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Comparison of the Rates of Polypropylene Fibre Degradation Caused by Artificial Light and Sunlight

Abstract

The rates of degradation of two types of polypropylene (PP) fibres caused by sunlight and xenon tube light were assessed. Changes in the tensile strength and elongation at break of the fibres as well as changes in the fibre surface topography due to their irradiation with the same doses of UV radiation of both types of light were compared. The tensile strength of PP fibres exposed to xenon tube light decreases four up to six times faster than that of PP fibres exposed to sunlight during the summer season. The elongation at break of these fibres decreases about four times faster. The light emitted by an xenon tube causes considerably greater damage to the fibre surface in the form of crosswise cracks, although there are fewer of which than in the case of sunlight.

Key words: polypropylene fibre, photo-degradation, UV, sunlight.

Introduction

Polypropylene, as a saturated hydrocarbon, should not absorb light with a wavelength above 180 nm, being considerably lower than the limiting wavelength - 290 nm, below which the earth's atmosphere completely absorbs UV light; thus PP fibre should be resistant to the action of sunlight. However, PP fibres contain a certain quantity of chromophoric groups formed during polymerisation and spinning due to thermal oxidation, which cause very slow photo-oxidation by absorbing light with a wavelength higher than 290 nm. Under the influence of sunlight, carboxyl and hydroxyl groups are formed in isotactic PP, and as a result of this PP fibres undergo further degradation [1]. The degradation in the fibre surface zone (up to 1 µm) proceeds within 100 - 150 h of xenon tube irradiation. Further exposure (>200 h) causes the oxidation of the inside part of the fibre [2]. PP fibres behave similarly in sunlight. Based on the microscopic observation of PP fabrics taken from motor-car seats after 5 years' exploitation, Barish [3] found cracks in fibres reaching their core.

The resistance of PP fibres to the action of light oscillates within very wide limits, depending on the polymer manufacturing conditions and agents added to the polymer, such as the stabiliser counteracting thermo- and photo-oxidation, and pigments [4 - 6]. The resistance of PP fibres to the action of UV radiation is usually assessed on the basis of changes in fibre tensile strength. The damage to fibre surfaces has also been analysed [3] as well as chemical changes in the poly-

mer resulting in the formation of carboxyl and hydroxyl groups. To this end one can also determine the amount of CO₂ emitted during the irradiation of PP films [7, 8]. In most cases, the resistance of PP fibres to light is examined with the use of the artificial light of a xenon tube, whose spectrum is similar to that of sunlight when using a system of filters. The merit of this way is the stability and reproducibility of irradiation conditions. Another approach to this issue is the examination of PP photo-degradation under natural conditions, where the progress of photolytic degradation is assessed versus the time of exposure to sunlight. In a temperate climate, sunlight irradiance is variable, and not only during the year but also particular years show different periods of insolation. A major study on the photodegradation of PP fibres is presented by Rabek [9] in his book. In the present study, the effects of the UV irradiation of a xenon tube and sunlight within the range of UV and visible spectra on PP fibres were compared using tensile strength tests and the microscopic assessment of changes in the PP fibre surface. In order to correlate the effects of both types of light, the fibre degradation rates were compared depending on the dose of incident irradiation.

The practical aim of the study was to correlate the irradiation of an xenon lamp and sunlight in the summer with the de-

gree of degradation of polypropylene fibres caused by the irradiation. The aim was the further testing of modified fibre photodegradation activators

Test materials

To examine the effect of sunlight and xenon tube light on the tensile strength of PP fibres, two types of polypropylene continuous fibres were produced from the following polymers: Daplen RT 581 [10] from Borealis, with MFI = 35 g/10 min (PP fibre ,,1") and Moplen HP462R1) from Basel Orlen, with MFI = 25 g/10 min, containing a 3%addition of Vibatan PP Biodegradable 03251²⁾ from Viba S.p.A (PP fibre "2"). The Polymers were selected for our tests as an example of polymers often used for the production of PP fibres. The fibres were spun by means of an extruder spinning machine at a temperature of (230 ÷ 240) °C, through a 17 orifice spinneret, with a yield of $(8.1 \div 11.9)$ g/min, at a rate of 600 m/min. A spinning preparation - Fasavin 2830 from Zschimmer and Schwarz, was applied on the fibres in the form of a 15% aqueous solution. The fibres were drawn on a J6/2 draw winder from Rieter at a rate of 267 m/min, with a draw ratio of 3.2 on a heated godet at a temperature of 65 °C.

Characteristics of the PP fibres used for testing are presented in *Table 1*.

Table 1. Physical properties of PP fibres used for testing; CV - variation coefficient.

Properties	Fibre PP "1 "	Fibre PP "2"
Mean value of linear density, dtex	97.3, CV = 3%	66.5, CV = 4%
Tenacity, cN/tex	36.3	34.0
Mean value of breaking elongation, %	99.0, CV = 7%	72.0, CV = 8%
Mean value of fibre diameter, µm	26.3, CV = 9%	21.7, CV = 10%

Experimental methods

To expose fibre samples to sunlight, a test stand was designed and produced at the Textile Research Institute. It was made from a steel rack fixed to the south building wall (*Figure 1*). The rack has wooden bars to hold up steel wire ($\varphi = 2$ mm) frames (dimensions: 55×120 mm) with wrapped PP fibre samples (*Figure 2*). Two irradiance sensors: CMP 3 and CUV 4 from Kipp&Zonen B.V. were fitted to the rack to measure light intensity within the following spectral ranges: CMP 3 – within the UV and visible range ($310 \div 2800$) nm; CUV 4 – within the UV range ($305 \div 385$) nm.

Single layers of PP fibres composed of at least 50 laps were wound on the steel frames. A strip of self-adhesive paper was stuck onto the reverse side of the layer (*Figure 2*) so as to be able, after slitting at this place, to remove the whole skein without tangling. Samples prepared in this way were installed on the wooden frame and subjected to solar irra-

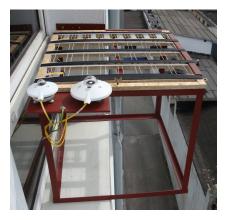


Figure 1. Test stand with frames for sunlight exposure.

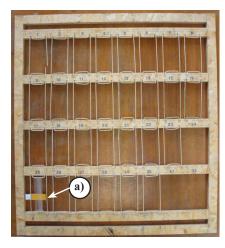


Figure 2. Frames for fixing fibre samples; a) stripe of a self-adhesive paper.

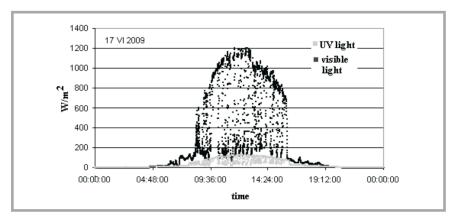


Figure 3. Diagram of the sun radiation energy on 17th June 2009.

diation. Everyday at the same hour, readings of the irradiance were taken within the whole range of the solar spectrum (sensor CMP 3) and UV range (sensor CUV 4), and the 24 h irradiation was calculated. Readings were recorded every 10 s on a CD cart in a Logbox recorder from Kipp&Zonen B.V. *Figure 3* shows an example of a 24 h recording of solar energy. The black points correspond to the visible range energy, while the grey points show the energy of the UV irradiation range. The 24 h integral P was calculated from the following formula:

 $P = \frac{24 \times 3.6 \times \sum W}{n} \tag{1}$

where:

P - irradiation, kJ/m²,

W- irradiance, W/m²,

n - number of measurements during 24 h.

From time to time, when changes due to PP degradation were expected, a fibre sample was taken from one frame to assess the fibre surface under a microscope. If changes were observed, the tensile strength (breaking force and elongation at break) was determined. At intervals of several or a dozen days or so, depending on the photo-degradation progress, successive fibre samples were assessed in the same way. After each sampling, the total energy dose to which the PP fibre sample was exposed until sampling was recorded.

The degradation of PP fibres under the influence of artificial light was examined with the use of Xenotest Alpha HE apparatus from Atlas. The apparatus was equipped with a 2200 W arc xenon tube cooled with air. Xenochrome 300 filters and an external Suprax glass cylinder were used to obtain a simulation of daily light outside buildings. Sensors provided temperature control inside the

chamber (CHT³)) and on the sample surface (BST⁴)). The distribution of the xenon lamp radiation used in this study is published in company materials of the Xenotest apparatus's manufacturer⁵), and by using appropriate filters it is similar to the average annual distribution of solar radiation in the temperate zone.

PP fibres were wound on standard holders of (135×45) mm dimension in such a way as to maintain a parallel arrangement of successive fibre sections and to avoid overlapping. The exposure surface was (121×35) mm, which provided a sufficient number of irradiated sections of a length required for testing the tensile strength of fibres.

Ageing tests simulating the degradation of PP fibres under the influence of sunlight were performed under irradiation conditions in the Xenotest Alpha HE apparatus, specially worked out for the test. These conditions are given in *Table 2*.

The surface degradation of PP fibres was assessed by means of a JOEL JSM-35C electron scanning microscope with a definition of 6 nm and a JFC-1100 'FINE COAT' APPARATUS for gold dust deposition from the Jeol. Computer filing and image analysis were performed with the use of the ORION 6 program. PP fibre samples were used to prepare specimens on carbon disks (Ø 10 mm). The surface of each specimen was dusted with gold and assessed by comparing microscopic images of an irradiated sample at the following magnifications: 600×, 1300×, 1500× and 2000× with microscopic images of an unirradiated sample. The length and width of crosswise crevices caused by photo-oxidation were measured and calculated for 10 sections of elementary fibres. Based

on statistical analysis of the results obtained, the degradation progress was determined. Tensile strength tests were performed according to Standard PN-EN ISO 2062:19976), using a Zwick 1120 tester, type CRE, with a head of load capacity 1 kN and with a constant elongation increment. For each variant of the fibres tested, 50 measurements of tensile strength were carried out.

In view of the length of the irradiated fibre section, 12 mm, the distance between the tester clamps was 100 mm. The clamps used were flat $(30 \times 30 \text{ mm})$ and covered with rubber to prevent fibre damage during clamping and drawing. The tension rate was set at 100 mm/min. The preload was established on the basis of the nominal linear density of PP fibres according to the principle: 0.5 cN/tex. The tensile strength of PP fibres was tested before irradiation (so called zero strength) and after each irradiation time established in the test schedule. The mean value of the breaking force and that of the breaking elongation were calculated. These results with their uncertainty are presented. The uncertainties were calculated according to *Equation 2*. Then the relative drop in tensile strength and elongation caused by irradiation was calculated.

$$u_A = \frac{s}{\sqrt{n}} \tag{2}$$

where:

 u_A - type A uncertainty,

s - standard deviation,

n - number of measurements.

Results and discussion

The effect of UV light on the topography of a PP fibre surface is shown in Figures 4 to 7 (microscopic photographs). Irradiation up to 30 000 kJ/m² causes no visible changes in the fibre surface, while higher solar irradiation, from 40 000 to 60 000 kJ/m², brings about individual damage, probably due to impurities from air e.g. polycyclic aromatic compounds from exhaust, which are the source of singlet oxygen, which can initiate polymer degradation [11]. An example of slight cracks on the surface of PP fibre ,,2" caused by sunlight with an irradiation of 38 305 kJ/m² is shown in Figure 4. An irradiation of 139 551 kJ/m² practically destroys PP fibre "1", developing deep crosswise cracks of various width and length (Figure 5). Similar but finer

Table 2. Conditions of PP fibres irradiation in Xenotest Alpha HE apparatus.

Testing conditions	I Phase without rain	II Phase with rain, 0.7 I/min		
Time of one radiation cycle, min	30			
Time of phase of radiation cycle, min	29	1		
Rotation of specimen holders	without rotation			
Testing time, h	changeable			
Irradiance energy, W/m ²	70 70			
BST temperature, °C	60	50		
CHT temperature, °C	40	35		
Air humidity, %	40	100 (rain)		
Filter	Xenochrome 300			

cracks on the surface of PP fibre "2" are caused by an irradiation of 116 646 kJ/m² (*Figure 6*). PP fibre "2" irradiated with an xenon light dose of 51 591 kJ/m² shows deep and wide crosswise cracks (sample 6 "2Xe", *Figure 7*). The microscopic images of crosswise cracks on PP fibres obtained by us are of the same character as reported photographs of PP fibres that underwent surface degradation under the influence of UV light [3].

Tables 3 and 4 (see page 56), changes in the tensile strengths of PP fibres "1" and "2" irradiated with sunlight and xenon tube light are reported. To compare the effects of irradiation with sunlight and xenon light, only UV radiation was used.

Changes in the tensile strength of PP fibres under the influence of sunlight are

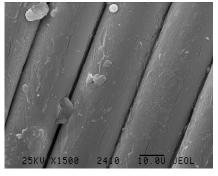


Figure 4. PP fibre "2" irradiated with sunlight, dose: 38 305 kJ/m² (specimen 23 2")

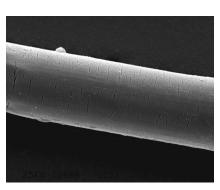


Figure 6. PP fibre "2" irradiated with sunlight, dose: 116 646 kJ/m² (specimen 21 "2").

smaller than those caused by xenon tube light. The same concerns fibre elongation. *PP* fibre "2" undergoes greater degradation than *PP* fibre "1" when irradiated with both types of light. After short irradiation with a dose of 11 654 kJ/m², the tensile strength of PP fibre "1" is even increased by 5%. This is consistent with the results of increased fibre strength reported after a relatively short time of irradiation with xenon light, which seems to be due to polymer cross-linking [2].

Figures 8 to 11 (see page 57) show the changes in the tensile strength and elongation of PP fibres "1" and "2", depending on the irradiation dose. The ratio of the breaking force drop rate of PP fibre "2" irradiated with an xenon tube to that of this fibre irradiated with sunlight amounts to 5.4, while the corresponding

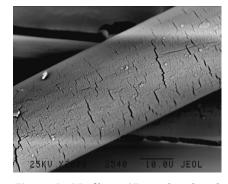


Figure 5. PP fibre "1" irradiated with sunlight, dose: 139 551 kJ/m² (specimen 5 "1").

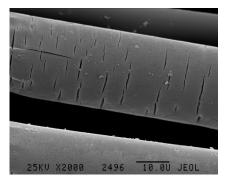


Figure 7. PP fibre "2" irradiated with xenon tube light, dose: 51 591 kJ/m² (specimen 6 "2 Xe").

Table 3. Percentage changes in the tensile strength of PP fibres "1" and "2" irradiated with sunlight.

	Exposure	Irradiation, kJ/m ²		Breaking	Breaking	Percentage change,	
of PP fibre specimens	time, days	UV	Visible	force, cN	elongation, %	Breaking force	Breaking elongation
8 "1"	9	11 654	145 962	314 ± 3	90.4 ± 1.3	+5.0	-4.3
2 "1"	16	22 456	266 854	287 ± 2	86.4 ± 0.7	-4.0	-8.6
7 "1"	31	46 950	501 112	262 ± 3	88.6 ± 0.9	-12.4	-6.2
6 "1"	41	64 909	626 032	262 ± 2	70.8 ± 0.9	-12.4	-25.1
5 "1"	91	139 551	1 094 386	236 ± 2	55.1 ± 1.5	-21.1	-41.7
24 "2"	15	25 864	234 258	178 ± 1	58.9 ± 0.9	-15.6	-21.6
23 "2"	24	38 305	334 776	171 ± 2	53.6 ± 1.4	-19.0	-28.6
22 "2"	47	80 277	631 760	169 ± 1	20.5 ± 0.7	-19.9	-72.7
21 "2"	75	116 465	897 532	140 ± 1	13.9 ± 0.2	-33.6	-81.5

Table 4. Percentage changes in the tensile strength of PP fibres "1" and '2" irradiated with xenon tube light.

Name	Fymaayya	Total	Breaking	Breaking elongation, %	Percentage change, %	
of PP fibre	Exposure time, days	irradiation, kJ/m ²	force, cN		Breaking force	Breaking elongation
1 "1Xe"	48	12 170	266 ± 2	95.1 ± 1,3	-11.0	+0.6
2 "1Xe"	72	18 255	254 ± 4	76.2 ± 2.0	-15.1	-19.4
3 "1Xe"	112	28 278	257 ± 4	71.6 ± 2.3	-14.0	-24.2
4 "1Xe"	228	58 309	180 ± 2	16.6 ± 0.5	-39.8	-82.4
1 "2Xe"	12	3 037	184 ± 1	68.1 ± 1.3	-12.8	-9.3
2 "2Xe"	24	5 979	171 ± 1	57.9 ± 1.0	-19.0	-22.9
3 "2Xe"	48	12 170	159 ± 2	32.4 ± 1.7	-24.6	-56.9
5 "2Xe"	112	28 278	148 ±2	16.9 ± 0.4	-29.9	-77.5
6 "2Xe"	204	51 591	51 ± 1	3,24 ± 0.1	-75.8	-95.7

Table 5. Rates of changes in fibre strength and elongation under the influence of sunlight and xenon tube light.

Light	Fibre	Speed of stren	ngth decrease	Speed of elongation decrease		
		× 10 ³ , cN/kJm ⁻²	R ²	× 10 ³ , %/kJm ⁻²	R ²	
Sunlight	1	0.5	0.836	0.3	0.924	
305-385 nm	2	0.5	0.850	0.6	0.821	
Xenon	1	1.9	0.954	1.4	0.940	
	2	2.7	0.927	1.4	0.855	
Sunlight 310-2800 nm	1	0.068	0.880	0.036	0.893	
	2	0.068	0.878	0.073	0.964	

Table 6. Measurements of cracks on PP fibres caused by photo-degradation.

	Cracks size							
Measurements/ specimen	Fibre PP "1", sunlight		Fibre PP "2", sunlight		Fibre PP "2", xenon light			
	specim	specimen 5 "1"		specimen 21 "2"		specimen 6 "2"		
Dose, kJ/m ²	139	139 551		116 469		51 591		
Mean number of the craks/100 µm	251	251.54		314.43		38.00		
Statistic parameter	Length, µm	Width, µm	Length, µm	Width, µm	Length, µm	Width, µm		
Mean value	2.40	0.19	1.47	0.19	4.91	0.42		
Median	1.97	0.15	1.26	0.17	4.66	0.39		
Standard deviation	1.68	0.07	0.79	0.07	2.63	0.15		
Number of measurements	160	60	200	50	100	50		
Min. value	0.56	0.07	0.45	0.07	1.20	0.12		
Max value	9.90	0.45	4.71	0.33	14.48	0.78		

ratio of elongation drop rates is 2.8. For PP fibres '1" the corresponding ratios amount to 3.8 and 4.7.

From the coefficients of the regression line slope in Figures 8 and 10, it follows that the strength of xenon light irradiated PP fibres "2" decreases 1.42 times faster than that of PP fibres "1", while the elongation drop rate is the same for both types of fibres tested. The strength drop of both PP fibres under analysis is the same when they are irradiated with sunlight, but the elongation of PP fibres ",1" decreases slightly faster than that of PP fibres "2". Polymer degradation under the influence of UV radiation proceeds in the surface layer, first of all from the irradiated side. The degradation inside fibres gradually proceeds as damage take place on the fibre surface, which may bring about a faster decrease in the strength of PP fibre "2" with a lower linear density. The rates of change in fibre strength and elongation caused by irradiation are listed in *Table 5*. This Table also contains line slopes of the dependence of strength and elongation drop rates on the irradiation within the range of visible light.

The ratio of the strength drop rate and elongation drop rate of both PP fibres is the same when exposed to equivalent doses of UV radiation, as well as when exposed to total solar radiation. The line slope values are lower by two orders of magnitude, although the irradiation within the range of near UV and visible light is higher by one order of magnitude than UV doses. Hence one can conclude that the photolytic degradation of a colourless PP fibre depends on the short-wave portion of UV radiation.

No simple correlation was observed between the decrease in fibre strength and the extent of fibre damage observed under a microscope. Cracks on the fibre surface are formed from the irradiated side, primarily on fibres uncovered by other fibres in the 17-filament bundle. During stretching, the most damaged fibres are broken first, but the strength depends on the least irradiated fibres. The figures given above show typical examples selected from the many microscopic specimens tested.

The damage of fibres caused by sunlight and xenon tube light was assessed by measuring the number, width and length of cracks visible in the microscopic photographs. In *Table 6*, the crack dimensions of both types of PP fibres are

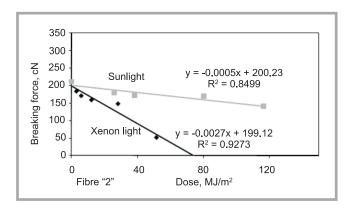


Figure 8. Comparison of the tensile strength decreases of PP fibres "2" irradiated with xenon tube light and sunlight.

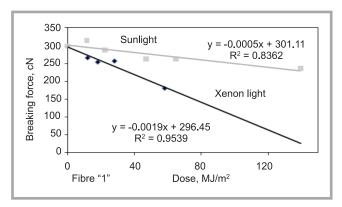
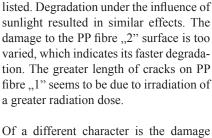


Figure 10. Comparison of the tensile strength decreases of PP fibres ",1" irradiated with xenon tube light and sunlight.



caused by xenon tube light - there are fewer cracks, but they have a considerably greater length and width. This explains the substantially faster drop in the strength of fibres irradiated in the Xenotest apparatus as the fibre strength depends on the weakest points, i.e. those with the greatest cracks. Therefore the greater but not so deep damage caused by sunlight does not cause such intensive decreases in strength. From the comparison of the strength results of fibres after irradiation and microscopic images of their surface, it can be concluded that clear cracks appear on the fibre surface only when their strength is decreased below 50%.

Conclusions

The tests performed indicate that the assessment PP fibre photo-degradation

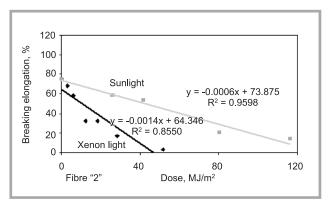


Figure 9. Comparison of the elongation decreases of PP fibres "2" irradiated with xenon tube light and sunlight.

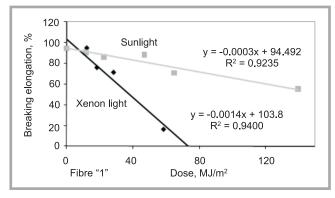


Figure 11. Comparison of the elongation decreases of PP fibres , I" irradiated with xenon light and sunlight.

with the use of an xenon tube provides similar results, at least in the case of colourless fibres, to those obtained during exposure to sunlight under natural conditions. Although the irradiation of an xenon tube reduces the tensile strength of PP fibres more than that with sunlight, the differences are not significant i.e. the drop in both the fibre breaking force and elongation at break. The damage to a PP fibre surface caused by the irradiation of a xenon tube is wider and deeper but less substantial than that brought about by sunlight. Slight differences in the photodegradation rate of PP fibres under the influence of artificial light and sunlight, despite the use of filters providing a spectrum similar to that of sunlight, can result from the day/night cycle during exposure under natural conditions, as well as from the temperature and humidity difference of samples.

PP fibres containing a biodegradation-accelerating agent are weakened faster under the influence of xenon tube light than pure PP fibres, but both types of fibres react to the action of sunlight similarly.

Based on the tests performed, the following conclusions can be drawn:

- 1. The assessment of PP fibre photodegradation with the use of an xenon tube provides similar results, at least in the case of colourless fibres, to those obtained during exposure to sunlight under natural conditions.
- The action of xenon tube radiation decreases the tensile strength of PP fibres to a greater extent than the radiation of sunlight. This concerns both the breaking force and elongation at break.
- **3.** Damage to the surface of PP fibres under the influence of xenon tube radiation is wider and deeper, though less substantial, than that caused by sunlight.
- 4. PP fibres containing a biodegradation-accelerating agent are weakened faster under the influence of xenon tube light than pure PP fibres, but both types of fibres react to the action of sunlight similarly.
- 5. Differences in the photo-degradation rate of PP fibres under the influence of artificial light and sunlight, despite the use of filters providing a spectrum similar to that of sunlight, can result from: the day/night cycle during exposure under natural conditions, as



11th World Filtration Congress

WFC11 Background Info:

The 11th World Filtration Congress (WFC11) will be held from April 16 - 20, 2012 in Graz, Austria and is expected to draw more than 2,000 attendees. WFC11 is hosted by the Austrian Chemical Society GÖCH.

"We are proud that Austria has been selected to host the 11th Word Filtration Congress", said Prof. Wilhelm Höflinger. The venue Graz is a modern Convention City with over 1000 years of tradition, superb meeting facilities and infrastructure situated in the heart of Europe. It is a top venue for the event in the field of filtration & separation. We are looking forward to welcoming filtration experts from all over the world.

The WFC11 in Austria will embrace the international F&S community five days from Monday to Friday. The first day is foreseen for technical short courses presented by leading experts. Three and a half days are planned for the technical congress. More than 350 technical presentations are expected and a major international Exhibition from the world wide filtration and separation industry as well as manufacturers of particle measurement instruments and equipment of related industries with 100 exhibitors. The afternoon of the last day is reserved for a post-congress plant tour to ANDRITZ headquarter followed by a wine-tasting tour to Eggenau Castle.

In GRAZ, a Unesco World Heritage Site, the Mediterranean atmosphere of the Old Town, the striking modern architecture and the great cultural diversity together create a unique and fascinating mix. As Graz is located in a valley surrounded by mountains, with the river Mur flowing through the heart of the City you will enjoy its mild spring climate. In 2003 the City of Graz was Europe's Cultural Capital and it offers delegates and visitors alike a unique experience in art, culture and history in a warm and welcoming atmosphere. It is also ideally situated to offer interesting sightseeing visits within short distances. The new international airport Graz-Thalerhof with its domestic and international flights is located only 10 km from the new Congress Venue. The Central Railway Station has both Austrian and international connections with fast and frequent services to the Capital Vienna.

Late abstracts for the 11th World Filtration Congress to be staged in 2012 in Austria are still accepted.

For more information:

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well as from the sample temperature and sample humidity difference.

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Editorial notes

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