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Biodegradable Polyamide 6.6 for Textile Application

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Abstract

The study evaluates comparatively some physical and chemical properties of polyamide 6.6 standard and biodegradable. It also evaluates the period of biodegradation of the biodegradable yarn sample and standard sample. The physical properties analyzed were tensile strength, elongation, and tenacity. The chemical properties were related to the behavior of the samples in dyeing and the evaluation of subsequent strength dyeing. The evaluated samples were taken from knitwear produced with polyamide textured filament yarn 80 dtex f 68x1, standard and biodegradable, being purged, bleached, and dyed. The results of the physical tests, although statistically different, have values very near the average, which in practice represent acceptable values within the statistical control process. Both standard and biodegradable samples had the same chemical behavior and there is no difference. Concerning to biodegradation time under laboratory conditions, the carbon dioxide produced by the samples was monitored and measured to determine the percentage of biodegradation according to ASTM D 5511. After 735 days the percentage of biodegradation of the biodegradable yarn was 81.7% and of the normal yarn was 5.2%. This is an expressive gain in ecological terms for synthetic fiber.

Keywords: textile biodegradation, shorter life-cycle, sustainability, low environmental impact

1. Introduction

According to Milan (2010), a problem with the environment is currently related to the quality of life and future generations, which makes it a search for the development of a collective conscience. Market competition and a new perspective are companies that seek solutions and technologies focused on reducing risks and optimizing production processes, greater in social and ecological responsibility, products with added value and competitiveness.

Textile is an indispensable element of human society throughout its history. In addition to the maintenance of basic needs—protecting themselves from the outside environment and maintaining necessary conditions for survival, human beings have been using textile products as means of expressing their identities, wealth, power, and the like, and it has been an important commodity traded globally over centuries (Tojo et al., 2012).

According to Refosco (2012), the textile industry transforms fibers into yarns, yarns into fabrics and fabrics into garments and home products, and into technical textiles with diverse applications. By analyzing the industries by branches of activity, the textile industry, with the whole chain of clothing-related activities, has not developed proportionally to its worldwide expansion the concerns about the materials and processes, causing grave consequences as textile garbage and the exorbitant quantity of textile wastes from the production process.

Pekhtasheva et al. (2012) state that along with general similarities in the structure of high molecular weight compounds, fibers differ from one another by chemical composition, monomer structure, polymerization degree, orientation, intermolecular bond strength and type, and so on which define the different physicomechanical and chemical properties of the different fibers. The resistance of fibers and fabrics to biodamage depends primarily upon the chemical nature of the fibers from which they are made. Plant-derived natural fibers, such as cotton and linen, are particularly susceptible to attack by saprophytic microflora. Man-made fibers and fabrics, especially synthetic ones, are more resistant to biodegradation, but biodegrading microorganisms can adapt so that they can use them as food sources.

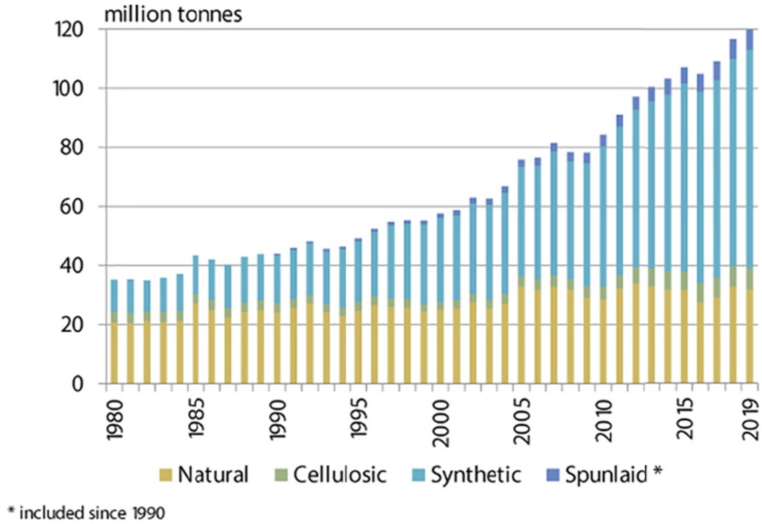

Figure 1. Global demand for textile products (Engelhardt, 2020)

Figure 1 shows the global demand for textile products is steadily increasing. Therefore, there is an increasing demand for synthetic fiber, but its rate of biodegradation is much slower compared to natural and artificial fibers, which consequently directly impacts the increase in the generation of textile waste on the planet.

According to Ellen Macarthur Foundation (2017), of the total fiber input used for clothing, 87% is landfilled or incinerated and 2% is sent to landfill or incineration from garments that are produced, yet never make it to market. Overall, one garbage truck of textiles is landfilled or incinerated every second. Less than 1% of the material used to produce clothing is recycled into new clothing. So, there is a large share of textiles that will go to waste and their destination will be the landfill. That is why the topic of biodegradability in textiles is of extreme importance and relevance.

There is a worldwide research effort to develop biodegradable polymers as a waste management option for polymers in the environment. The works of Krikštanavičienė et al. (2014), Ozgen (2012), Ekevall (2004) investigated biodegradable fibers made of polylactic acid (PLA) from renewable resources.

Regarding de degradation of polyamide fiber, Klun et al. (2003) investigated the Polyamide-6 fiber degradation by filamentous fungus Phanerochaete chrysosporium under submerged cultivation conditions. Szostak-Kotowa (2004) reviewed some studies about polyamide. Some studies state that polyamide is not biodegradable and others state that can be degraded in special conditions. Shamey and Sinha (2008) reviewed the environmental processes degradation of nylon 6.6 and explain the degradation mechanisms, analysis methods to measure the degradation, types of degradation. Biodegradation is classified by the authors as a type of degradation. Several publications have been devoted to study the development of biodegradable polyamide. However, the works are on a laboratory scale. This work presents the results of tests of a biodegradable polyamide produced on an industrial scale.

Despite all the importance in the issues of reducing the life cycle that a biodegradable product can present, in the case of a textile yarn it is also extremely important that the product has properties compatible with textile processing regarding aspects such as processability, good dyeing, and good mechanical properties that allow later use in a garment.

The contribution of this research is to investigate the physical properties: tenacity, elongation, and strength and chemical properties: colorfastness to light, colorfastness to perspiration, and colorfastness to wash of the biodegradable polyamide 6.6 yarn compared with the respective properties of polyamide 6.6 yarn standard. In addition, the biodegradation test was also performed.

1.1 Textile and the Environment

The interaction between textile materials and the environment is a complex one taking two distinct forms. First, the effect of a change in properties that the environment can bring about in the textile, is classed as degradation. Second, there is the way the production or use of textiles can impinge on the environment, classed under the term' pollution for the negative impact', but also including environmental protection by pollution reduction where, say, a landfill liner is used to prevent leaching. A further need is to produce textiles to be possible by using the resources available on the earth without depleting them irreplaceably. Each of these factors is important and should be considered separately to build up a complete view of how textiles and the environment can impinge on one another (Slater, 2000).

1.2 Polyamide 6.6 Fiber

Polyamide, also known as nylon, is a synthetic polyamide with repeating amide groups (-CONH-) in its backbone Polyamide 6.6 is produced from hexamethylene diamine and adipic acid. According **to** Mahdi (2016), biodegradation of synthetic polyamides is known to be poor, although its chemical structure (presence of amide bonds in the main chain) resembles those of natural proteins and synthetic polypeptides. The high resistance to degradation of synthetic polyamides is caused by the high symmetry of their molecular structures and strong intermolecular cohesive force caused by hydrogen bonds between molecular chains, which results in highly crystalline morphology.

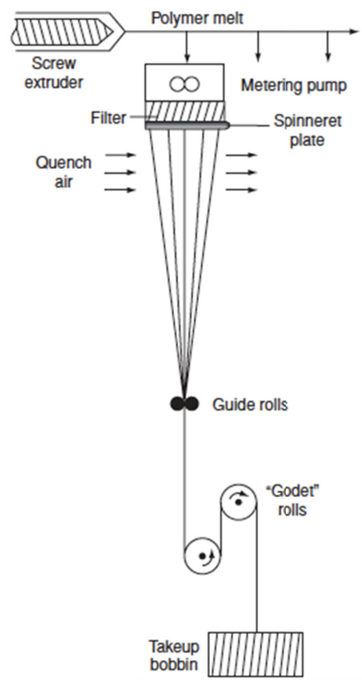

Figure 2. Melts pinning process scheme (Denn, 2008)

The melt spinning process for the manufacture of textile fibers is shown schematically in Figure 2. The polymer from the extruder, after passing through a filter to remove small gel particles, is forced through a small hole known as a spinneret, which is typically about 200–400 μm in diameter. The jet emerges into an ambient environment that is below the solidification temperature. The filament is taken up on a roll moving at a much higher linear velocity than the extrusion velocity; the take-up speed is typically more than 3,000 m/min while the average linear velocity through the spinneret is typically two orders of magnitude smaller (Denn, 2008).

1.3 Biodegradation of Polymers

Biodegradation of polymers is seen as one of the solutions for current plastic waste management problems (Chonde, 2012) since plastics are very resistant to decomposition and may stay in the environment for centuries (Peng, 2020).

Biodegradable polymers are plastics with equivalent properties to conventional plastics, but they are capable of being decomposed and catabolized eventually to carbon dioxide and water by microorganisms and/or enzymes (Tokiwa, 2015) and may involve both hydrolysis and oxidation (Balaji et al., 2018). Sometimes, depending on the environmental conditions, the biodegraded matter is converted into methane (Siegenthaler et al., 2011). The rate of biodegradation may vary substantially and depends on the molecular structure, morphology, surface area, etc. (Klun, 2003). A degradation of polymers may proceed by one or more mechanisms, including microbial degradation in which microorganisms such as fungi and bacteria consume the material (Chonde, 2012).

Both the chemical and physical properties of polymers influence the mechanism of biodegradation. The surface conditions (surface area, hydrophilic, and hydrophobic properties), the first-order structures (chemical structure, molecular weight, and molecular weight distribution), and the high order structures (glass transition temperature, melting temperature, modulus of elasticity, crystallinity, and crystal structure) of polymers play important roles in the biodegradation processes (Tokiwa et al., 2009).

Biodegradation is not dependent on the origin or the raw material base of a substance but is only a function of its chemical structure (Siegenthaler et al., 2011).

Biodegradable polymers come from various sources, from synthetic to natural polymers, and can be classified according to their origin into three classes: naturally produced renewable polymers, synthetic polymers derived from renewable resources, and synthetic polymers derived from petroleum-based resources (Ashter, 2016).

Biodegradable plastics can be transformed in nature into water and CO_2 or CH_4 during a period comparable to that of organic matter (Sanz-Lázar, 2021).

1.4 Biodegradable Polyamide 6.6 Fiber

Polyamide 6.6 had good mechanical strength but shows low biodegradability and has a high melting point (Okamura et al., 2002). According to Redondo (2018), the patented biodegradable polyamide fiber was obtained by adding a biodegradation agent during the melt-spinning extrusion of polyamide 6.6, so the biodegradation agent is melt-mixed with the polyamide, before the formation of the fiber. The additive enhances the biodegradation process through a series of chemical and biological processes when disposed of in a biologically active landfill. The biodegradation process begins with swelling agents that, when combined with heat and moisture, expand the polyamide molecular structure. The biodegradation agent causes the polyamide to be an attractive food source to certain soil microbes, encouraging the polyamide to be consumed more quickly than polyamides without the biodegradation agent. The polyamide 6.6 of this study is degraded by an anaerobic biodegradation process which is, according to Siegenthaler et al. (2011), typically found in aquatic degradation processes, in landfills or is used technically to produce biogas from biomass, because of the major gaseous degradation product is methane along with CO_2.

2. Materials and Method

2.1 Materials

In the experiments were used 100% standard and biodegradable polyamide 6.6 yar, both textured filament with yarn count 80 dtex f 68 x 2, produced by Rhodia Solvay in Brazil.

Knitted fabrics were produced using both polyamide 6.6 yarn, resulting in a biodegradable and standard sample. The fabrics are constructed in a circular knitting machine in a single jersey and have the following characteristics:

Regular sample: single jersey 100% polyamide 80 dtex f 68 x 2 standard

Biodegradable sample: single jersey 100% polyamide 80 dtex f 68 x 2 biodegradable

2.2 Textile Process

After knitted fabrics were prepared, the following processes were done in the samples:

2.2.1 Scouring

The standard and biodegradable samples were boiled in an alkali scouring solution prepared from 1.0 g/L detergent and 2% g/L Na_2SO_3 at 80°C for 30 min with a liquor ratio of 1:20. After scouring the samples were washed for 10 min, neutralized with citric acid for 10 min, and washed again for 10 min.

2.2.2 Scouring + Bleaching

The samples were bleached to verify how the bleaching process can influence the biodegradable samples and simulate the industrial use of the yarn.

Scoured samples were bleached according to the following conditions: Components of bleaching solution used were 0.1 g/l citric acid and 0.45% Uvitex NFW 450% (optical bleach); liquor ratio 1:20; heating from 30 to 100 °C, exposure at 100°C for 60 min.

2.2.3 Scouring + Dyeing

The dyeing of scoured samples was performed through the instructions of the dye manufacturer. The samples were dyed at 100℃ for 60 min with 0.35% Blue Astracid ASG 200% (acid dye), 2% Lyogen P (Leveling agent), and 2 g/l Ammonium sulfate. After dyeing, the samples were washed three times. Dyeing was performed in closed beakers at a 1:20 liquor ratio in a laboratory dyeing machine.

2.3 Methods

Table 1. Tests and standards

Test	Standard
Tensile strength and elongation at break	ASTM D3822
Color Fastness to Light	ABNT NBR ISO – 105 B02:1994
Color Fastness to Perspiration	NBR 8431
Color Fastness to Wash	ABNT NBR 13097
Transfer of color	NBR 8429 And NBR 8430
Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions	ASTM D5511

2.3.1 Tensile Strength and Elongation at Break

Tensile property can be defined as the maximum force/load that is required to break the material. The tensile property is one of the key factors that determine the quality of the fabric. This type of measurement shows how a material will behave when it is subjected to a tensile pull or force. The breaking load and the elongation at break are the most valuable information derived from a tensile test.

Tenacity is the measure of the breaking strength of a textile. It is also defined as ultimate breaking strength and is the maximum force a textile fiber, yarn, or fabric can bear without breakage.

2.3.2 Color Fastness to Light

Colorfastness to Light testing is an accelerated method that assesses the fabric’s ability to resist fading or other color degradation when exposed to a Xenon Arc light. Colorfastness to light testing is important in determining how well a textile will hold up to sunlight exposure over time. A blue scale is used to determine the color change.

2.3.3 Color Fastness to Perspiration

This test is used to assess the change in color of the fabric when exposed to perspiration.

2.3.4 Color Fastness to Wash

This method is used for assessing the resistance of the color of the dyed fabric of wash in water with soap and detergent.

2.3.5 Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions

For each, sample the test is run in triplicate and compared to a positive control, a negative control, and an inoculum control. The method consists of the selection and analysis of plastic samples to be placed in sealed fermentation vessels filled with a required amount of inoculum derived from a mix of composted solids and active wastewater treatment plant sludge.

The percentage of biodegradability is obtained by determining the percentage of carbon converted to carbon in the gas phase (CH_4 and CO_2).

2.3.6 Testing Planning

The scope of the tests was determined according to the scheme shown in Table 2.

Table 2. Testing planning

Process	Sample Fabric	Test
Scouring	Scoured standard	Tensile Strength and elongation at break
	Scoured biodegradable	Color Fastness to light
Bleaching	Bleached standard	Tensile Strength and elongation at break
	Bleached biodegradable	Color Fastness to light
Dyeing	Dyed standard	Color Fastness to Perspiration
	Dyed biodegradable	Color Fastness to Wash
		Biodegradation

3. Results

3.1 Tensile and Elongation at Break

By observing the graphic results in Figures 3, 4 and 5, with a 95% confidence interval, it was noted that the bleached sample presents a difference of average values of the elongation and the tensile strength between standard and biodegradable yarn. While scoured sample presents a difference in average values of the elongation and the tenacity between standard and biodegradable yarn. Despite the ANOVA statistical analysis differences, for the manufacturer's process control, the statistical differences are not significant.

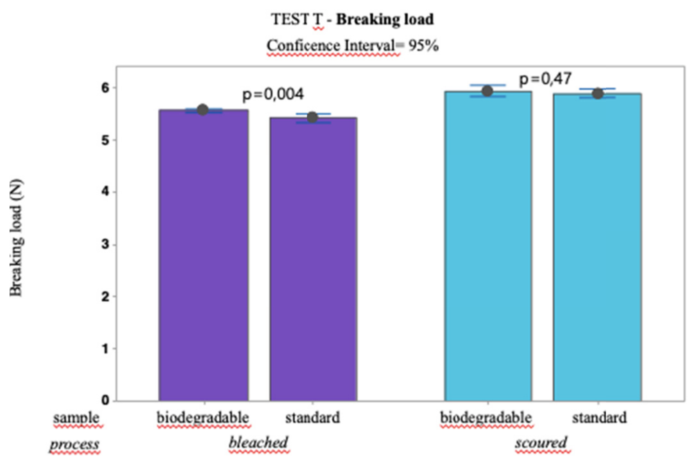

Figure 3. Tensile strength interval plot chart—Minitab

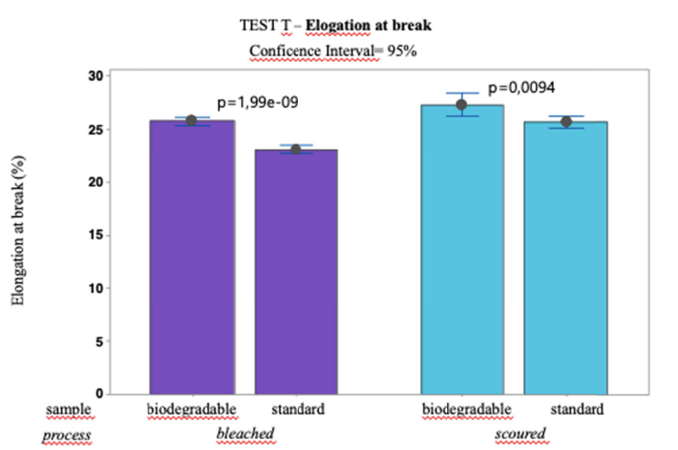

Figure 4. Elongation interval plot chart—Minitab

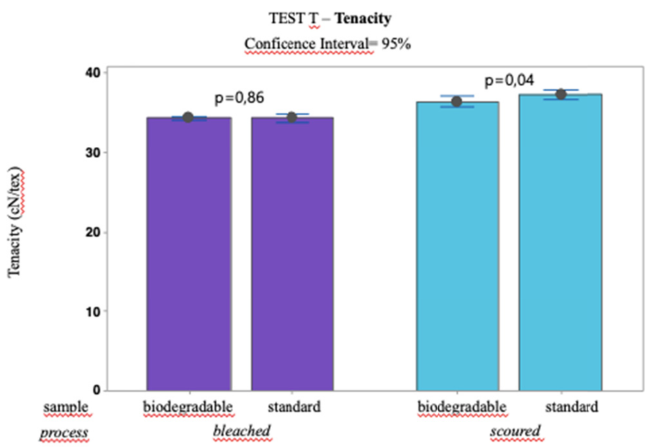

Figure 5. Tenacity interval plot chart—Minitab

Table 3 shows the average values of the properties and their decrease after the bleaching. It has been found that there is a loss of mechanical properties when comparing the scoured and bleached samples. The decrease is slightly higher for standard yarn.

Table 3. Tensile strength and elongation at the break—comparison of the mechanical properties

	Standard		
	Tensile Strength (N)	Elongation (%)	Tenacity (cN/tex)
Scouring	5.91	25.75	37.42
Bleaching	5.44	23.17	34.43
Loss	-9%	-11%	-9%
	Biodegradable		
	Tensile Strength (N)	Elongation (%)	Tenacity (cN/tex)
Scouring	5.96	27.42	36.55
Bleaching	5.59	25.83	34.47
Loss	-7%	-6%	-6%

By analyzing the values obtained in the tests, Table 3, it can be stated that the tensile strength, tenacity, and elongation of the biodegradable polyamide yarn are like that of the standard polyamide yarn. It should be noted that the bleached biodegradable yarn has 11.5% elongation greater than standard yarn.

3.2 Color Fastness to Light

After bleaching, yellowing of the samples occurs, for both standard yarn and biodegradable yarn, as shown in Table 4 with the light fastness test scores. Although the biodegradable yarn has one more point in note (2), both results in practice are poor.

Table 4. Color fastness to light results after scouring process

Scouring	
Standard polyamide	Biodegradable polyamide
7	7

Table 5. Color fastness to light results after bleaching process

Bleaching	
Standard polyamide	Biodegradable polyamide
1	2

3.3 Color Fastness to Perspiration

For both yarns, normal and biodegradable, the color fastness to perspiration score shown in Tables 6 and 7 is the same. The samples had good results on the colorfastness to perspiration test, both alkaline and acidic.

Table 6. Color fastness to perspiration after dyeing process (standard samples)

Standard polyamide		
	Color Staining	Color Change
Color fastness to acidic perspiration	5	5
Color fastness to alkaline perspiration	4/5	5

Table 7. Color fastness to perspiration after dyeing process (biodegradable samples)

Biodegradable polyamide		
	Color Staining	Color Change
Color fastness to acidic perspiration	5	5
Color fastness to alkaline perspiration	4/5	5

3.4 Color Fastness to Wash

Polyamide standard and biodegradable obtained the same results of colorfastness to washing showed in Table 8. Both presented excellent results.

Table 8. Color fastness to wash after dyeing process (standard and biodegradable samples)

		Color Staining	**Color Change**
Color fastness to wash (40ºc ISO I)	Standard samples	5	5
	Biodegradable samples	5	5

3.5 Biodegradable Test

Table 9 shows the percentage of biodegradation of normal and biodegradable polyamide samples. Values were measured after 735 days of testing.

Table 1. Biodegradation percentage

	Negative Control	**Positive Control**	**Standard Sample**	**Biodegradable Sample**
Biodegradation Percentage (%)	- 2.4	87.3	4.5	71.3
Adjusted Biodegradation Percentage (%)	- 2.8	100%	5.2	81.7

By analyzing the values presented in Table 9, it can be stated that under the conditions of the test carried out, after 735 days the biodegradable sample degraded 16 times more than the standard sample. Figure 6 shows the biodegradation graphic.

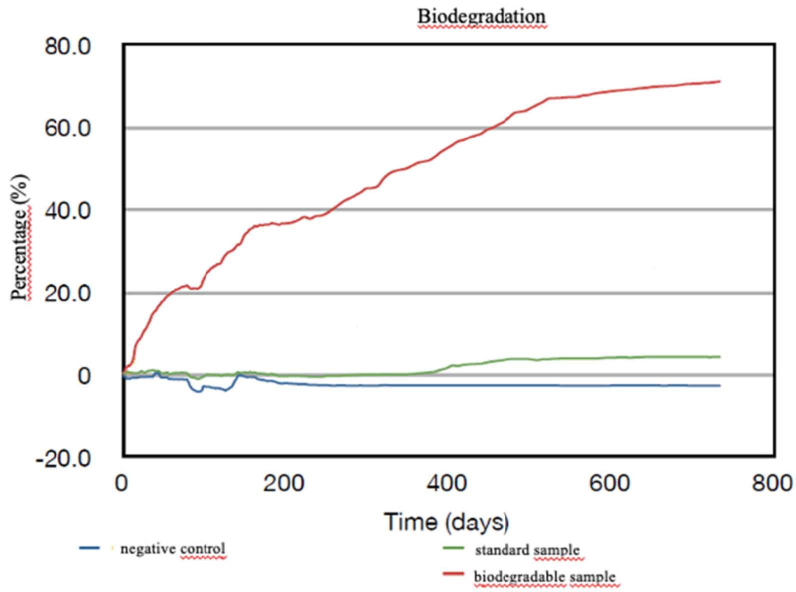

Figure 6. Biodegradation test chart

4. Discussion

Biodegradable polyamide yarn had equivalent results of physical properties when compared to standard polyamide yarn.

The same can be said for the results of the chemical properties. Both biodegradable and standard polyamide samples obtained equivalent results for colorfastness tests.

The percentage of biodegradation of the biodegradable polyamide yarn after 735 days was 81.7% while that of the normal polyamide yarn was 5.2%.

In this way, the research carried out shows that the biodegradable polyamide yarn has a high biodegradation index maintaining the main properties of mechanical resistance and dyeing behavior.

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