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Rayon fibre rope: A biodegradable alternative for marine use?

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ABSTRACT

Rayon fibres are well-known materials that were primarily utilised as reinforcement in tyres. Today these materials are perceived as a promising substitute for synthetic fibres, exhibiting good mechanical characteristics and biodegradation in many environments. This paper investigates their potential use for marine structures. It first describes the tensile properties of the fibres and their derived yarns and ropes. These properties are then monitored during seawater ageing and the ultimate biodegradation is characterised by respirometry tests. Both material scales demonstrate rapid degradation rates under biotic conditions (90 % strength reduction after 2 weeks for yarns and 6 months for small ropes) and a relative stability in abiotic conditions. Additionally, the fibres show rapid bio-assimilation rates. The rope construction is demonstrated to have a significant impact on the degradation kinetics, suggesting possible strategies to enhance durability. The results indicate that these rayon fibre ropes may offer an attractive alternative to synthetic fibre ropes to reduce impact where there is a high risk of rope loss at sea.

1. Introduction

Regenerated cellulose fibres were the first artificial fibres used by the textile and clothing industry, introduced in the 1850's (Chen, 2015; Woodings, 2001). There are four main types of fibres derived from cellulose: viscose, lyocell, cupro and acetate. The classification of these different fibres is based on the production method. Viscose rayon is the principal regenerated cellulose fibre produced today and represented >93 % of the market in 2015 (Chen, 2015). Viscose rayon fibres are produced by making alkaline cellulose and reacting with carbon disulphide to produce cellulose xanthate (Chen, 2015; Manian et al., 2018; Woodings, 2001).

The polymer chains in cellulosic fibres consist of $\beta(1/4)$ -linked anhydrous p-glucose units. These are believed to lie alongside each other forming strand- or thread-like clusters, which are described as a "fringed fibrillar structure" (Hearle, 2001). The residues obtained after hydrolysis and ultrasonication of cellulosic fibres, when examined under a transmission electron microscope, resemble aggregates of beaded stringlike structures, or fibrils, and this supports the above model (Lenz et al., 1988). The arrangement of polymer chains in the fibrils range from highly ordered ("crystalline") domains to highly disordered ("noncrystalline" or "amorphous") domains. In native cellulosic, only the adjacent chains in sheets are linked (Northolt, 2001), and the crystalline structure is labeled "Cellulose I." In regenerated cellulosic, each polymer chain is linked by hydrogen bonds to four others in the sheets and the structure is labeled "Cellulose II."

The internal fibre structure at the nanometric scale is fundamental in determining the mechanical properties and depends on the manufacturing conditions (drawing operations for example) (Sawada et al., 2021). Morphological studies indicate a skin/core structure, analysed by X-ray diffraction of individual fibres and electronic diffraction on transverse longitudinal sections of fibres (Müller et al., 2000). Both techniques provide diffraction maps which reveal that (1) the cellulose molecules are much better aligned in the skin than in the core, and (2) that there is no significant difference between the skin and core in terms of the mean crystal size or crystallinity.

Many factors can influence the tensile behaviour of these fibres and their derived braids. The amount of water absorbed by viscose fibres has been observed to strongly affect their tensile properties (Mendes et al., 2021; Woodings, 2001). Mendes et al. (Mendes et al., 2021) presented a set of data comparing properties in both wet and dry conditions. The presence of water led to a reduction in tensile strength and an increase in failure strain. Furthermore, the tensile behaviour of braided fibres is of course related to the fibre properties but the hierarchical rope

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constructions will also contribute to the measured response (Leech, 2003). Important parameters include the rope geometry (lower strength efficiency with increasing rope diameter) (Del Vecchio, 1992), the braid angle, and the levels of twist in the constituents (Amaniampong and Burgoyne, 1995; Davies et al., 2016; Rao and Farris, 2000). A multi-scale approach is therefore necessary in order to understand degradation mechanisms (characterisation at the fibres, yarn and braid levels).

Natural fibres such as cotton and flax can biodegrade at the end of their lifecycle. This property is highly important to mitigate the global plastic pollution crisis (Narancic and O'Connor, 2019; Shen et al., 2020). Biodegradation is the process resulting in the decomposition of organic materials into small molecules by microorganisms (Laycock et al., 2017). It contributes to the biogeochemical cycles and controls the environmental impact during the disposal of polymers. Biodegradation is a complex phenomenon that involves a large number of factors. The most important is the microorganism communities present in the environment of degradation: specific enzymes are necessary to degrade each type of polymer (Laycock et al., 2017; Pathak and Navneet, 2017). The interactions between microorganisms and polymer materials depend on the chemical and physical structures of polymers (Kim et al., 2022; Park et al., 2004). Various environmental factors influence the biodegradation processes (Erdal and Hakkarainen, 2022; Harrison et al., 2018; Laycock et al., 2017; Wang et al., 2021). Depending on the environment, the degradation can be solely biodegradation or a combination of several processes, such as chemical and enzymatic hydrolysis, photodegradation, and oxidation. For regenerated cellulose fibres and more specifically for viscose fibres this has been investigated and demonstrated in a large number of environments (Erdal and Hakkarainen, 2022; Kim et al., 2022) including soil (Park et al., 2004), tap water (Kwon et al., 2021; Zambrano et al., 2020) and seawater (Royer et al., 2023). More generally, the expected end-of-life options for biodegradable plastics include industrial and home compost, soil, wastewater, freshwater, and seawater. The degradation rate of materials in different man-made or natural environments will depend on the combination of the material/environment, but it is generally fastest in industrial compost under controlled conditions, high temperature, humidity, with a high concentration of microorganisms; in freshwater and saltwater, the average temperatures and concentrations of microorganisms are much lower (Erdal and Hakkarainen, 2022). For example, Park et al. (Park et al., 2004) studied tests in soil: They showed that biodegradability decreased in the following order: rayon > cotton \gg acetate. Rayon fibres, which have a low crystallinity and a low degree of orientation, showed the highest biodegradability in most cases.

Environmental impact has become a critical parameter for industrial applications and it is no longer acceptable to claim a green textile image simply because the fibres are bio-sourced. The manufacture of cellulose fibres from renewable resources, but obtained using a chemical process, requires an analysis of each step of the production. This subject is examined in several publications (Kim et al., 2022; Muthu et al., 2012; Shen et al., 2010). Analysis of these papers requires caution as their objectives, application sectors and methodologies vary. The functional unit, the renewable resource, the production technology and location all affect the analysis. For example, the functional units of these papers consider either manufacture of viscose fibres (cradle to factory gate) (Kim et al., 2022; Shen et al., 2010) or the complete life cycle (cradle to tomb) (Muthu et al., 2012). The impacts have then been either compared between different types of regenerated cellulose (rayon, cellulose acetate, and lyocell fibres) (Kim et al., 2022), or between natural and synthetic fibres (Viscose, Modal, Tencel, cotton, PET and PP) (Shen et al., 2010) or a wider range (conventional cotton, organic cotton, wool, flax, polyester, nylon 6, nylon 66, polypropylene, acrylic and viscose) (Muthu et al., 2012).

The results of Kim et al. (Kim et al., 2022) indicated that the environmental impact of viscose rayon was higher than those of natural cellulose fibres, including cotton and flax, and similar to those of synthetic fibres, such as nylon, PET, and polypropylene (PP), in terms of damage to human health, ecosystem quality, and resources. Ideally, in order to compare these fibres it would be necessary to have all the production details (entrants and exits of materials and energy), and to compare them on an identical basis for a given application, so that the fibre production cycle could be optimized (Shabbir and Mohammad, 2017).

To date, studies on marine biodegradation have highlighted the potential use of regenerated cellulose as an alternative to conventional plastics in textile applications (Royer et al., 2023). These applications represent a significant source of marine pollution, either due to inadequate waste management practices or the release of unfiltered fibres into wastewater from washing machines (Boucher and Friot, 2017; Browne et al., 2011; McCormick et al., 2014). However, the accumulation of plastic debris in the oceans is also a consequence of the loss of structural materials at sea, including fishing gear and ropes (Morales-Caselles et al., 2021). An illustrative example is Fish aggregating devices (FADs) (Maufroy et al., 2017). These floating structures are employed extensively to attract tuna fish, and 100,000 FADs are estimated to be deployed each year (Gershman et al., 2015); most of these end their lives in the deep-sea after one year. In order to mitigate the environmental impact of these applications at the end of their life cycle, two key factors must be addressed for the potential alternative material:

- Ensuring that it is biodegradable in environments where it may be lost, including not only coastal marine environments but also the deep sea, depending on the application.
- Ensuring that the material possesses the requisite mechanical properties and durability to maintain its functions in service. For FAD tails for example, lengths of rope up to 60 m long which limit drift, this entails maintaining their mechanical integrity.

Different materials have already been investigated for FADs (Murua et al., 2023) and fishing gear (Le Gué et al., 2024, 2023). But to the author's knowledge the state of knowledge on these two key factors is very limited for regenerated cellulose and more specifically for rayon fibres. A few studies have demonstrated the biodegradation of viscose (Royer et al., 2021) and rayon (Royer et al., 2023) in coastal marine environments. And regarding the second factor, the literature is even more limited. The use of ropes in marine environments is a complex topic requiring extensive characterisation. This encompasses the examination of material deterioration under marine conditions, the assessment of mechanical properties during immersion and the analysis of the impact of rope structure on both aforementioned aspects. To our knowledge no studies have addressed these issues for rayon or, more broadly, regenerated cellulose fibres.

This paper will focus on rayon fibres commercialized by Cordenka. Their process involves dissolving natural cellulose paste in a sodium hydroxide and carbon disulfide solution (Meredith et al., 2013). This is then matured, filtered, degassed and extruded in a bath to produce fibres and the cellulose is regenerated. The product is used to reinforce tyres as it shows good adherence to rubber and excellent thermal and dimensional stability. The rayon was used in the form of continuous fibres, yarns and braided ropes. First the impact of the structure on the initial mechanical properties is investigated for the three scales studied. Subsequently the changes in the mechanical properties of the yarn and the rope through ageing in coastal environments are quantified. Finally, the biodegradation of these fibres is assessed via respirometry, to verify that true biodegradation occurs in coastal environments. Based on the results from these analyses the potential of cellulose fibre ropes for marine applications is discussed.

2. Materials & methods

2.1. Materials

Rayon materials studied in this article were provided by Cordenka.

Three different structures made of viscose rayon are studied: elementary fibre, yarn and 8 yarns braided together to produce ropes (all with the same fibre). Coating information was not provided nor studied in this work. Initial structural and mechanical properties provided by the supplier are given in Table 1, the materials, developed to increase the stiffness and decrease the shrinkage of the yarn, are not yet commercially available.

2.2. Ageing

Yarn and rope samples were immersed in tanks filled with natural seawater, pumped from the Brest estuary (France). Two conditions were investigated. In the first, the seawater is continuously renewed and maintained at 15 °C, hence nutrients and microorganisms are representative of natural biodegradation. In the second, samples are immersed in sterilized natural seawater without renewal at the same temperature of 15 °C. Sterilization was performed in an autoclave (20 min at 121 °C) and antibiotics, chloramphenicol and penicillin G, were added to the seawater (Chamley et al., 2024).

These two conditions are respectively referred to as biotic and abiotic conditions hereafter in this paper. Samples were removed for testing after one, two, four, six, eight and twelve weeks (respectively referred to as W1, W2, ..., W12) for the yarn and after one, three and six months of immersion for the rope (referred to as M1, M3 and M6). After each sampling, samples (yarn and ropes) were stored in a controlled environment (21 °C, 50 % R.H.) until their weight stabilised. Drying at higher temperature was avoided to ensure that no degradation occurred during this desorption step. The ageing of the samples is then characterised using multiple methods described in the following sections.

2.3. Structural characterisation

For the measurement of the density of the materials, an AccuPycTM II gas pycnometer was used. This equipment makes 30 measurements on each sample (see Le Gall et al., 2018 for more detail on the protocol). Three samples of each structure were tested.

Linear density was calculated by weighing 4 m of each material with an electronic balance (precision of 10^{-3} g) after 24 h in a controlled environment (21 °C, 50 % RH).

To provide information on the braided construction (yarn number, braiding angle), X-ray tomography was performed with a Tomoscope[™] XS Plus 200 kV tomograph from WERTH.

2.4. Mechanical testing

2.4.1. Elementary fibres

The tensile properties of the elementary fibre were characterised using a Dia-Stron[™] system. Results from 50 valid tests (failure in the gauge length) were analysed for each test series, during each test the fibre cross-section was measured at 10 points by a rotating laser. The tests were carried out at a speed of 1 mm/min, at 21 °C, and at a relative humidity between 50 % and 70 %. Nominal stress was then calculated using the mean cross-section determined during the test and the Young modulus was determined via linear regression on the initial part of the stress strain curve (AFNOR, 2015).

2.4.2. Yarn and rope

The tensile properties of the rayon yarn and the ropes were

Та	ble	1

Properties provided by fibre :	supplier.
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	Braided rope	Yarn
Linear density (tex)	1890	184
Breaking strength (N)	531 ± 26	-
Elongation at break (%)	16.5 ± 0.8	-

characterised with an Instron[™] test machine equipped with a 10 kN load cell for the rope and a 500 N cell for the yarn. Two different types of pneumatic grips, designed for use in rope and yarn testing, were employed. Tests were conducted at a displacement speed of 20 mm/min, with a free length between grips of 300 mm. Strain measurements are obtained through the use of two markers and two cameras (Chailleux and Davies, 2005). All tests were performed in a controlled environment at a temperature of 21 °C and a relative humidity of 50 %. For each condition, five replicates were characterised.

Results from tests on other polymer fibre low twist yarns are also shown for comparison. Two petro-sourced yarns, polyamide 6 (PA6) and polyethylene terephthalate (PET) were tested in other projects; these are yarns used for offshore mooring lines, with linear weights of 0.19 g.m⁻¹ and 0.11 g.m⁻¹ respectively. Results for biosourced, compostable poly lactic acid (PLA, 0.04 g.m⁻¹) and biodegradable poly butylene succinate (PBS 2.5 g.m⁻¹) yarns are also shown. These were all tested on the same test frame as the rayon yarns, under displacement control (50 mm.mn⁻¹) with a 500 N load cell and the optical non-contact extensometer mentioned above to measure strain.

2.5. Surface characterisation

Surface observations were made using a FEI Quanta[™] 200 Scanning Electron Microscope (SEM). Prior to observation, each sample was coated with a 60 % gold and 40 % palladium coating, to avoid surface charging.

2.6. Respirometry

Respirometry tests were conducted in accordance with the ISO 19679 standard (ISO, 2020) over a period of 214 days. These tests evaluate the assimilation of the tested materials by a set of microorganisms. The fibres were placed in an incubation medium comprising a solid-liquid mixture of natural seawater and freshly collected intertidal sand on the coast near Lorient (Brittany, France) in a first glass. During the experimentation, the CO₂ emitted by the microorganisms is captured in a second flask via reaction (1) in a NaOH solution of known concentration. Once a week, the NaOH solution is sampled, analysed, and replaced. The Na₂CO₃ is precipitated using an excess solution of BaCl₂ (reaction 2) and the NaOH remaining is titrated with HCl using thymolphthalein as an indicator (reaction 3). The CO₂ consumed in reaction (1) can therefore be determined and the biodegradation rate is calculated via Eq. (4). The initial carbon content of the materials was determined through a preliminary elemental analysis (pyrolysis followed by gas chromatography), and this value is denoted as mCO₂(theoretical) in the following equations.

The test is conducted in a closed-circuit respirometer (Fig. 1). The oxygen necessary to sustain microbial respiration is adequately available in the free volumes of the flasks. The continuous aeration process ensures the proper homogenisation and oxygenation of the incubation medium. The atmosphere within the respirometers is periodically replenished during the course of CO_2 analyses and sensor replacements. To maintain a constant temperature, the respirometers are immersed in a thermostatically controlled water bath set to 20 °C, which simulates near-real environmental conditions. CO_2 emissions are measured weekly through acid-base titration. Special care is taken to prevent any evaporation from the flasks.

$$CO_2 + 2 \operatorname{NaOH} \rightarrow \operatorname{Na_2CO_3} + H_2O \tag{1}$$

$$Na_2CO_3 + BaCO_3 \rightarrow 2 NaCl + BaCO_3$$
⁽²⁾

 $NaOH + thymolphtalein + HCl \rightarrow H_2O + thymolphtalein$ (3)

$$\tau_{degradation} = \frac{mCO_{2}(emitted) - mCO_{2}(witness)}{mCO_{2}(theoretical)} \times 100 \tag{4}$$



Fig. 1. Diagram illustrating the closed-circuit setup. Left flask: CO₂ captured; Right flask: incubation medium.

The behaviour of the materials during the respirometry tests is typically modelled using either the Hill or Boltzmann equations (depending on the form of the curve obtained, Hill for single sigmoid and Boltzmann for double sigmoid) (Deroiné et al., 2015; Salomez et al., 2019).

3. Results

3.1. Initial characterisation

3.1.1. Elementary fibres

The mechanical properties of the tested fibres are summarized in Table 2. The tensile stress curves obtained with fibres extracted from the yarn are plotted in Fig. 2. In this graph, the mean curve and its standard deviations are highlighted. Mechanical properties of the fibres are in agreement with published values on other rayon fibres from Cordenka, with a slightly higher strain and stress at failure (Table 1). Considerable variability between the fibres is observed with coefficients of variation of 22 % and 18 % for stress and strain at failure (Fig. 2).

3.1.2. Yarn and rope

Mechanical properties obtained on the yarn and the rope are summarized in Table 3. Properties are slightly lower than the performance indicated by the supplier for the rope (Table 1). Fig. 3 shows how the mean tensile properties are transferred from the elementary fibres to the yarns and the braided rope. From fibres to yarn to rope the properties drop due to twist but for the rope the strain to break is higher than for



Fig. 2. Tensile curves for rayon fibres extracted from the yarn.

Table 3	
Mechanical properties and linear density of the materials studi	ed.

	Break load (N)	Elongation at break (%)	Break tenacity (mN.tex ⁻¹)	Linear density (tex)
Fibre	$\begin{array}{c} \textbf{0.097} \pm \\ \textbf{0.017} \end{array}$	15.4 ± 2.8	527 ± 92	0.138
Yarn	$\textbf{71.4} \pm \textbf{0.6}$	13.3 ± 0.2	388 ± 3	184
Rope	476 ± 4	20.4 ± 0.6	251 ± 2	1890



Fig. 3. Multiscale comparison: Specific stress - strain curve at three levels.

the yarns due to construction elongation.

Fig. 4 shows details of the rope construction. The tomography images reveal the close packing of fibres in the strands of the braid.

Table 2

Mechanical properties of the elementary fibres. Comparison with the literature on other rayon fibres produced by Cordenka.

Elementary fibre type	D (µm)	Std	Modulus (GPa)	Std	Stress at failure (MPa)	Std	Strain at failure (%)	Std	Reference
Fibres from yarn	10.4	1.5	21.0	4.8	1171	258	15.7	2,8	
Fibres from rope	10.5	0.6	16.9	6.0	887	202	12.5	3,1	
Twisted fibres			21.1	3.2	797	111	14.0	3,9	Rozite et al., 2012
Non twisted fibres			22.1	2.9	770	67	13.5	4,7	
Sized fibres	14.4	0.5	27.4	4	944	22	10.2	-	Shamsuddin et al., 2016
Unsized fibres	13.5	0.2	28.8	3	943	94	10.4	-	
Cordenka RT 700			20	1	830	60	13.0	2,0	Kalia et al., 2011



Fig. 4. Braided rope images from (A) SEM and (B-D) X-ray tomography. Rope composed of 8 yarns braided together. Each yarn composed of multiple filaments.

3.2. Degradation during laboratory immersion

3.2.1. Yarn

Yarn samples were retrieved after one (W1), two (W2), four (W4), six (W6), eight (W8) and twelve weeks (W12), for a total ageing period of 90 days. The mean tensile curves are plotted in Fig. 5. After two weeks of ageing under biotic conditions, the yarn shows a large drop in mean load and strain at break $(1.2 \pm 0.4 \%, -91 \%)$. A plateau seems to be reached immediately afterwards and the properties remain stable until the end of the experiment (Fig. 6). Under abiotic conditions, the properties of the mean strain at break $(14.7 \pm 0.9 \%, +10 \%)$ observed at the end of the ageing and a slight decrease of the mean load at break observed after 18 days which



Fig. 5. Load – strain curve of yarn samples aged 1–12 weeks (W1-W12) in biotic and abiotic conditions.

then remains stable until the end of the ageing (Fig. 6). This phenomenon could be attributed to a slight plasticisation, which is frequently observed after ageing in water of synthetic fibres (Humeau et al., 2018).

3.2.2. Rope

Rope samples were retrieved at one, three and six months of ageing, the end of the experimentation. For each sampling, three samples were tested and the mean values are plotted in Fig. 7. The tensile tests were highly reproducible (except for M3 in the abiotic condition, see standard deviation in Fig. 8). As for the yarn, samples aged under biotic conditions showed critical degradation.

After six months of ageing, mean load at break (55 \pm 1 N) and mean strain at break (8.2 \pm 1.8 %) decreased by 95 % and 60 % from initial values, respectively. (Fig. 8). Conversely, samples aged in abiotic conditions showed significantly lower degradation. Within the same ageing duration, load at break was reduced by 20 % and mean strain at break was increased by 20 % (24.7 \pm 0.4 %), respectively. (Fig. 8).

3.3. Microscopic observations

SEM observations on aged fibres revealed the presence of surface degradation after one week of ageing under biotic conditions (Fig. 9.D). Initially, these degraded areas were highly localised. After 12 weeks of ageing under the same conditions, the degraded areas became more severe, exhibiting greater degradation depths and affecting larger areas (Fig. 9.B, Fig. 9.E). In contrast, fibres aged under abiotic conditions did not exhibit any changes in surface morphology (Fig. 9.C, Fig. 9.F).

The appearance of these degraded areas is also observed in aged ropes (Fig. 10). After one month of ageing under biotic conditions, degradation is localised to certain fibres and remains shallow (Fig. 10. B). After six months, much more severe degradation is observed, with some fibres being completely degraded, predominantly at the interlacing points (Fig. 10.C, Fig. 10.D). The degradation is superficial, with fibres located at the core of the braid remaining intact, thereby



Fig. 6. Evolution of load and strain at break (%) of yarn samples during the ageing, with respect to the initial value. Standard deviations are represented by shaded areas.



Fig. 7. Evolution of Load – Strain of rope samples aged 1–6 month (M1 – M6) in biotic and abiotic conditions.

preserving the overall structure of the braid. In contrast, ropes aged under abiotic conditions do not exhibit any signs of surface degradation after the same ageing period (Fig. 10.A).

3.4. Respirometry

The respirometry tests conducted in this study show considerable variability, as illustrated by the standard deviations represented by the shaded areas (Fig. 11). Despite this variability, substantial mineralisation was observed in the rayon samples. The mean degree of mineralisation observed in the samples following 200 days of experimentation

was 98 %, with each test demonstrating a degradation rate exceeding 80 % (Table S1). The mineralisation of the rayon was faster and more extensive than that of the reference cellulosic material, which exceeded the critical 60 % threshold after 180 days of testing for all three samples, confirming its validity according to the EN ISO 19679 standard (ISO, 2020). The models applied are effective in describing the results obtained (Fig. 11), and the parametric values are provided in the supplementary material (Table S2).

4. Discussion

4.1. Initial characterisation

First, concerning the elementary fibres tests significant variability was measured (Fig. 2). Bunsell has discussed elementary fibre testing and provided information on variability (Bunsell, 2018). For example, a set of 30 tests on commercial PET fibres resulted in a coefficient of variation (standard deviation as a percentage of mean values) for break strengths of 8 %, which is significantly lower than the values obtained here (17 %). High variabilities in tensile properties are also usually observed for natural fibres; for the latter, numerous factors have been identified (agricultural practices, climatic factors, fibre extraction, and processing). Flax fibres show 30-50 % variability in tensile strength (Baley et al., 2020). The aforementioned studies conducted on rayon fibres demonstrate lower variability (14, 8, 2, 10 and 7.2 %, Table 2). The fibre under examination in this study was a prototype developed for an increased tenacity; therefore, the higher variability could be a consequence of the processing experimentation associated with this development. Twisted samples, extracted from the rope, show a reduction in mechanical properties (-20, -25 and - 20 % respectively for)module, stress and strain failure) (Table 2). This observation differs from those of Rozite et al. (Rozite et al., 2012) on fibres extracted from a twisted yarn. The degree of twist could explain this difference: in the



Fig. 8. Evolution of load and strain at break of rope samples during the ageing, with respect to the initial value. Standard deviations are represented by shaded areas.



Fig. 9. SEM photographs of aged fibres in yarn at two magnifications, upper: x500, lower: x5000. (A, D): 1 week in biotic conditions. (B, E): 12 weeks in biotic conditions. (C, F): 12 weeks in abiotic conditions.



Fig. 10. SEM photographs of aged ropes. (A): 6 months in abiotic conditions. (B): 1 month in biotic conditions. (C-D): 6 months in biotic conditions.



Fig. 11. Biodegradation test of rayon and reference cellulose.

present work on fibres extracted from a rope, the braided structure induces more severe strain concentrations than in the case of a twisted yarn.

The stress-strain curves from tensile tests on regenerated cellulose fibres are non-linear and composed of two parts (Northolt, 1985). There is an initial region, up to around 1 % strain, with a high stiffness, followed by an elastic limit and a second almost linear region with a lower stiffness. The explanations proposed for this behaviour are as follows (Manian et al., 2018):

- Up to the elastic limit the strain results in a re-alignment and orientation of the polymer chains along the tensile axis without breakage of the intermolecular hydrogen bonds so the strain is mainly elastic.
- Beyond that limit plastic strain occurs which is related to hydrogen bond breakage and relative displacements between chains, leading to final failure.

A comparison of the rayon yarn tensile properties with results from tests on common fossil based conventional plastics and other potential marine biodegradable yarns for marine applications is shown in Fig. 12. The properties of the yarn from Cordenka demonstrate a stiffness that is comparable to that of PET and is superior to the other materials tested. The elongation at break is comparable to that of PET and markedly



Fig. 12. Rayon stress-strain behaviour compared to two high tenacity petrosourced yarns used in marine applications, and two other biopolymer candidates tested under similar conditions.

lower than that of PA6 and other materials under consideration (31 % for PLA and 130 % for PBS). However, its tenacity at break (0.39 N. tex^{-1}) is nearly half that of conventional plastics (0.73 and 0.72 N. tex^{-1} for the PET and the PA6 respectively) but considerably higher than that of PLA (0.24 N. tex^{-1}) and PBS (0.15 N. tex^{-1}). This brief comparison indicates that rayon is a much more promising biodegradable candidate than the PBS or PLA fibres presented. A comprehensive review of the literature on this topic would be beneficial, as the properties of these fibres are influenced by numerous factors and could potentially be further improved (Deroiné et al., 2019).

While fibre and yarn properties are important, the end user is interested in the performance of the rope. Rope strength efficiency or conversion factors provide an indication of the effective transfer from yarn to rope. Here, the values in Table 3 indicate a strength conversion for rayon of around 65 %. This is within the general range of 50 to 85 % for synthetic fibres (McKenna et al., 2004), though these values are very dependent on terminations and test procedures. The range of rope constructions depends on available machine, but for the offshore oil and gas industry a very efficient long lay length parallel strand construction was developed in the 1990's (American Bureau of Shipping, 2021). These first results on small diameter short lay length braids could certainly be improved through reducing the braid angle, but an alternative parallel strand construction would improve strength and would also provide an external protection to limit degradation if longer durability is required.

4.2. Seawater degradation

The results obtained on the yarns demonstrate a fast degradation process under conditions representative of the Brest coastal marine environments (France). The viscose rayon, showed a rapid assimilation (Fig. 11) and a significant decline in mechanical properties, with break strength dropping by approximately 90 % within a two-week immersion period (Fig. 5). While the seawater was natural it may be noted that the tests were not conducted in real marine environments, in which apart from seawater other factors can impact on degradation, such as waves, currents, light intensity, rainfall-salinity changes, etc.

The respirometry results are in accordance with the findings of previous studies on viscose and Lyocell, indicating that regenerated cellulose is rapidly assimilated in coastal marine environments (Royer et al., 2023, 2021; Zambrano et al., 2020). However, the inocula used in the aforementioned studies are all from the North Atlantic coast (Belgium (Royer et al., 2023), North Carolina (Zambrano et al., 2020) and France for the present study). A more extensive range of inoculum collection sites is therefore required before the degradation of these materials in coastal marine environments can be stated.

The loss of mechanical properties observed in this study is particularly important. The kinetics are fast in comparison to the other biodegradable candidates. PBSAT and PBS/PBAT fibres retained almost all their properties after a 240 ageing period under exactly the same conditions (15 °C, in seawater bath) (Le Gué et al., 2024, 2023). Similar results were observed in other studies (Brakstad et al., 2022). Two other promising fibres were shown to demonstrate similar/slower degradation rates with 0–75 % retained mechanical properties for a PHBV fibre and 50–75 % for a PCL fibre whereas the PBS fibre retains the majority of its properties after one year of ageing (Sekiguchi et al., 2011). Kim et al. observed 85 % of tensile strength retention after 3 months for another PBS fibre (Kim et al., 2023).

These tensile property losses could be due to the formation of surface defects on the fibres, which lead to stress concentrations and facilitate crack initiation. The early work of Griffith on glass fibres showed the role of defects in brittle materials (Griffith, 1921) and more recent studies have highlighted the importance of defects in synthetic fibre degradation (Richard et al., 2023). The defects observed with the SEM (Fig. 9 Fig. 10) are characteristic of biodegradation mechanisms, whereby microorganisms present in the biofilm secrete extracellular

enzymes that degrade the surface of the biodegradable material. These mechanisms have been extensively characterised during injectionmoulded materials biodegradation studies (Laycock et al., 2017; Lucas et al., 2008; Wang et al., 2021). For these materials the impact on mechanical properties is only observable when the sample thickness is sufficiently small in comparison to the degradation kinetics, or when the degradation times are sufficiently long (Laycock et al., 2017). With regard to the biodegradation of fibres in marine environments, the observed degradation mechanisms appear to be similar (Le Gué et al., 2023, 2024; Sekiguchi et al., 2011). The diameter of the rayon fibres under consideration in this study is approximately ten microns, consequently minor surface defects result in a notable drop in properties. It is important to note that the diameters of the fibres employed in the studies referenced in the preceding paragraph are in the range of hundreds of microns (200-550 µm), which may contribute to the discrepancies in degradation kinetics observed and could be a means of increasing the durability of the rayon fibres. For the rope, the increased effective thickness of the structure results in slower degradation kinetics (95 % breaking strength loss over six months, and SEM observations show that fibres located inside the braid are preserved (Fig. 10), suggesting that the enzymes were incapable of diffusing within the structure. Consequently, enhancing the structural complexity could also significantly enhance the durability of the application, for example, through the development of a core and sheath structure.

The evolution of breaking strength (-20 %) and strain at break (+10 %)%) after six months of ageing demonstrated a relative stability of the rope mechanical properties in abiotic conditions. Fig. 7). Consequently, the prevention of biological actors would significantly enhance the durability of the ropes. The formation and maturation of the biofilm is a process that takes several days (Dang and Lovell, 2016; Qian et al., 2022). In the initial phase, so-called colonising species create the conditions necessary for other species to settle. This is followed by a maturation stage, during which specialist species take a larger proportion (i.e. those depending on available nutrients, substrate, and environmental variables). It is during this second stage that extracellular enzymes are produced and become active. Consequently, applications such as fishing nets, which are not static in the water for long periods, may not be affected by these degradation mechanisms. Protective measures against this factor can also be considered, such as the use of antifouling treatments (while considering their impacts on biodiversity (Li et al., 2023)).

The results of this study demonstrate that rayon undergoes rapid degradation in coastal marine environments with strong signs of biodeterioration and bioassimilation. However in the case of structural applications lost at sea, such as fishing gear or ropes, it is not uncommon for them to be deposited in deep marine environments (Harris et al., 2023; Morales-Caselles et al., 2021). These environments are markedly different from coastal marine environments and are less conducive to biodegradation (oligotrophic environments, lower concentration of microorganisms, low temperature and high hydrostatic pressure) (Chamley, 2024). A number of studies have highlighted concerns regarding the presence of regenerated cellulose in significant quantities within sediments at various deep-sea locations (Woodall et al., 2014), identification questioned by the work of Comnea-Stancu et al. [59]. Consequently, the biodegradation of cellulosic fibres should be investigated in deep-sea environments before confirming their biodegradability in marine environments. Further experiments using the same ropes in deep-sea environments are underway to elucidate these observations. These include instrumentation lines for oceanography and fish aggregation devices. Further work is needed, both to examine material parameters (additives, constructions) and the influence of other marine environment parameters (UV, stress coupling, temperature, pressure...).

Finally, the results of Kim et al. (Kim et al., 2022) indicated that the environmental impact of viscose rayon was higher than those of natural cellulose fibres, including cotton and flax, and similar to those of

synthetic fibres, such as nylon, PET, and polypropylene (PP), in terms of damage to human health, ecosystem quality, and resources. Ideally, in order to compare these fibres it would be necessary to have all the production details (entrants and exits of materials and energy), and to compare them on an identical basis for a given application, so that the fibre production cycle could be optimized (Shabbir and Mohammad, 2017).

5. Conclusions

The findings of this study indicate that the fibres produced by Cordenka are suitable for the manufacture of ropes intended for short term structural applications in seawater. The mechanical properties are highly promising in comparison to some conventional synthetic fibre ropes with similar characteristics and can be further enhanced through the application of improved processing techniques.

Furthermore this material demonstrates a rapid biodegradation and mineralisation in French marine coastal environments. The investigation into the mechanical properties has revealed that biotic factors are predominantly responsible for the considerable mechanical deterioration observed during the ageing process.

The multiscale approach has demonstrated a significant impact of the structure on the mechanical property evolution during immersion, and therefore seems necessary for the study of these phenomena. The results obtained confirm that the biodegradation process for these small ropes is surface-based, as evidenced by the degradation of the surface of the fibres submerged in seawater (in biotic conditions). The water exchange surfaces and the diameter of the elementary fibres are thus of significant importance for these processes. To modulate the durability of the ropes, the modification of the elementary fibre diameter and the use of external protection (sheath, protective membrane) should be considered.

Further research is required to gain a deeper understanding of the conditions required for microorganisms to initiate degradation. First, a more comprehensive understanding of the colonisation process could facilitate the identification of an optimal immersion duration, preceding which there is minimal risk of biotic degradation, thereby markedly enhancing the conservation of mechanical properties. Second, results are necessary in other marine environments (continental shelves, slopes, abyssal zones) and in other ocean basins to ensure the presence of the requisite microorganisms across all marine realms. These studies are also necessary to assess the durability of rayon in each of these marine environments, which is essential for the design of the potential applications.

CRediT authorship contribution statement

Alexandre Chamley: Investigation, Writing – original draft. Wilfried Troalen: Investigation, Writing – review & editing. Louis le Gué: Investigation. Peter Davies: Writing – review & editing. Floriane Freyermouth: Writing – review & editing. Christophe Baley: Writing – review & editing.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the first author used DeepL Write in order to improve the readability and language of the manuscript. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the published article.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Chamley Alexandre reports financial support was provided by General Directorate of Armaments. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.marpolbul.2025.117917.

Data availability

Data will be made available on request.

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