Degradation mechanisms in PBSAT nets immersed in seawater

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Abstract :

Fishing gears are known to continue fishing after being abandoned, lost, or discarded through a phenomenon called ghost fishing. After this ghost fishing period, disintegrated nets contribute to plastic pollution. Biodegradable nets could be an alternative to conventional nets to reduce ghost fishing but must strike a delicate balance between durability and degradation. This study evaluates the seawater degradation of a net made of polybutylene(succinate-co-adipate-co-terepthalate) (PBSAT) at several scales: monofilament, knot, and net. Mechanical testing was used to monitor the strength at each scale during immersion at several temperatures: 4 °C, 15 °C, 25 °C, 40 °C. Steric exclusion chromatography (SEC), scanning electron microscopy (SEM) and X-ray tomography were used to investigate degradation processes. While no degradation was observed for samples immersed for 240 days at 4 °C, hydrolysis led to embrittlement at 40 °C. Biotic degradation was observed at both 15 °C and 25 °C with distinct degradation patterns and bacteria shapes. At both temperatures, the degradation was accelerated in the knot, leading to an unusable net after 240 days at 15 °C while no loss of strength was detected at the monofilament scale. These findings suggest that the durability of the knot is critical for successful development of a biodegradable polymer for application in gillnets.

Highlights

► Internal stress due to curvature accelerated biotic degradation in the knot. ► Durability of the knot should be emphasised when developing a biodegradable net. ► Biotic degradation and bacteria shapes were different at 15 °C and 25 °C. ► PA6 net retained its strength during ageing at all temperatures.

Keywords : Biodegradable net, Seawater degradation, Biotic degradation, Hydrolysis, Ghost fishing

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1. Introduction

Plastic pollution by Abandoned, Lost, or Derelict Fishing Gear (ALDFG) is present everywhere in the marine environment, from urban coastline [1] to isolated Arctic islands [2], and from ocean surfaces [3] to deep sea [4]. ALDFG are known to continue catching individuals after being lost, due to a phenomenon called ghost fishing [5]. Ghost fishing impacts all marine species including birds [6, 7], mammals [8, 9, 10], corals [11, 12], and fish [12]. Besides causing death and injuries to endangered species or juveniles [8, 9], ALDFG is also a threat to vessels and navy ships [13]. Around 6% of fishing nets are lost worldwide every year [14], and among them, gillnets present the highest risk once abandoned, discarded or lost in the marine

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Ref.	Bottom type	Depth [m]	Ghost fishing [days]
[16]	rocks	13	105
[17]	sand	45-86	155
[18]	sand	15-25	240-330
[19]	sand	13	200
[19]	artificial reef	13	284-561
[20]	mud + seaweed	9-14	106-112
[21]	sand/mud	117-135	224
[22]	sand/mud	117-135	375
[23]	sand	15-18	105-140

Table 1: Ghost fishing periods of lost static nets experiments.

environment [15]. Several studies have focused on the duration of ghost fishing of static nets, the results are summarized in Table 1.

Despite the persistence of fishing gear made of common petrochemical polymers [24], the fishing abilities of ghost nets decrease over time. The ability of a ghost net to fish is lost within a year of its loss, with no apparent influence of bottom type, as suggested by the ghost fishing periods presented in Table 1. This decrease is due to several factors: reduced headline height caused by the weight of the catch and greater visibility due to bio-fouling [25]. In deeper waters, biofouling is slower, so lost nets can remain dangerous to deep-sea ecosystems for several years [23]. Once collapsed, entangled in themselves, buried in the sediment or disintegrated, ALDFGs continue to impact the environment by breaking down into microplastics [26], particles less than 5 mm in size [27]. Despite the loss of mechanical properties that led to fragmentation, the chemical structure of the polymers remains intact [28], with molar masses that are too high to be mineralised [29]. This chemical stability leads to the accumulation of these particles in the marine environment [30, 31, 32, 33], where they can be ingested [34], leach toxic additives [35], and be a vector for organic pollutants [36].

Biodegradable polyesters have been developed to mitigate plastic pollution by undergoing faster degradation than conventional polymers at sea [37]. The faster breakdown of those biodegradable polymers is due to the presence of ester linkages in their chains [38] that induce chain scission by hydrolysis due to the effect of water and microorganism enzymes [39, 40]. This enzymatic hydrolysis is known to be faster in the amorphous zone of biodegradable polymers [41]. Biodegradation is complete when the polymer's molecular weight is low enough to be mineralized by microorganisms under aerobic or anaerobic conditions [42].

Adopting seawater biodegradable polymers offers a potential solution to mitigating ghost fishing incidents in situations where gear retrieval is not possible [43]. These polymers degrade faster in the marine environment, leading to a quicker loss of mechanical strength compared to conventional nets [44]. Additionally, this approach could address the accumulation of microplastics, as these materials can be fully mineralized by marine microorganisms within a reasonable time frame [29]. However, biodegradable polymers do not resolve everything, the process of biodegradation relies heavily on the microbial communities that exist in a particular environment. As a result, the effectiveness of biodegradation is influenced by the specific conditions of the environment [45]. Microplastics coming from bioplastics can also physically impact the ecosystem before being mineralized [46]. Furthermore, using biodegradable polymers does not address the issue of releasing toxic additives during the degradation process. This phase might even be accelerated due to the faster degradation of these polymers, especially if they contain similar additives to those found in conventional plastics [47].

Polyhydroxyalkanoates (PHA) are emerging as promising polymers due to their potential for customisable mechanical properties and degradation rates [48]. These characteristics open up the possibility of adapting to various operational characteristics (e.g. lifespan, target species) of different types of fishing gear used in commercial fisheries. Nevertheless, there is currently a lack of information regarding their application in monofilament form within the existing literature. Several other biodegradable polyesters have been examined for their suitability as fishing gear. Yu et al. (2023) [49] studied the catch efficiency of polylactic acid (PLA) gillnets, despite the persistence of this material in marine environment [50]. All the other studies were conducted on poly(butylene succinate) (PBS), poly(butylene succinate) blended with poly(butylene adipate terephthalate) (PBS/PBAT), and polybutylene(succinate-co-adipate-co-terepthalate) (PBSAT). As these materials biodegrade slowly at sea [51], they could offer a good balance between fishing capacity and low persistence in the marine environment compared with current materials. Some assert an equivalence in fishing efficiency [52, 53, 54, 55, 56, 57, 58, 49], while others suggest that biodegradable nets result in lower catch rates [59, 60, 44, 61, 62, 63, 64, 65]. This disparity has been linked to differences in mechanical properties, evidenced through tests conducted on monofilaments and knotted filaments, without specification of the knot type employed [49, 66, 67], or on meshes [63].

Brakstad et al. (2022) [24] investigated the degradation of a PBSAT fishing monofilament for 36 months in seawater and sterilised seawater with and without sediments at 20°C. Their findings revealed degradation in both media, with monofilaments exhibiting greater degradation in the non-sterilised seawater. Kim et al. (2023) [68] studied the biodegradation of a PBS net in a marine sedimentary medium and found around 30% of biodegradation after 180 days, with bulk hydrolysis, both leading to a slight decrease of the strength of the monofilament cut from the net.

While most studies in the literature monitored net mechanical properties change by looking at the monofilament [24, 57, 68] or the mesh [61], no study was found describing the ageing of a biodegradable weaver's knot. Knots are known to cause important mechanical properties' loss due to stress concentration [69] with failures happening in high curvature zones [70], which exacerbates the differences between

biodegradable and conventional nets [71]. This study aims to investigate the mechanisms of knot degradation at various temperatures to gain a better understanding of the degradation of biodegradable nets at sea.

The mechanical properties of fishing nets exposed to seawater and deionised water at varying temperatures were monitored using a multiscale method developed in a previous study [71]. Physical and chemical observations were used to approach the mechanisms involved in ageing and assess how the knot affects the kinetics of degradation.

2. Materials & methods

2.1. Materials

Two types of materials were used in the present study. A PBSAT with a melting temperature of 105° C and a melting enthalpy of 68.3 J.g⁻¹ was studied as the biodegradable alternative. The mean molecular weight of PBSAT pellets was 225 kg.mol⁻¹ as given by the technical sheet of the supplier. A polyamide 6 (PA6) with a melting temperature of 224 °C and melting enthalpy of 69.4 J.g⁻¹ was used as the reference material. Materials were supplied as custom-made net panels manufactured by S-EnPol (Korea). Net panels were made with a 0.57 mm monofilament, and with a mesh size (half mesh) of 75 mm. The monofilament has an elongation at break of 25 %, a break load of 72 N and a Young's modulus of 1.06 GPa.[71] Double weaver's knots were used to manufacture both nets.

Monofilament and knot samples were cut at randomised locations throughout the panels to characterise the mechanical properties at different scales. Monofilament samples could not be longer than 75 mm, as this was the mesh size (i.e., half mesh size between two knots). Net samples were obtained by randomly cutting samples of 7x3 meshes of the main net panels. All samples were stored in a refrigerator before mechanical testing and ageing.

2.2. Mechanical testing

Mechanical testing at each scale and ageing condition was performed on a 10 kN capacity test machine equipped with a 500 N load cell for monofilament and knot testing, and a 10 kN load cell for net samples.

Monofilament samples were tested by clamping them at both ends, because of the limited length available after cutting them from net panels. Displacement speed was set at 10 mm/min. Change in strain was measured by following two markers placed on the samples with a digital camera.

Knots were tested by blocking their four ends in clamps. Ends of the same monofilament were clamped together according to the ISO-1806 standard. Tightening measurements were made using markers on the four ends followed during the test with a digital camera. Knot tightening was calculated using virtual points defined as the mean position of the 2 markers on the same horizontal side of the knot. The tightening of the knot was defined as the relative increase in the distance between those two virtual points during the test.



Figure 1: Schematic view of the method used for mechanical testing at each scale.

Net testing was performed using snap hooks connecting the upper and lower meshes to beams fixed on the testing machine. The displacement rate was set to 50 mm/min for all samples. The same camera as for monofilament and knot testing was used to follow the knots during testing. The strain between knots was then calculated for each test.

The different test methods used in the study are summarised in a schematic view in Figure 1. Further details can be found in [71].

All tests were performed in a room maintained at 21°C with a relative humidity of 50%.

2.3. Ageing

The samples were immersed in renewed natural seawater from the Brest estuary, on the western French coast, maintained at several temperatures. Yarn, knot, and net samples were placed in plastic pots. The pots had holes in them to ensure water circulation. All sample types were immersed in seawater at 15°C, 25°C, and 40°C. Yarn and knot samples were also immersed in seawater tank at 4°C and deionised water tanks at 15°C and 25°C. PA6 net samples were immersed in similar conditions for comparison.

Samples were collected at diverse immersion times for testing. Before any analysis, they were dried for at least 12 hours in the same controlled room used for mechanical testing $(21^{\circ}C, 50\% \text{ RH})$.

Mechanical testing of aged samples was performed according to the same methods described in the mechanical testing section, with 3 replicates for each condition.

2.4. Steric Exclusion Chromatography

PBSAT samples were tested before and after ageing by Steric Exclusion Chromatography (SEC) to study the change in molecular weight with ageing. After dissolving the samples in a TCM chloroform solution for 24 hours, SEC was performed at 30°C, with an injection volume of 100 μL , a flow rate of 1.0 $mL.min^{-1}$ an Agilent-DRI refractive index detector and three columns: a PS/DVB Agilent 5 μm precolumn, and two Agilent Mixed-C 5 μm columns. Calibration was performed with chloroform as well as polystyrene standards.

2.5. SEM observations

Surface observations of pristine and aged samples were obtained by Scanning Electron Microscopy (SEM) using FEI Quanta 200 equipment. The samples were coated with a 60% gold and 40% palladium coating before being placed under the microscope.

For microorganism observations, knot samples were taken from seawater tanks and immediately immersed in a seawater solution with 2. 5% glutaraldehyde. After one night of soaking, the samples were rinsed in seawater for one hour to remove excess glutaraldehyde. Following this, progressive dehydration was conducted using absolute ethanol, with each immersion lasting 30 minutes. This dehydration process involved transitioning the samples through various ethanol concentrations in seawater: 25/75, 50/50, 70/30, and 80/20. Finally, the sample underwent dehydration in 90/10 ethanol solution, followed by immersion in two consecutive absolute ethanol baths, to eliminate the remaining seawater. The samples were then subjected to critical point drying to remove the ethanol and observed using the same method as that used for the initial and aged samples.

2.6. X-ray tomography

X-ray tomography was performed on PBSAT knots at the initial state and after 120 days of seawater ageing at 15°C and 25°C. The tests were conducted at a resolution of 5 μ m with a voltage of 60 kV and a current of 70 μ A, resulting in a total power of 4.2 W. The acquisition produced 1800 images, each exposed for 2 seconds, over one hour.

3. Results

3.1. Mechanical properties

Figure 2 presents an example of the change in mechanical behaviour on each scale after different immersion times in seawater at 25°C. The tension applied to knots and nets generates complex multiaxial stresses. To facilitate comparison across different scales, the description of the load was expressed in Newtons. At the level of individual monofilaments, the mechanical behaviour was characterised by the load that depended on the strain. Knots and nets are different structures, so different units of strain were used to plot their mechanical behaviour. At the knot level, the load was plotted against the tightening of the knot, while at the net level, the load was plotted against the average strain between knots. An origin offset on each of



Figure 2: Change in tensile test curves with seawater ageing at 25°C for each scale (monofilament, knot, net) and immersion time (initial, 45, 120 and 240 days).

the curves was applied for clarity. Reductions in both loss of strength and strain at break were observed at the monofilament scale, with no effect on the modulus at the monofilament scale and no effect on the initial part of the curve for the knot and net scales. At the knot scale, a progressive loss of strength was also observed, with a decrease in the knot's tightening capacity. For net testing, the decrease in load at break was noticed, accompanied by a reduction of the mean strain between the knots of the net after immersion. The aged curves remained as linear as the initial curve.

Figure 3 illustrates the relative changes in strength over immersion time for all examined temperatures (4°C, 15°C, 25°C, 40°C) and scales (monofilament, knot, net). Each panel represents a temperature, and each line style and marker form correspond to a scale. After 240 days of immersion in seawater at 4°C, there was no significant change in the strength of the monofilament and knot.

The strength of the monofilament immersed in seawater at 15°C was stable with ageing time, with no significant loss after 240 days. For the knot scale, no difference was observed after 45 days of ageing, followed by a fast decrease to 50 % of the initial strength after 120 days of immersion. After 240 days of immersion, the PBSAT knot exhibited a reduction in strength by approximately 65 % compared to its initial state, as well as compared to the monofilament. At the net scale, the PBSAT followed a similar trend to the knot scale, but with a higher amplitude. No significant differences were observed after 45 days of ageing. However, after 120 days, the net lost nearly all its initial strength with a decrease of 95 %. After 240 days, the net was too brittle to be placed on the tensile test machine, breaking at the knot.

During immersion at 25°C, the strength of the PBSAT monofilament decreased following a linear trend until its property reduced by 40 %. Both knot and net scales followed a similar trend but lost 50 % of their initial strengths after 240 days of ageing.

For ageing at 40° C in seawater, the loss is faster than for other temperatures, with all scales losing more than 30 % of their initial strength during the first 45 days. After 240 days respectively 15 % and 10 % of the monofilament and knot strengths remained, while net samples were too brittle to handle.



Figure 3: Relative change in the load at break [%] after immersion at several temperatures up to 240 days for the three scales (monofilament, knot, net).

	initial	$15^{\circ}\mathrm{C}$	$25^{\circ}\mathrm{C}$	$40^{\circ}\mathrm{C}$
PBSAT	297 ± 6	0*	125 ± 14	0*
PA6	1061 ± 97	971 ± 82	984 ± 41	1100 ± 65

Table 2: Strength [N] of PBSAT and PA6 nets aged 240 days at several temperatures. *samples broke during handling operations

The strengths of PBSAT and PA6 nets before and after 240 days of seawater ageing at various temperatures are shown in Table 2. At the initial state, PBSAT nets are 72 % weaker than PA6 nets. This difference increased with ageing since the strength of PA6 nets were unaffected after ageing at all temperatures, whereas PBSAT nets became brittle at 15°C and 40°C, or saw their resistance divided by 4 at 25°C.

PBSAT knot samples have also been immersed in deionised water. Table 3 presents the relative difference with initial strength after ageing 120 days at 15°C and 25°C in renewed natural seawater and deionised water. For ageing at 15°C, with 26 \pm 8 N the loss is ten times higher for knots immersed in seawater than for the knot immersed in deionised water that showed a strength of 52 \pm 5 N. The loss is slightly higher for knots aged at 25°C in seawater with 37 \pm 1 N than for the knots aged in deionised water with 40 \pm 1 N.

	$15^{\circ}C$, 120 days	25° C, 120 days
seawater	-52 %	-33 %
deionised water	-5 %	-25 %

Table 3: Relative difference with initial strength after immersion at 15°C and 25°C in seawater and deionised water for PBSAT knot samples.



Figure 4: a) Change in molar mass [Da] distribution for pristine PBSAT and after 45, 120 and 240 days of immersion at several temperatures, b) Change in molecular weight M_w for PBSAT knot samples after immersion at several temperatures (the grey box is the initial dispersion).

3.2. Steric exclusion chromatography

Steric exclusion chromatography was applied on initial and aged samples to gain a better understanding of the causes of the deterioration of the PBSAT. Figure 4a presents the evolution of the molar mass distribution with ageing time for each temperature. Each panel corresponds to a temperature, and within each panel, the lighter the colour, the longer the ageing time. Ageing in a 4°C seawater did not change the molar mass distribution, after 240 days of immersion the aged distribution overlaps the initial one. The same observation can be made for an ageing temperature of 15°C: the distribution did not evolve with immersion time. The molar mass distribution was more sensitive to seawater at 25°C since a slight shift to the smaller molar mass was observed. A similar shift toward a smaller molar mass can be observed at 40°C, but the magnitude is more important, indicating a faster decrease in the molar mass distribution.

Figure 4b summarizes the change in molecular weight M_w with immersion time. The grey marker and the light grey area represent the initial molecular weight and its standard deviation, 79.6 ± 7.5 kg.mol⁻¹. At 4°C and 15°C, measured molecular weight are respectively 76.3 kg.mol⁻¹ and 82.5 kg.mol⁻¹. They are



Figure 5: Load at break versus molecular weight M_w for each PBSAT tested scale (monofilament, knot, net).

included in the initial dispersion: the molar mass at these temperatures stayed stable. At 25°C, even though it is hard to describe a general trend at 120 days of ageing, a significant decrease of 29% is observed with a molecular weight of 56.2 kg.mol⁻¹ after 240 days. The degradation process was more pronounced for PBSAT subjected to seawater ageing at 40°C, with a gradual decrease in molecular weight down to 56.2 kg.mol⁻¹, 28% of its initial value.

The load at break for each condition has been plotted against the corresponding molecular weight for all scales on Figure 5. The ageing temperature is represented by the marker's shape and colour. At the monofilament scale, low temperatures and short ageing time values are in the initial dispersion. Conditions where a loss of strength was observed, have a linear distribution, which is consistent with a previous study on a PBS/PBAT monofilament [37]. When testing knots, losses of load at break were observed at 15°C, where no drops in the molecular weight were noted. This led to a non-linear distribution of load at break versus the molecular weight. At 25°C, losses in both load at break and molecular weight were observed, the load at break then appeared linear with molecular weight at this temperature. However, while the same observations were made for ageing at 40°C, a significant shift appeared in the loads at break. For example, a higher load at break was observed for the least aged 40°C condition, than for the most aged 25°C condition. For nets, the trend is similar to that observed for knots, with higher amplitude and lower scatter in each condition. For two conditions the load at break was set to zero because samples could not be tested: 15°C after 240 days, and 40°C after 240 days. However, net samples immersed for 240 days at 15°C were too brittle to be tested despite showing a molecular weight similar to the initial state, unlike samples immersed at 40°C during 240 days that were brittle and presented a low molecular weight.

The molecular weight was measured for the monofilament and knot only to study the influence of the knot on the degradation process after 240 days of ageing at 25° C. No significant difference was observed with molecular weight being respectively 56.1 kg.mol⁻¹ and 53.0 kg.mol⁻¹ in the monofilament and the knot.

The differences in molecular weight after 120 days in natural seawater and deionised water at 15°C and

	$15^{\circ}C$, 120 days	$25^{\circ}\mathrm{C},120~\mathrm{days}$
seawater	-6 %	-5 %
deionised water	-10 %	-19 %

Table 4: Relative difference with initial molecular weight after immersion at 15°C and 25°C in seawater and deionised water for PBSAT knot.



Figure 6: SEM observations of PBSAT knots after 240 days of immersion at several temperatures.

25°C were also examined. Table 4 presents the relative differences compared with the initial molecular weight for these conditions. While no significant differences were observed between seawater and deionised water at 15°C, the molecular weight decreased more in deionised water than in seawater at 25°C. However, regarding the molecular weight in number, the values were close, respectively 21.6 kg.mol⁻¹ and 22.1 kg.mol⁻¹ for seawater and deionised water.

3.3. SEM observations

SEM observations were made on various samples to investigate changes in surface topology during ageing. Figure 6 shows observations made on knots before and after 240 days of ageing for each temperature. At the initial state, the surface of the monofilament was smooth with no defects. After 240 days in a 4°C seawater, no significant change was observed on the surface of the monofilament or in the knot. Immersion at 15°C led to surface erosion on knot samples. Important cracks in the length direction can also be observed inside the knot. Outside the knot the erosion was less important and no cracks were observed. After immersion at 25°C, an erosion of the surface also happened, but with a different pattern and deeper holes. The erosion appeared more important in the knot than outside the knot, with deeper and bigger holes on the surface oriented to the outside. Seawater ageing at 40°C for 240 days did not modify the knot's surface, which was in the same state as initially.

Figure 7 shows observations made on samples immersed in renewed natural seawater and deionised water for 120 days. After 120 days of immersion in seawater at 15°C, knots showed holes and important longitudinal cracks, as observed after 240 days. After the same amount of time in deionised water, no holes



Figure 7: SEM observations on PBSAT knots after 120 days of immersion in seawater (SW) and deionised water (DW) at 15°C and 25°C.

and no cracks were present on the knot's surface, which was in the same condition as in the initial state. Ageing at 25°C led to an erosion of knot samples which was more important inside the knot. This was not observed for samples immersed in deionised water, where the knots remained unaltered, similarly to the initial state.

PBSAT samples immersed in seawater at 15°C and 25°C have also been treated to allow the observations of biological specimens on their surfaces using a scanning electron microscope. Figure 8 depicts the samples' surfaces alongside the microorganisms on them. The images at the bottom are zooms of the images at the top. While presenting different forms, bacteria were observed in degraded areas for both ageing temperatures. At 15°C, the predominant bacterial morphology observed was spherical, characterised as cocci [72], whereas at 25°C, rod-shaped bacteria, known as bacilli [72], were more prevalent.

Figure 9 shows details of a degradation pattern observed on samples immersed at 15°C. The presence of rectangular-shaped holes was consistent across observations, which means that the curvature in the knot did not influence the presence of these defects. The area surrounding the holes appeared intact, while within the holes, the surface seemed to have collapsed inward, revealing visible bacteria, unlike the surrounding surface.



Figure 8: Observations of microorganisms on the degraded surfaces of PBSAT samples immersed at 15°C and 25°C.



Figure 9: Observation of a degradation pattern caused by microorganisms during ageing at 15° C.



Figure 10: X-ray tomography observations for the PBSAT at the initial state and after 120 days of seawater immersion at 15° C and 25° C.

3.4. X-Ray tomography

X-ray tomography was used to further inspect the degradation of knot samples after 120 days of immersion in seawater at 15°C and 25°C. Figure 10 shows different cross-sections for both initial and aged samples. Their colours correspond to the position of the slices on the diagram on the left. Observations made on the sample at the initial state did not reveal any defects or cracks. After 120 days of ageing at 15°C, X-rays revealed internal cracks and holes inside the monofilament. These defects appeared near contact surfaces and penetrated the material to several hundred microns, with cracks and holes running through the monofilament. Seawater ageing at 25°C led to surface erosion as shown in Figures 6 and 7. This erosion only affected the first hundred microns, and was greater in the monofilament parts subjected to tensile stress because of the curvature.

4. Discussion

The durability of a PBSAT gillnet has been studied at various scales and temperatures. Figure 3 depicts the change in strength at various scales under different immersion temperatures. No change was observed for samples aged at 4°C, indicating material stability at this temperature. At 15°C, the monofilament did not lose strength, unlike the knot and the net. This significant difference between the monofilament and the knot suggests that ageing is accelerated in the knot or that it is more sensitive to complex mechanical stresses induced during knot testing. Since no loss was observed after 120 days in 15°C deionised water, the degradation does not seem to be linked to hydrolysis. At 15°C, the net's strength decreased even faster than that of the knot, indicating non-uniform ageing within the knots in net samples. This means that some knots were more degraded than the individual knots that were tested. Immersion in 25°C seawater led to a gradual loss of strength at all scales, as also evident in Figure 2. Similarly to what was observed at 15°C, the monofilament lost its properties less rapidly than the knot and net scales, which at this temperature experienced equivalent losses, reflecting a more uniform ageing of the net's knots. Contrary to what was observed at 15°C, the properties of the knot dropped by an equivalent extent in both seawater and deionised water at 25°C, indicating that water has an impact on the material's ageing process at this temperature. Samples immersed in 40°C seawater experienced a fast loss of properties leading to a net that was breaking during handling operations. At the same time, the monofilament retained more strength than the other scales, as observed at 15°C and 25°C. With no loss at 4°C and 15°C, a gradual loss at 25°C, and a faster loss at 40°C, monofilament [37], where the degradation processes causing those losses were identified as hydrolysis and biotic degradation.

SEC measurements were performed on initial and aged samples to further investigate the degradation process and how they impact the mechanical properties across the three scales. After 240 days in 4°C seawater, no change in polymer chains was observed, with a molecular weight distribution that fits the initial one, as seen in Figure 4a, which leads to a stable molecular weight with ageing as seen in Figure 4b. The observation of constant strength at the monofilament and knot scales is consistent with the stability of the polymer's molecular structure. Observations at 15°C are the same as for ageing at 4°C. However, while the stability of the monofilament is consistent with the stability of the polymer's chemical structure, the properties of the knot decline without a corresponding decrease in molecular weight, which disrupts the linear relationship between breaking force and molecular weight observed at the monofilament scale. Hydrolysis of polyesters like PBSAT induces the breaking of their polymer chains by water at the ester linkage [73]. The absence of chain scission in the samples aged at 15°C in both seawater and deionised water for 120 days then suggests that hydrolysis is not involved in the degradation process observed in the knot at this temperature. As seen by the change in distribution at 25° C in Figure 4a, the polymer was sensitive to water at this temperature, leading to a loss of molecular weight. 28% of the initial molecular weight after 240 days as seen in Figure 4b. A loss of strength was observed at this temperature for samples aged in seawater and deionised water, with a drop in molecular number after 120 days of ageing in deionised water. These findings indicate that hydrolysis could be one of the degradation processes taking place at 25°C. At 40°C, molecular weight and strength decreased following the same trend, leading to a linear distribution of points for monofilament and net scales in Figure 5, with an exception for nets because the samples aged 240 days were too fragile to be tested. As a decrease in molecular weight occurred at this temperature without any surface degradation, hydrolysis could be involved in the degradation of material properties.

Figure 11 presents the curvature change inside the two monofilaments composing the weaver's knot, each plot representing the curvature in one monofilament. 3D views with the curvature in the concerned monofilament were added next to the corresponding panels. The weaver's knot exhibits high curvature zones where the monofilaments are bent and tied together, inducing residual stress. While some defects were found where the curvature was low, the presence of defects in high curvature zones led to the propagation of cracks due to residual stress and the failure of the monofilament in these zones. This explains the collapse observed in SEM images after ageing at 15°C, for example, in Figure 7, the knot broke in a high curvature zone of the monofilament shown in the upper panels of Figure 11. The monofilament represented on the bottom panels in Figure 11 presents a constant maximum curvature at the level of the loop, where failures were also visible in Figure 6. These large defects induced by ageing resulted in a faster decrease in strength for the knot and the net than for the monofilament, as shown in Figure 3. These findings also further explain the loss of properties despite the loss of molecular weight, coming from a damaged knot structure rather than a degradation of the molecular structure. A more important erosion of the surface was observed on samples aged in 25°C seawater as shown in Figure 6. Uniformly distributed holes were found in the monofilaments outside the knot, which is a degradation pattern previously observed in the literature [37, 57]. This pattern was also observed outside the knot after 120 days of ageing, as shown in Figure 7. For both ageing times, erosion had less impact on the monofilament compared to inside the knot where certain parts of the monofilament were washed out, leading to larger holes. As seen on X-ray tomographies, the erosion is limited to the surface of the monofilament. Deeper holes both on Figure 6 and Figure 10 are consistent with the high curvature zones observed in Figure 11. When a monofilament is bent, the outer side is under tensile stress because it is stretched over a longer distance compared to the inner side, which experiences compressive stress from being compressed over a shorter distance. In Figure 10, the last row of images displays a zoom on a cross-section of a monofilament. The outer side of the bent monofilament can be seen on the left-hand side of the image, with larger holes compared to the rest of the section, which shows that the bending of the monofilament caused the surface defects to open up and propagate. In addition to a loss of molecular weight, the presence of these defects has contributed to the degradation of the mechanical properties of the polymer. At both 15°C and 25°C, the degradation was faster in high curvature zones, which are also where the failure happens when testing a knot [70]. Tightening of the knot often occurs during fishing operations, increasing the likelihood of cracks and their propagation due to internal stresses within the knot. Degradation could thus be faster during real fishing operations. After being aged at 40°C for 240 days, knot samples showed no signs of degradation despite their brittle behaviour and significant loss of chemical integrity. This suggests that chain scission did not lead to the surface degradation observed at 25°C and that another degradation mechanism is involved at this temperature. Internal and external erosion did not happen for samples immersed in osmosis-deionised water, where marine micro-organisms were less or not



Figure 11: Change in curvature κ [mm⁻¹] along the normalised length of the two monofilaments of a PBSAT knot [71].

present, indicating that biotic degradation occurred at 15°C and 25°C. However, the degradation patterns observed at both temperatures were different.

Microorganisms were observed on the surface of the samples for both temperatures, as shown in Figure 8. At 15°C, where the degradation was mainly internal and led to important cracks in the knot, bacteria appeared as cocci. As seen in Figure 9, bacteria degraded the inside of the material by digging holes, rather than colonising the surface. Biotic degradation at this temperature did not lead to a decrease in molecular weight, as bacteria degraded accessible chains, which led to a local reduction in molar mass that is small compared to the bulk molecular weight that remained stable due to the low temperature. At 25°C the degradation was more spread and limited to the surface, observed bacteria were then in a bacillus shape. The more generalised biotic degradation observed at 25°C could be explained by an increase in the enzymatic activity of bacteria with temperature [74]. but also by a coupling of hydrolysis and biotic degradation, the latter being accelerated when the molar mass decreases [75]. Observing the different shapes of bacteria provides a first clue as to why the degradation appeared different at 15°C and 25°C. However, it is not possible to determine the type of bacteria only from their shape, nor to determine if they are different. Bacteria can indeed change their shape from bacillus to coccoid to better adapt to environmental pressure, such as colder temperatures [76]. These observations suggest that the temperature at which the bacteria are subjected affects their shape and their strategy for breaking down the polymer. Biotic degradation can occur at even lower temperatures, as some psychrophilic bacteria can degrade polymers at temperatures close to

0°C [77]. However, no degradation was observed at 4°C. These cold-adapted bacteria are present in cold environments such as the Arctic or alpines regions [78] and do not duplicate and slowly die when exposed to temperatures above 20°C [79]. This implies that natural seawater sampled from the Brest estuary may lack microorganisms capable of adapting to a 4°C environment, and even if they are optimally adapted to this temperature [80], their maximum enzyme activity exceeds their upper growth threshold [74]. As prolonged exposure to 40°C is lethal for a large percentage of marine bacteria [79], the absence of biotic degradation is coherent for samples aged at this temperature.

The observed susceptibility of PBSAT to seawater degradation makes it a potential material to reduce ghost fishing from lost gillnets. As shown in Table 2, the PBSAT gillnet lost all of its mechanical properties after 240 days of ageing, and therefore its ability to fish after being lost. After 240 days at 25°C, the PBSAT net lost 50% of its initial strength, which could increase the likelihood of an animal escaping if caught in the net after it has weakened. In contrast, the PA6 net did not lose any of its properties at any of the temperatures studied over the given period, thus proving the persistence of these nets in the environment once lost, and confirming that the losses observed by several authors are linked to abrasion and not to ageing of the material in the marine environment [62]. However, while seawater degradation reduces the impact of nets after loss, it also poses the question of the durability of nets during the operation phase, and the balance between degradation and stability of alternative nets used to limit ghost fishing. The results then underline the technical challenge of offsetting the degradation process discussed by [81]. Furthermore, the capability of PBSAT nets to undergo biotic degradation was highlighted, but this study did not assess the assimilation of the potential degradation products nor their toxicity for the marine environment.

5. Conclusion

This study aimed to gain better insights into the durability of biodegradable gillnets to reduce ghost fishing. A mechanical multiscale approach was used to assess the durability at several scales: monofilament, knot, and net. SEC measurements, SEM observations and X-ray tomographies were also conducted to understand the degradation mechanisms in PBSAT samples. The multi-scale approach used in this study revealed the importance of assessing knot durability. Indeed the knot induced high curvature zones that led to faster and more severe biotic degradation. This means that knots are not only the weakest point of the net but also the point at which degradation is most rapid. Conducting tests on net structures also showed that individual knot testing provides a good indication of how the net strength evolves with immersion time in seawater. Therefore, testing the knots can help further qualify a material for use in fishing gear netting, both in terms of initial properties and durability in seawater. However, further emphasis on the biotic degradation of PBSAT nets is necessary to fully understand their fate in seawater. These findings could help future development for less impacting fishing gear that meets the technical challenges of having a tailor-made lifetime to reduce ghost fishing while being non-toxic and completely biodegradable at sea.

CRediT authorship contribution statement

Louis Le Gué: Conceptualisation, data gathering, and investigation, formal analysis, visualisation, writing – original draft, writing - review and editing.

Esther Savina: Conceptualisation, data gathering, and investigation, formal analysis, writing – original draft, writing - review and editing.

Peter Davies: Conceptualisation, data gathering, and investigation, formal analysis, writing – original draft, writing - review and editing.

Mael Arhant: Conceptualisation, investigation, formal analysis, writing - review and editing. Nicolas Gayet: Data gathering, investigation, visualisation, writing - review and editing. Benoit Vincent: Conceptualisation, investigation, writing - review and editing.

Data availability

The datasets used and/or analysed during the current study will be made available from the corresponding author upon reasonable request.

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Declaration of competing interest

The authors declare no financial interests/personal relationships that may be considered as potential competing interests.

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