

Natural ageing of polyaramide fiber from ballistic armor

Vitor Hugo Cordeiro Konarzewski^{1*} , Fernando Ludgero Spiekemann¹ and Ruth Marlene Campomanes Santana¹

¹Laboratório de Polímeros – LAPOL, Universidade Federal do Rio Grande do Sul – UFRGS, Porto Alegre, RS, Brasil

*vitorhk@sinos.net

Abstract

Ballistic armor has been manufactured primarily based on polyaramide (Kevlar and Twaron) or Dyneema but the lifespan warranty in Brazil is only 5 years and after this time period they are incinerated or comminuted and ground up. This study aims to evaluate the changes on the physical, mechanical and morphological properties of polyaramide fibers of ballistic armor after natural aging. These samples with different fabrication (2005 and 2010) and usage time were exposed to natural weathering in the city of Porto Alegre, southern Brazil, during the period of one year. Morphology fiber results surfaced after ageing, it showed fiber swelling, stress cracking and defibrillation, and the results of the mechanical tensile testing of the polyaramide fibers showed a pronounced decrease (80%) in tensile strength. It can be concluded that the weight, the dtex of the fiber and the kind of fabric can influence the degradation degree under natural exposure.

Keywords: ballistic armor, aramid fibers, polymer degradation.

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1. Introduction

The ballistic armor market in Brazil is approximately one million users, who act directly in police actions and other inherent activities^[1,2]. With an expiration date of five years from the date of manufacture, the legislation in Brazil determines the destruction of the material by shredding or incineration after the theoretical expiry date, without taking into account the commercial value of the polyaramide or the stability of the raw material^[3-5]. The use of lightweight ballistic vests was increased after the 1960s with the development of high-performance aramid fibers, or poly (p-phenylene terephthalamide), referred to as Kevlar by DuPont, registered in 1971^[6]. Another registered polyaramide trademark of Teijin, Twaron, whose properties of heat resistance, flexibility, dimensional stability and high mechanical resistance have made the polyaramide fibers quickly gain space in the most demanding industrial applications and also in the security area, being subject to constant study and improvement for use in ballistic armor^[7-9]. There are usually two basic characteristics that respond to the trust placed in the aramid shield until now: the chemical composition of the fiber and the way the yarns are woven and interlaced in multilayers. Under the impact of the projectile, the energy is dissipated through the panel, by friction, absorption and dissipation of the shock waves avoiding perforation^[10-17]. The aromatic polyamides were obtained by reactions that lead to the formation of amide bonds between aromatic rings of high thermal stability and high strength^[18-21]. The exceptionally rigid molecular chain structure, good orientation and organization of the crystalline structure provides high strength and low elongation of the orientation

of the molecular chains, offering high tensile strength, impact and with differentiated thermal stability for various temperature ranges for an extended time^[22-25].

The susceptibility of aramid fibers exposed to light and moisture is confirmed by several researchers, such as Bittencourt^[12], Wang et al.^[26] and De Paoli^[27], among other authors. These works describe the fiber surface and the photo degradation process, which gradually reduces its molar mass, mainly due to oxidation and the splitting of the amide group. Another factor that may influence the degradation is the presence of impurities in the fiber due to the weakening of the structure by the formation of free radicals and causing the polymer chains to break up, which characterizes the hydrolytic degradation of the polyaramide fibers^[28-33]. The absorption of humidity together with the presence of oxygen and moisture can produce molecular reorganization, from the breakdown of a molecular chain to its oxidation^[9,34]. Another factor that may influence the polymer life cycle is related to the effect of stress cracking, which may influence the macroscopic properties of the tissues, such as the weave and warp of the fabric^[35,36]. The degradation process may also cause changes in the properties of the polyaramide, such as loss of brightness, changes in color, cracking and the decrease of mechanical properties^[26,37]. The blades of armor are not exposed directly to solar radiation, nor direct exposure to weathering, since the blades are arranged in layers, coated by at least two additional layers of different impermeable fabrics^[1,38].

In this sense, the objective of this study is to evaluate the loss of the mechanical properties of polyaramide fibers

used as raw material for the manufacture of ballistic armor with different dates of manufacture and use after exposure to natural weathering.

2. Materials and Methods

2.1 Material

The polyaramide fibers came from tissues samples of ballistic armor that was more than 5 years old, that had been disposed of by the logistic center of the Brigada Militar of the State of Rio Grande do Sul. The fiber samples had a dtex of 1670 (2010 - unused vest) and 933 (2005, for a vest that was normally used for five years), referred to as K10 and K05 respectively. The fabrics were trimmed into squares sized 100 mm × 100 mm, and were exposed to natural weathering following ASTM standard^[39-41]. The main characteristics of the fibers used for the tensile test are summarized in Table 1 and Figure 1 showing the difference of the weave of the polyaramide fabric samples from 2005 (left) and 2010 (right) fabrics.

2.2 Exposure to natural ageing

The specimens tested (100 mm × 100 mm) were exposed to weathering for 12 months (from February 2014 to February 2015), under real climatic conditions in accordance with ASTM 1435-13. Specimens tests were exposed to natural sunlight outdoors in northern direction at 45° inclination as can be seen in Figure 1.

The geographical location of the samples was latitude (30° 05' South), longitude (51° 11' West), and altitude (174 m) in the city of Porto Alegre, in the south of Brazil. This allowed for a normal incidence of solar radiation over the entire surface of the samples. During the natural weathering test it was monitored the average UV radiation index, temperature, and the rainfall that was provided by the CPTED-INPE (Center for weather and climate studies - National Institute for Space Research - Brazil). Figure 2 shows the climatic conditions (maximum Precipitation and

Table 1. Characteristics of the polyaramide fibers.

| Sample | K05 | K10 |
|------------|------|------|
| Denier | 840 | 1500 |
| Dtex | 933 | 1670 |
| Warrantee* | 2010 | 2015 |

*Warrantee given by the armor manufacturers.



Figure 1. Samples from armor fabrics used in this research.

maximum UV index (UVI), moisture and average total monthly rainfall) during the total period studied.

It was observed that the highest temperatures occurred during the months of November 2014 to March 2015, but the maximum temperature in January 2015, which reached over 2014 to January 2015, and between September 2014 to January 2015 the frequency of rainfall was higher, reaching 778 mm values. As a result, the relative humidity was high during this period, between 70 to 90%. Regarding the UVI, the values were high during the spring and summer, reaching values over 12, which is classified as extreme.

2.3 Colorimetric and optic analysis

The surface color of the polyaramide samples (every two months) were measured using a Spectro-Guide spectrophotometer, BYK, in accordance with ASTM D2244. Spectrometer's Spectro-Guide software transforms spectral data into CIELAB color coordinates (L^* , a^* , and b^*) based on a D65 light source. Lightness (L^*) and two chromaticity coordinates (a^* and b^*) were measured at three different positions on each sample. In addition, the gloss (G) of the samples was measured. An increase in L^* value means the color of the sample becomes lighter. A positive a^* signifies a color shift towards red, and a negative a^* signifies a color shift towards green. A positive b^* signifies a shift towards yellow, and a negative b^* signifies a shift towards blue.

2.4 Tensile test

The fiber tensile test was performed on the INSTRON 3382 universal testing machine based on ASTM D7269M-11^[42]. By limiting the length of the fibers to 100 mm, it was opted for the D3822M-14 standard^[43]. As can be observed in Figure 3, the fibers were glued on to a cardboard base, with a grammage of 180 g. m⁻² and dimensions of 2 × 5 mm, being cast to the center with a diameter of 9 mm. Tensile tests of polyaramide fibers were carried out according to ASTM D3822M-14 in a universal test machine using specimens tested with a length of 100 mm and a load cell of 1000 N, an extension speed of 5 mm min⁻¹, and a gauge length of 25 mm. Each tensile value reported is the average of a 10 specimen test.



Figure 2. Climatic precipitation and Ultraviolet Index (UVI) in the city of Porto Alegre (2014-2015).

2.5 Morphologic analysis, SEM

For the scanning electron microscopy (SEM) analysis, the samples were deposited in a carbon type stuck to stub, metalized with gold. For image acquisition, a SEM model JOEL 6060 was used with 2 kV and magnification of $1000 \times^{[44]}$.

2.6 Diameter calculation of the polyaramide fiber

For the calculation of the polyaramide fiber diameters a 50 micron scale was used and based on the image it was divided into an area of nine equal divisions containing a significant amount of fibers for analysis, the average was

obtained. Within each area, the amount of fibers “j” were counted, obtaining the same diameter as “fj”. With both of the samples, the average of the area “Zi” was obtained. The procedure was repeated in the delimited areas, and the mean diameter was calculated. The advantage of this method was the correct division of the zones with a low error in the diameter estimation, with two weights, considerably reducing the error.

3. Results and Discussions

3.1 Colorimetric and optical proprieties

Figure 4 shows the results of the colorimetric parameters of the polyaramide K05 and K10 samples before and after exposure to natural weathering. The most significant change in color with a tendency to darken the samples from yellow to a darker shade occurred in the first two months of exposure, with a reddish tendency due to the increase of parameter “+a” to positive values, changes that can be better visualized in Figure 5, with woven samples before and after exposure. In addition, a reduction of parameter “b*” lower yellowness was observed, because of the increase the redness (+a) of the sample. These color changes indicate chain cleavage due to the hydrolytic degradation of the polyamides.

Such photo degradation features are reported by Bittencourt^[12] and Billingham^[34], describing the degradation processes of the polyaramide fibers. The change of coloration, as well as the loss of brightness, represented by the decrease of Luminosity (c) (the fiber became more opaque) and increase of fiber roughness. The decrease of the Gloss (d), can be attributed to hydrolysis too, in addition to this, there was environmental stress cracking too, due to the climatic

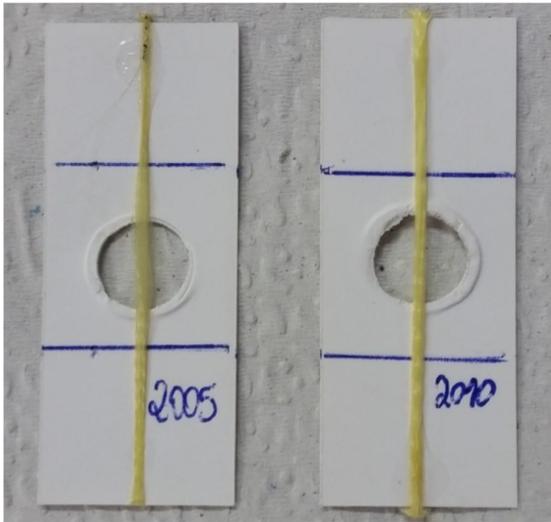


Figure 3. Specimen test preparation for tensile test.

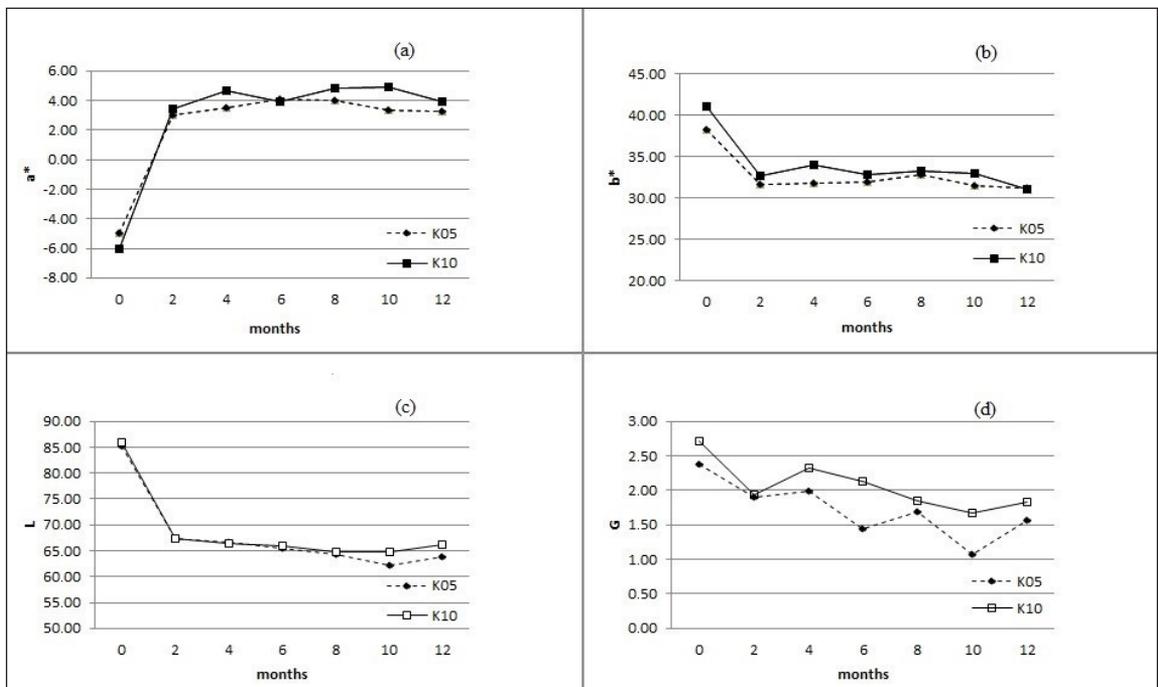


Figure 4. Colorimetric and optical results of the samples before and after natural ageing: (a) parameter “a”; (b) parameter “b”; (c) Luminosity; and (d) gloss.

conditions to which they were exposed, greater periods of precipitation and temperature variations, which depleted the fibers, being faster in samples k10. The signs of degradation reached the superficial layers of the tissues, causing an initial discoloration, facts reported by Wang et al.^[26] and De Paoli^[27]. The initial colorimetric results of the degradation of the samples were very similar for the K05 and K10 samples. However, due to the difference in the fabric weaving and the yarn, after two months of continuous exposure to the

weather, it was observed that the brightness indicators related to the roughness were not uniform. Such signs may indicate a fiber degradation process, which is directly related to the exposure area, as well as the interlacing of the threads.

3.2 Morphological properties, SEM

Figure 6 shows the micrographs of the polyaramide fibers of samples K05 and K10 before and after exposure to natural weathering. It was possible to verify that both of them presented a clean and regular surface before exposure, but after four months it was observed that the fibers K 05 presented less aggression on their surface when compared to the fibers K10, this was probably influenced by the type of weave, weight and lower dtex as shown in Table 1. The degradation of the samples after exposure to natural weathering is related to UV radiation, acid rain and impurities, which under temperatures ranging between 3 to 40 °C altered the fibers over a year. The increase of striations and surface deformations can be attributed to hydrolysis, stress cracking as well as UV radiation, with morphological changes being evident visually similar to the two aramid samples^[26,27]. For the initial sample, without exposure to the k05 and K10, no signs of premature wear of the samples were observed. Such morphology denotes the stability of the fiber, since it is not exposed to light effects^[33,36]. These effects were observed with the progressive appearance of striations, roughness, surface defects and incrustations, indicating the progressive degradation of the fiber^[36,37]. After four months the morphological changes became more visible, with the appearance of cracks showing a few obvious signs of fiber



Figure 5. Color changes of the samples before and after natural ageing.

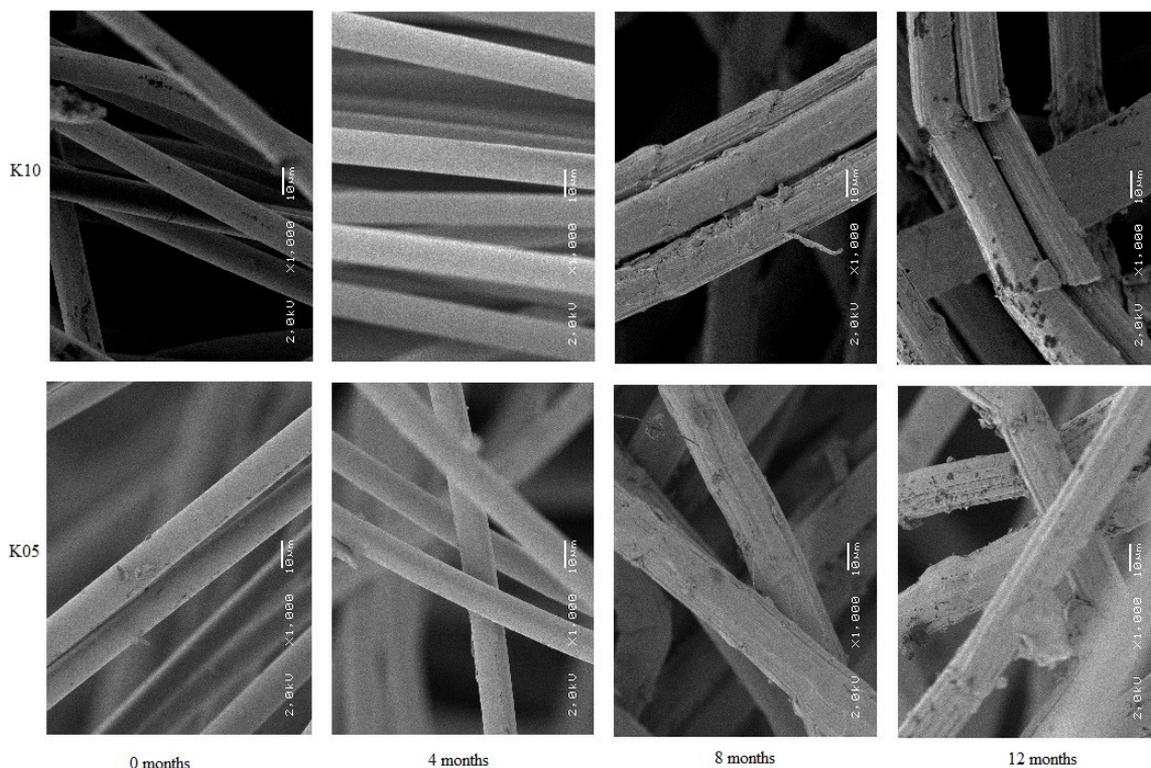


Figure 6. MEV from samples K05 and K10 before and after natural ageing.

degradation, especially in the K10 sample, which despite having a larger dtex, had a more open tissue weave.

Photo degradation was more severe with the more open frame, exposing the samples to light and humidity, increasing photochemical degradation, hydrolysis and stress-cracking^[27,30,31]. The variation of fiber diameters during one year, as shown in Figure 7, caused a light increase of the diameter of the aramid fibers of sample K10, when compared to sample k05.

Comparing the sample K10 with K05, from a ballistic vest used over 5 years, it was observed that the diameter of the fibers is very similar in the initial sample “0 months”, not presenting high degradation or absorption of significant humidity over five years of sample difference^[12,39]. With the exposure time, the K 05 sample presented a slightly lower humidity absorption over the period of one year, however an increase of the sample diameter occurred, caused by hydrolysis degradation. The initial diameter of the fibers corresponds to that reported by other researchers when characterizing the diameter of the polyaramide fibers 49, around 12 μm , for new fibers^[6,20,22]. These results reinforce the hypothesis of fiber stability, even after five years of use (before natural ageing), maintaining similar morphological characteristics as when it was new, thanks to the protective cover of the ballistic panels, avoiding direct exposure to the sun or hydrolysis of the fiber.

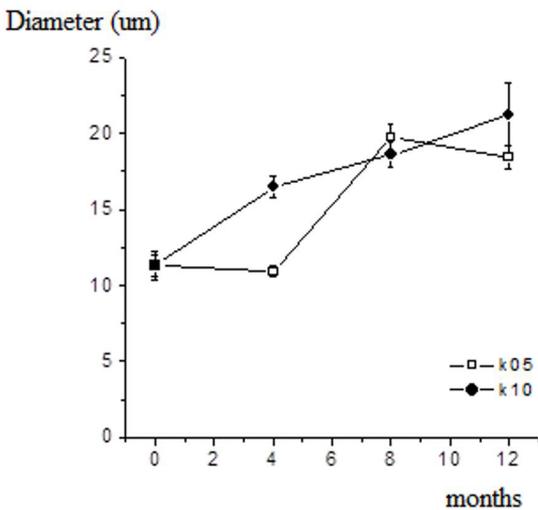


Figure 7. Diameter changes of polyaramide fiber after natural ageing.

3.3 Mechanical properties

Table 2 shows the average results of the maximum tensile stress of fibers K05 and K10 before and after exposure to natural weathering. Comparing the initial results, the polyaramide fiber presented good stability, even considering the time of use, storage and the fact that the samples k05 and K10 were removed from fabrics, theoretically subjected to mechanical degradation in function of the weaving^[11,23,30]. Table 2 also summarizes the mechanical properties of maximum stress until fiber rupture and elongation, in mN/tex, recorded during the tensile test. Considering the difference of origin of the fibers, it was decided to compare the data in mN/tex, and it was possible to verify the reduction of strength force and elasticity over a year^[16,26,30]. The hydrolysis, stress-cracking environment and photo degradation mechanisms present on the fibers degradation process were more accentuated in the first 60 days, remaining stable for two months with resistance decline and reduced after 6 months.

As can be seen in Figure 8, the major change occurred after two months, when K10 fiber presented only 52% of the maximum stress until rupture and the sample K05 34%, proving the loss of properties of the polyaramide when submitted to natural ageing.

The initial reduction of resistance of samples k10 and k05 can be attributed to the amount of fibers and the stress-cracking environment, hydrolysis and photo degradation, since the sample k 10 had a higher amount of fibers when compared to sample K05, which when exposed to the weather were affected from the outside to the inside^[31]. From the fourth month of exposure until the sixth month, the sample k 10 had a more pronounced loss of strength, after that the samples started to have a similar resistance decrease, reaching 18% of the original resistance of the yarns in 12 Months of exposure^[22,26,38].

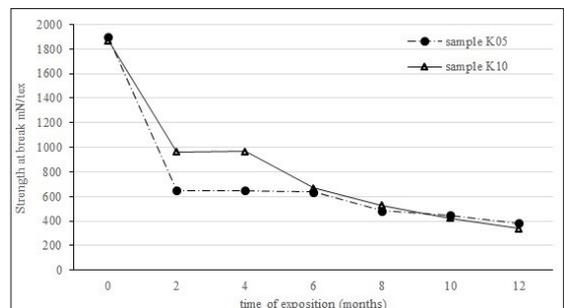


Figure 8. Maximum tension from polyaramide by natural ageing (mN/dtex).

Table 2. Mechanical proprieties of polyaramide fibers.

| Exposition time (months) | Breaking strength (mN/tex) | | Elongation at break (%) | |
|--------------------------|----------------------------|-------------|-------------------------|--------|
| | K 2005 | K 2010 | K 2005 | K 2010 |
| 0 | 1898 ± 159.4 | 1870 ± 95.2 | 6.0 | 7.0 |
| 2 | 649 ± 134.3 | 964 ± 180.5 | 5.0 | 5.0 |
| 4 | 650 ± 57.2 | 968 ± 118.4 | 4.0 | 5.0 |
| 6 | 637 ± 151.4 | 667 ± 85.6 | 3.8 | 4.0 |
| 8 | 483 ± 115.7 | 526 ± 31.7 | 3.0 | 3.3 |
| 10 | 445 ± 88.4 | 422 ± 54.7 | 2.0 | 2.7 |
| 12 | 382 ± 63.3 | 339 ± 23.3 | 3.0 | 2.3 |

4. Conclusions

Results of this study showed that new or used polyaramide blankets (before exposure to natural weathering) did not present colorimetric, optical or mechanical changes. However, after exposure to natural weathering for a total period of one year, in the city of Porto Alegre, RS, Brazil, they showed a pronounced color change and a considerable loss in the mechanical performance of the fibers during the first 2 months. Highlighting the largest changes in K10 samples, although this has never been used, but with different weft and dtex from the sample used for 5 years K05a. K05 samples showed higher mechanical performance and lower swelling and less aggression from environmental stress-cracking in the first 2 months. Finally, it is concluded that all samples analyzed after exposure to natural weathering presented hydrolytic degradation, environmental stress-cracking and photochemistry.

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