acid 0.1 mol.L^{-1} [15].

The identification and quantification of interactions between the degradation agents is crucial to understand and predict the behaviour of geosynthetics under real conditions (where the agents do not act in isolation). In the existence of relevant interactions between the degradation agents, the reduction factors obtained directly from the multiple exposures may be different from those determined by the traditional methodology (determination of reduction factors in isolation for each degradation agent and further multiplication). Indeed, the traditional methodology was unable to predict with accuracy (by underestimating) the reduction factor for the combined effect of sulphuric acid 0.1 mol.L^{-1} and thermo-oxidation. The definition of more reliable reduction factors (taking into consideration the interactions that may occur between the degradation agents) may contribute for a better design and thereby allow a better application of geosynthetics in Civil Engineering.

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Abbreviations and Symbols

α – Confidence level
E_{ML} – Elongation at maximum load
HALS – Hindered amine light stabiliser
n – Number of specimens
PP – Polypropylene
RF – Reduction factor
RF_L – Reduction factor for the action of liquids
RF_{TO} – Reduction factor for thermo-oxidation
RF_{L+TO} – Reduction factor for the combined effect of liquids and thermo-oxidation
$RF_{L+TO\ Trad}$ – Reduction factor for the combined effect of

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liquids and thermo-oxidation (traditional methodology)
s – Sample standard deviation
t – Student's t-distribution value
T – Tensile strength
$T_{Damaged}$ – Tensile strength of exposed samples
$T_{Residual}$ – Retained tensile strength
$T_{Undamaged}$ – Tensile strength of undamaged sample (unexposed)
TO – Thermo-oxidation
μ – Population mean
UV – Ultraviolet
x – Sample mean

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