THE GENERATION AND AQUATIC BIODEGRADATION OF MICROFIBRES PRODUCED FROM LAUNDERING FABRICS

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ABSTRACT

Microplastics in water bodies have been established as a significant environmental pollutant and can originate from the laundering of clothing. The purpose of this research is to quantify the microfibres generated from the laundering of cotton, polyester, rayon, and polyester-cotton blended knitted fabrics and understand how cotton and rayon spun filaments perform during laundering relative to synthetic polymer filaments. Carefully controlled simulated laundering experiments using an SDL Atlas Launder-Ometer and home laundering experiments using a Whirlpool washing machine and dryer were performed. The effects of fabric type, temperature, detergent use, and different mechanical action on the quantity of microfibres released were assessed. Cotton and rayon fabrics released more microfibres during laundering than polyester, influenced mainly by the presence of detergent solution. The small scale laundering has a greater effect on the microfibre generation due to the intense mechanical action of the metals balls used to simulate accelerated home laundering abrasion. In general, the SDL Launder-Ometer generates 40 times more fibres than a regular washing machine per mass of fabric washed. An additional objective of this study was to characterize how these microfibres biodegrade in an aquatic environment (ISO 14851:1999 2005). Yarns of the materials used in the laundering experiments were exposed to biosolids from a local wastewater treatment plant as an inoculum (30 mg/liter) in the presence of excess oxygen and nutrients at 25 ^{o}C. After 220 days, a reference material of microcrystalline cellulose was degraded by 86% (indicating inoculum effectiveness). The cotton, rayon and polyester had 74%, 61%, and 6% degradation after 220 days. The biodegradation results indicate that natural-based microfibres such as cotton and rayon can be degraded during the wastewater treatment process and in natural aquatic environments. Even though the polyester fabrics released less microfibres than the other fabric types tested, these microfibres are expected to persist in the environment for long periods of time.

Despite the benefits of plastics (longevity, versatility, and raw material availability), issues with their accumulation in the environment are a subject of concern for industries, governments, and communities. In the last century, the utilization and production of plastic products have increased exponentially, bringing many benefits to society (Åström 2016; Thompson, Browne, and Galloway 2010; Thevenon, Carroll, and Sousa 2011). However, less than a half of this plastic end up in landfills or recycled; the rest is still in use or littering the continents and oceans (Rochman et al. 2013). The accumulation of plastics in the environment has become very important in the last 50 years, especially in water sources (Olsen et al. 2004).

Microplastics are small particles below 5 mm in size. Primary microplastics are discharged to the environment in micro size (scrubbing agents, pellets from larger plastics manufacturing processes, etc.) or produced from the abrasion during wear and use of plastic goods such as tires and synthetic textiles; while secondary microplastics are generated in the environment due to the degradation of bigger plastics pieces (Wagner et al. 2014; Moore 2008; Eriksen et al. 2014; Åström 2016; Boucher and Friot 2017; Thevenon, Carroll, and Sousa 2011).

It has been estimated that a minimum of 5.25 trillion plastic particles weighing 270,000 tons are floating in the world's oceans. This represents only 0.1% of the plastic world annual production (Eriksen et al. 2014). Nevertheless, that is not the only place where microplastics have been found. Microplastics are present also in seas, rivers, lakes, and sediments with more abundance in densely-populated areas, with a clear relationship between its abundance and human population density (Browne et al. 2011; Wagner et al. 2014; Miller et al. 2017; GESAMP 2015; Thevenon, Carroll, and Sousa 2011).

Microplastics in water bodies can be the origin of different problems such as aesthetic issues, entanglement and suffocation of marine animals in plastic nets, plastic ingestion by the fauna, adsorption of pollutants organisms in microplastics and transportation in the ecosystems (Moore 2008; Thevenon, Carroll, and Sousa 2011; Wagner et al. 2014; Åström 2016; GESAMP 2015).

Some studies have found that Wastewater Treatment Plants (WWTPs) are a source of microplastics, the composition (polyester, acrylic, etc.) and morphology (fibers instead particles) of these microplastics suggested that they are derived from sewage via the washing of clothes (McCormick et al. 2014; Åström 2016; Browne et al. 2011; Thompson, Browne, and Galloway 2010). According to a study made by the International Union for Conservation of Nature (IUCN) (Boucher and Friot 2017), synthetic textiles are the second biggest source of primary microplastics in the world after plastic pellets.

Several studies have quantified the amount of microfibres released during home laundry. Browne, M. A. et al. observed that polyester garments (blankets, fleeces, and shirts) can shed >1900 fibers per wash. All garments released >100 fibers per liter of effluent, with > 180% more from fleeces (Browne et al. 2011). Åström, L. tested synthetic textile fabrics (polyester, acrylic, and polyamide) in a lab scale washing machine. In this research it was observed that fleece and microfleece fabrics shed the greatest amount of fibers, up to 7360 per m^2 in one wash, indicating that the fabric construction plays a major role in the shedding ability of fabrics (Åström 2016). Likewise, Napper, L et al. did laboratory controlled laundering experiments in home laundering washing machines. It was observed that 6 kg of synthetic materials (polyester, polyester-cotton blend, and acrylic fabrics) could release between 140,000-700,000 fibers per wash (Napper and Thompson 2016). Another study showed that the recovered microfibre mass per garment tested (polyester, acrylic, and polyester-cotton blend) ranged from approximately 0 to 2 g, exceeding 0.3% of the unwashed garment mass (Hartline et al. 2016). The most recent study made on polyester fabrics showed that the use of detergent during home laundering is the aspect that has the greater effect on the generation of microfibres, 75% more microfibres were released when using detergent (Hernandez, Nowack, and Mitrano 2017). Despite the fact that most of the studies have been focused on microfibres released during the washing cycle, one study indicated that the tumble drying cycle released 3.5 times more microfibres than during washing (Pirc et al. 2016). This result coincided with the fact that an important amount of synthetic and natural fibers was collected from the atmosphere in Paris close to the Seine River (2-355 particles/m^2/day) (Dris et al. 2016).

Even though several studies have focused on the quantification of microfibres released from home laundering, the results reported differ due to the different washing protocols used and restrictions presented in home laundering scale experiments. In most cases, different fabrics constructions and textile goods were evaluated leading to an unfair comparison. Moreover, despite all the efforts made with synthetic fibers, a comprehensive study has not been completed to answer the question of cellulosic microfibres being removed during regular consumer washing of cotton and rayon fabrics (Boyter Jr. 2016). In terms of microfibre size distributions, the analysis have been made using methods that rely on the accuracy of an image analysis software and the person that processes pictures taken with an optical microscope and non-representative sample sizes. On the other hand, little is known about the biodegradation of the textile fibers in aquatic environments. Most studies have been focused on biodegradable and synthetic polymers intended for packaging applications in composting environments (Lešinský, Fritz, and Braun 2005; Lucas et al. 2008; Karamanlioglu and Robson 2013; Eubeler et al. 2009; Eubeler, Bernhard, and Knepper 2010; Pagga, Beimborn, and Yamamoto 1996; Starnecker and Menner 1996). This research provides a picture of how different types of fabric material with the same type of knitted fabric structure produce microfibers and the rates at which these materials degrade in water bodies.

MICROFIBRE GENERATION DURING SMALL SCALE ACCELERATED LAUNDERING

The weft knitted interlock fabrics were provided by Cotton Incorporated, four different fabrics with no finishing were used for the laundering experiments; 100% cotton, 100% rayon, 100% polyester, and 50%/50% polyester/cotton.

For the small scale laundering experiments performed in the SDL Atlas Launder-Ometer, the effect of fabric type, temperature, and detergent use were assessed. Normal washing cycles (16 min) were performed at 25 ℃ and 44 ℃ with deionized water (DI) and detergent solution, 1.47g of the 2003 AATCC Standard Reference Liquid Laundry Detergent with optical brightener (AATCC Monograph 2-2005 2017) in 1 litre of DI water (AATCC Test Method 135-2015 2017). The SDL Atlas Launder-Ometer canisters have 550 ml capacity and were filled with 150 ml of DI water or detergent solution, 25 metal balls, and the pre-cleaned fabric sample (4in*4in with secured edges). Two different analyses were made with the water collected after the washing cycles: quantification of the mass of microfibres recovered on a filter paper after filtration and the determination of the count and size of the microfibres in the laundering water using an OpTest Fiber Quality Analyzer (FQA) capable of measuring 1000's of fibers quickly and automatically.

In Figure 1 the effect of detergent use on gravimetric analysis is presented for the second wash (after the cleaning cycle) at 44 ℃. There was a significant increase in the microfibre mass recovered throughout filtration when detergent solution was used for all fabric types.

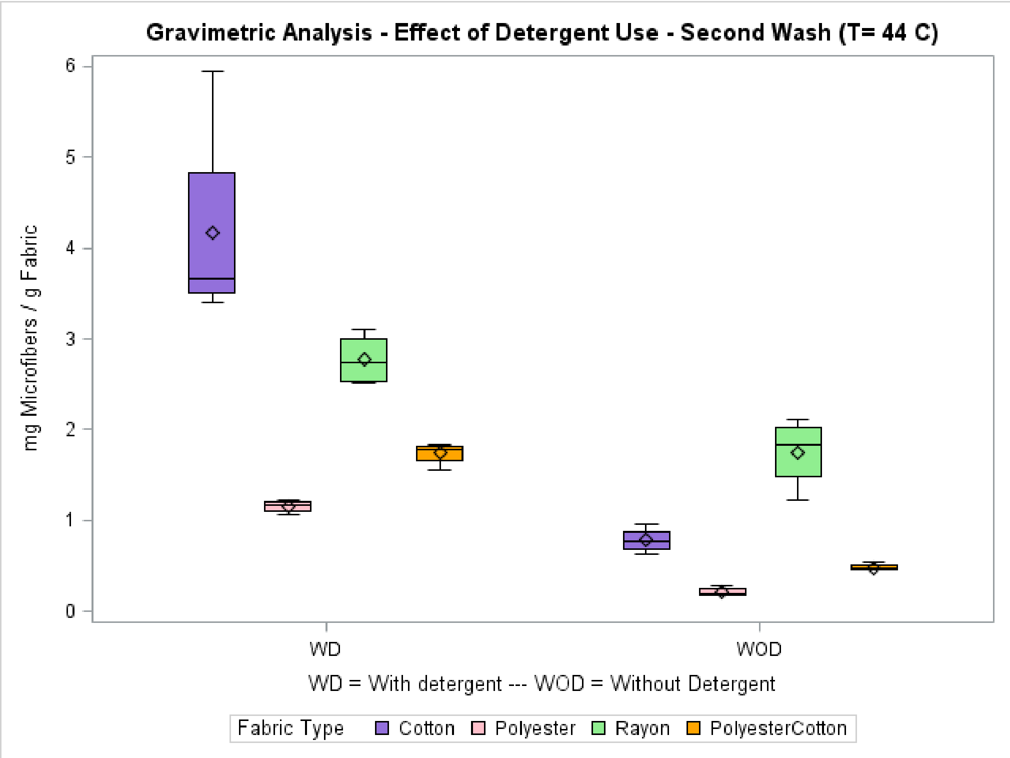
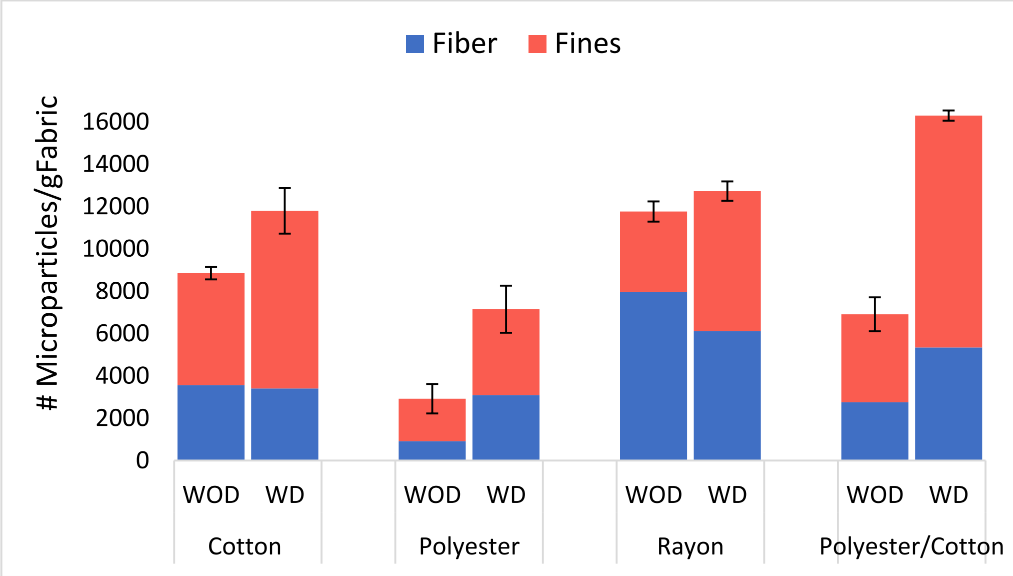


Figure 1. Gravimetric Analysis of Microfibres in Wash Filtrate Using a SDL Atlas Launder-Ometer Accelerated Laundering– Effect of Detergent Use (Second Wash, T=44 °C).

In the gravimetric analysis (Figure 1), rayon, cotton, and polyester/cotton fabrics released significantly more microfibres than polyester fabrics and polyester/cotton blended fabrics.

Microfibres quantification using the FQA showed the trends in the amount of microparticles (microfibres and fines) released during laundering to be in agreement with the gravimetric analysis and was generally influenced significantly by the presence of detergent, except for rayon fabrics (cf. Figure 2).



Fibers Length= 0.2mm - 10mm **Fines Length=** 0.025mm - 0.2mm

Figure 2. FQA Analysis in Wash Filtrate Using a SDL Atlas Launder-Ometer Accelerated Laundering– Effect of Detergent Use (T=44 °C).

These results support the findings of previous studies; the surfactant presence helps in the mobilization/release process of the broken fibres from the fabric surface or network to the washing solution (Hernandez, Nowack, and Mitrano 2017; Napper and Thompson 2016).

The effect of temperature on the microfibres generation was studied in the presence of deionized water and detergent solution. In all scenarios, there was not a significant difference when increasing temperature (25 and 44 °C, data not shown). This lack of temperature dependence has been previously reported, even at extended washing times (Hernandez, Nowack, and Mitrano 2017; Napper and Thompson 2016).

In accordance with the gravimetric results, the quantification of microfibres obtained with the Fiber Quality Analyzer in Figure 2 showed that fabrics made of cotton, rayon, and polyester/cotton released more microfibres than fabrics made of polyester. In contrast to the results of the gravimetric analysis, there was not a clear relation between cotton, rayon, and polyester/cotton fabrics at the different conditions studied. In all the experiments made, the fines generation had the same or higher magnitude than the fibres released, except for rayon when washed with deionized water.

In summary, natural-based fabrics released more microfibres (2-4 mg/g fabric) during small scale laundering than polyester (0.25-0.5 mg/g fabric). Moreover, the Fiber Quality Analyzer detected 5000 to 15000 microfibres/g fabric washed. The detergent use causes more microfibers to be released from fabrics during laundering and the influence of temperature is not significant.

MICROFIBRE GENERATION DURING HOME LAUNDERING

After carefully cleaning the washing machine and the dryer and preparing the fabrics (4 Lb. of each fabric sample with the edges secured, large load), the washing (Table I) and drying (Table II) cycles were performed. Each fabric was subjected to 3 consecutive washing/drying cycles. The microfibres generated during washing were collected with a nylon mesh filtering screen of 20 µm Sefar 03-20/14 that was previously connected to the outlet pipe of the washing machine. For the dryer, the microfibres were collected in the front venting using the same type of nylon mesh.

Table I. Washing Cycle (Washing Machine Model WTW57005WO).

Temperature Control	Program 8 (115 F for wash cycle and 80 F for rinse cycle).
Load Size	Large (AATCC Test Method 135-2015 2017)
ExtraRinse	Off
Fabric Select	Normal
Cycle	Regular 10
Detergent Dosage	100 g of 2003 AATCC Standard Liquid Detergent (AATCC Test Method 135-2015 2017)

Table II. Drying Cycle (Dryer Whirlpool Model WED57005WO).

Cycle	Timed Drying
Temperature	High (Heavy)
Wrinkle Shield	OFF
Time	60 min

The samples collected during washing on the nylon mesh were re-dispersed in water prior to further analysis. The microfibres generated during the drying cycle were collected in a 2-gallon plastic container, washing collected fibres from the nylon mesh installed in the front venting of the dryer. The water was filtered, some fibres were collected for FQA, and the filter paper was dried and weighed.

In the home laundering experiments, the drying cycle generates more microfibres than the washing cycle for all fabrics (cf. Figure 3 and Figure 4). In general the amount of microfibres generated by washing and drying are correlated. The amount of microfibres generated reduces from the first washing and drying cycle to the subsequent cycles. In addition, the difference between drying and washing in microfibres generation is more significant for Rayon and Cotton than for polyester. In agreement with the small scale experiments, fabrics made of natural-based fibers released considerably more microfibres than polyester during laundering as well as during drying (cf. Figure 3 and Figure 4).

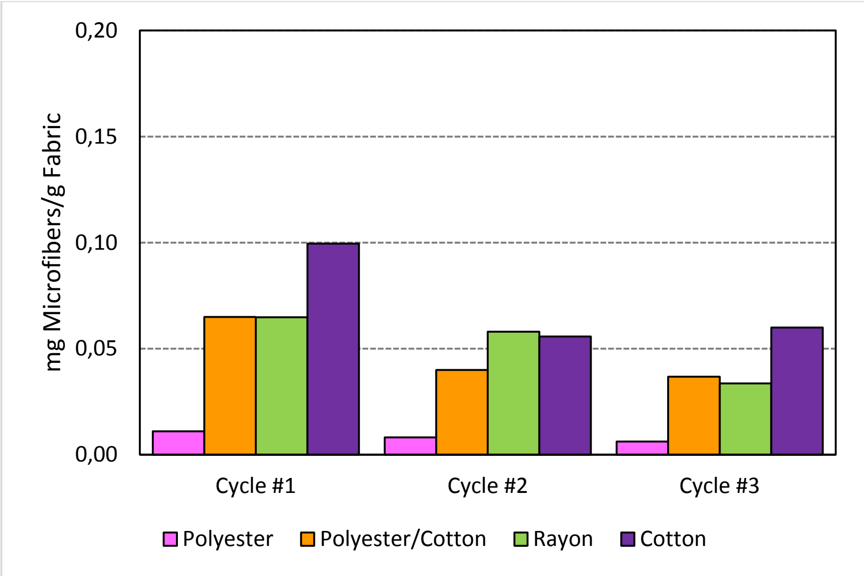


Figure 3. Microfibres Generated during Washing – Home Laundering.

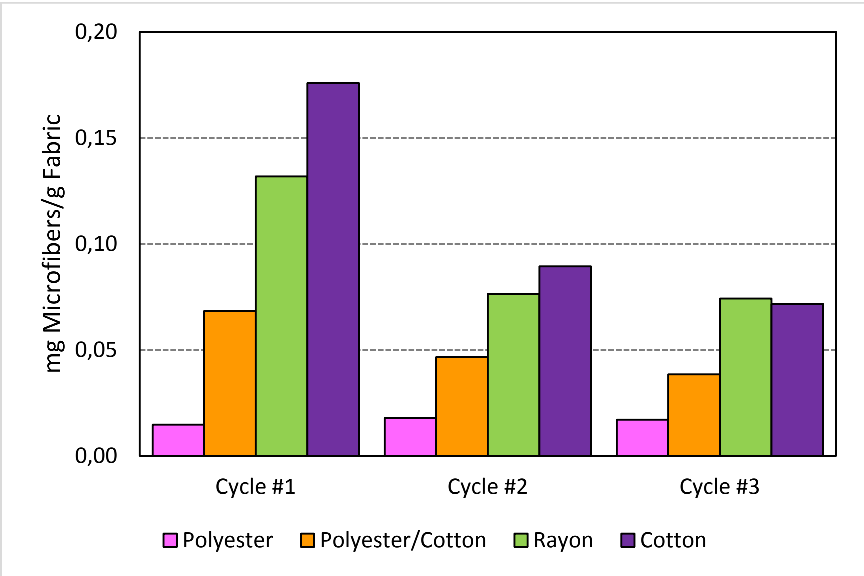


Figure 4. Microfibres Generated during Drying – Home Laundering.

The shedding capacity of the fabrics depends mainly on the fuzz formation (in this process the microfibres are loosened from yarn and fabric structure) and how readily these fibres break by the mechanical action of the washing machine (S. Okubayashi et al. 2005; S. and B. T. Okubayashi 2005; Geology 1966). In order to understand how the fiber type affects the microfibre generation during laundering, the tensile properties of the yarns used to knit the fabrics were evaluated in the dry and wet state according to the Tensile Properties of Yarns by the single-strand Method

(ASTM D2256 2015). The tensile tester MTS Q Test 5 was used to perform the tests in *1 Grab 10 inch GL* mode (data not shown). The polyester yarns presented the highest breaking load, about two times greater than the cotton and rayon in both wet and dry states, explaining to some extent why polyester fabrics released less microfibres than the other fabrics studied.

Due to the high intensity mechanical action of the metal balls in the small scale accelerated laundering experiments, more microfibres are generated per weight of fabric washed than in the home laundering washing experiments. For example, cotton generated 4 mg of microfibres per g of fabric during the accelerated laundering in the Launder-Ometer versus 0.1 mg of microfibres per g of fabrics during home laundering experiments.

Regarding the size of the microfibres, in the home laundering experiments longer microfibres were obtained than in the accelerated laundering experiments. Rayon fabrics generated the longest fibres, followed by cotton, polyester/cotton, and polyester, respectively, for both the accelerated and the home laundering experiments.

AQUATIC BIODEGRADATION OF YARNS

The microfibre fate assessment after laundering is not complete without the understanding of the behavior of these fibers in aquatic environments. The aquatic biodegradation of the yarns was evaluated using a standard method to assess the ultimate aerobic biodegradability of plastic materials in aqueous mediums (ISO 14851:1999 2005). It was assumed that the yarns and the microfibres would have similar degradation behavior.

The experiment started on June 13, 2017 with the preparation of the flask without the test material or the reference material. The bottles were incubated for one week to achieve the inoculum stabilization. Then, the corresponding sample was added to each bottle to start the biodegradation on June 20, 2017. The results presented herein correspond to the first 220 days of the experiment, after the sample addition. The last measurement reported herein was made on January 26, 2018. As of the time of this report the experiments were continuing.

The reference material (microcrystalline cellulose) reached 85.75% degradation (cf. Figure 5). This fact indicates that the inoculum is working effectively. The biodegradation curves presented could change if at the end of the experiment if there are any nitrification interferences detected as important. However, according to nitrogen content of the samples, a considerable oxygen consumption due to nitrification is not expected.

After 220 days of reaction, the BOD of the Blank was 10.87 mg/l (it does not exceed 60 mg/l), while the percentage of biodegradability in the materials tested was:

- 100% Cotton Yarns (73.95±2.48%),
- 100% Rayon Yarns (61.22±3.41%),
- 50%/50% Polyester/Cotton Yarns (43.06±1.85%),
- 100% Polyester Yarns (5.74±0.73%).

The biodegradation curves are presented in Figure 5. The biodegradation rate is higher for the microcrystalline cellulose followed by cotton, rayon, and polyester/cotton yarns, respectively. From these results, it can be seen that polyester was not appreciably biodegradable under the test conditions. In addition, the 50/50% polyester/cotton yarns have approached the plateau phase at a level under 50% of biodegradation. It is expected that the cotton in the blend was degrading. The cotton, rayon, and reference material are starting to approach the plateau phase.

These results are in line with the expected behavior; natural fibers are expected to be biodegradable. Even though natural based fabrics are releasing more microfibres per gram of fabric in laundering, they have the potential to be biodegraded in the wastewater treatment process and aquatic environments.

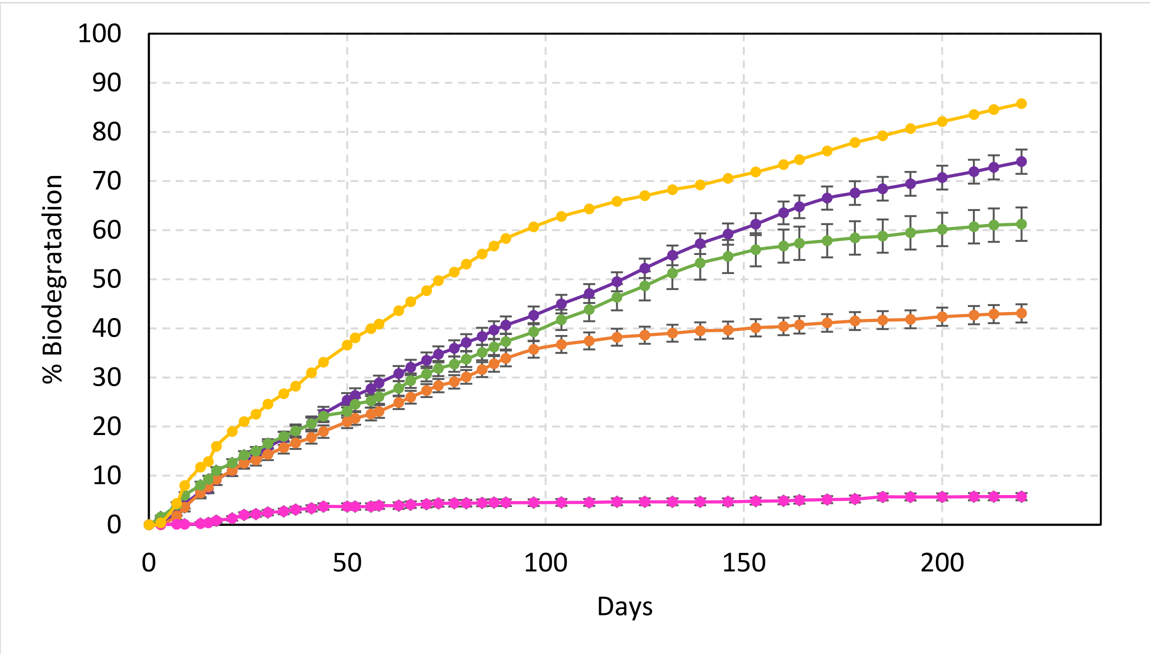


Figure 5. Biodegradation Results of Yarns in an aerobic aquatic environment (in decreasing extent of degradation: Microcrystalline cellulose, Cotton, Rayon, 50%/50% Polyester/Cotton, 100% Polyester).

Future research is ongoing to define better the fate of these microparticles in a wastewater treatment plant (mechanical screening, flocculation, activated sludge) as well as biodegradability experiments in fresh and coastal waters.

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