



Propaedeutic Study of Biocomposites Obtained With Natural Fibers for Oceanographic Observing Platforms

Simona Aracri^{1,2*}, Marco Contardi³, Ilker S. Bayer³, Muhammad Zahid³,
Francesco Giorgio-Serchi² and Adam A. Stokes²

¹ Soft Systems Groups, Scottish Microelectronics Centre, Institute for Integrated Micro and Nano Systems, School of Engineering, The University of Edinburgh, Edinburgh, United Kingdom, ² Department of Earth System Sciences and Environmental Technologies, Institute for the Study of Anthropogenic Impacts and Sustainability in Marine Environment, Italian National Research Council, Genoa, Italy, ³ Smart Materials, Istituto Italiano di Tecnologia, Genoa, Italy

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*Correspondence:

Simona Aracri
simona.aracri@cnr.it

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In response to the pervasive anthropogenic pollution of the ocean, this manuscript suggests the use of biodegradable elastomers in marine applications. The present study characterizes 25 samples of highly biodegradable polymers, obtained blending a base elastomer with natural fibers. Mechanical analysis and Scanning Electron Microscope imaging, reveal how base polymers behave differently depending on the plant fiber chosen, on the external forcing—exposure to water—and on the doses that constitute the final biocomposite. Results suggest that EcoflexTM 00-30 and EcoflexTM 00-50, mixed with potato starch, perform best mechanically, maintaining up to 70% of their maximum tensile strain. Moreover, early signs of degradation are visible on polysiloxane rubber blended with 50% vegetable fibers after 19 hours in distilled water. Analyses demonstrate that highly biodegradable elastomers are good candidates to satisfy the requirements of aquatic devices. Furthermore, the discussed materials can improve the dexterity and biodegradability of marine technology.

Keywords: bioplastic, elastomers, marine observation network, sustainable marine science, soft robots, natural fiber-reinforced composites, biocomposites

1. INTRODUCTION

Nowadays reducing the carbon footprint has become a mandatory call for all the technological fields. Indeed, the Paris agreement states that signatory countries will have to take measures today in order to reach the net zero emissions goal set by 2050. Embracing this goal translates into numerous measures to change the way we produce energy, consume products and dispose of them. These measures regard industrial, domestic and scientific environments. In this study we will focus on how to reduce the carbon footprint of marine research (Net Zero Oceanographic Capability, 2021).

The boundaries of marine sciences expand through exploration and hence the need of new data (Levin et al., 2019), for this reason researchers continuously create and call for novel technology (Madin et al., 2019). In the last two decades, the scientific community investigated the realization of a sustained observational network (Garzoli et al., 2009), which encompasses innovative technology (Nassar et al., 2018; Shaikh et al., 2019; Aracri et al., 2021), increased spatial resolution of data logging platforms and integration of new autonomous systems. Despite the

continuous technological evolution, some data platforms are never recovered, some fail at sea, others are destined to be lost in the water.

For instance, anchor parachutes, are routinely used to make the deployment of oceanographic moorings more gentle; highly biodegradable elastomers could already be employed to render them dissoluble in marine water. Also, biocomposites could be employed to build short term observations drifters, used to collect short term wind and current data and useful to validate coastal and lagoon models (Quattrocchi et al., 2021). Another example of sea platforms often lost at sea comes from animal based telemetry. In recent years, it increased exponentially, sustained, among other things, by reduction of manufacturing costs and miniaturization of novel devices (Harcourt et al., 2019). Marine scientists started to use marine animals tagging for data logging in the 1980s (Harcourt et al., 2019). This technique proved to be useful not only to study the behavior of marine

mammals or other sea animals (Hooker and Boyd, 2003; Lear and Whitney, 2016; Treasure et al., 2017), but also to collect environmental data, such as pressure, salinity and temperature of the water (Treasure et al., 2017). Marine animal tagging is particularly handy in patches of the sea covered by ice (Treasure et al., 2017), which are out of reach for traditional measurement methods—tethered Conductivity Temperature Depth (CTD), Autonomous Underwater Vehicles (AUVs), Remote Operated Vehicles (ROVs), Gliders, Argo floats.

Perhaps the most appealing field for biodegradable materials is soft robotics, i.e., a field of robotics that raised significant interest among scientists in the last 20 years (Trivedi et al., 2008). Among many of the applications of soft robots there are offshore operations (Sayed et al., 2018, 2021) and ocean exploration (Aracri et al., 2021), which both entail a significant risk of losing the device performing the task. More specifically, elastomers such as EcoflexTM 00-30 are widely used in aquatic

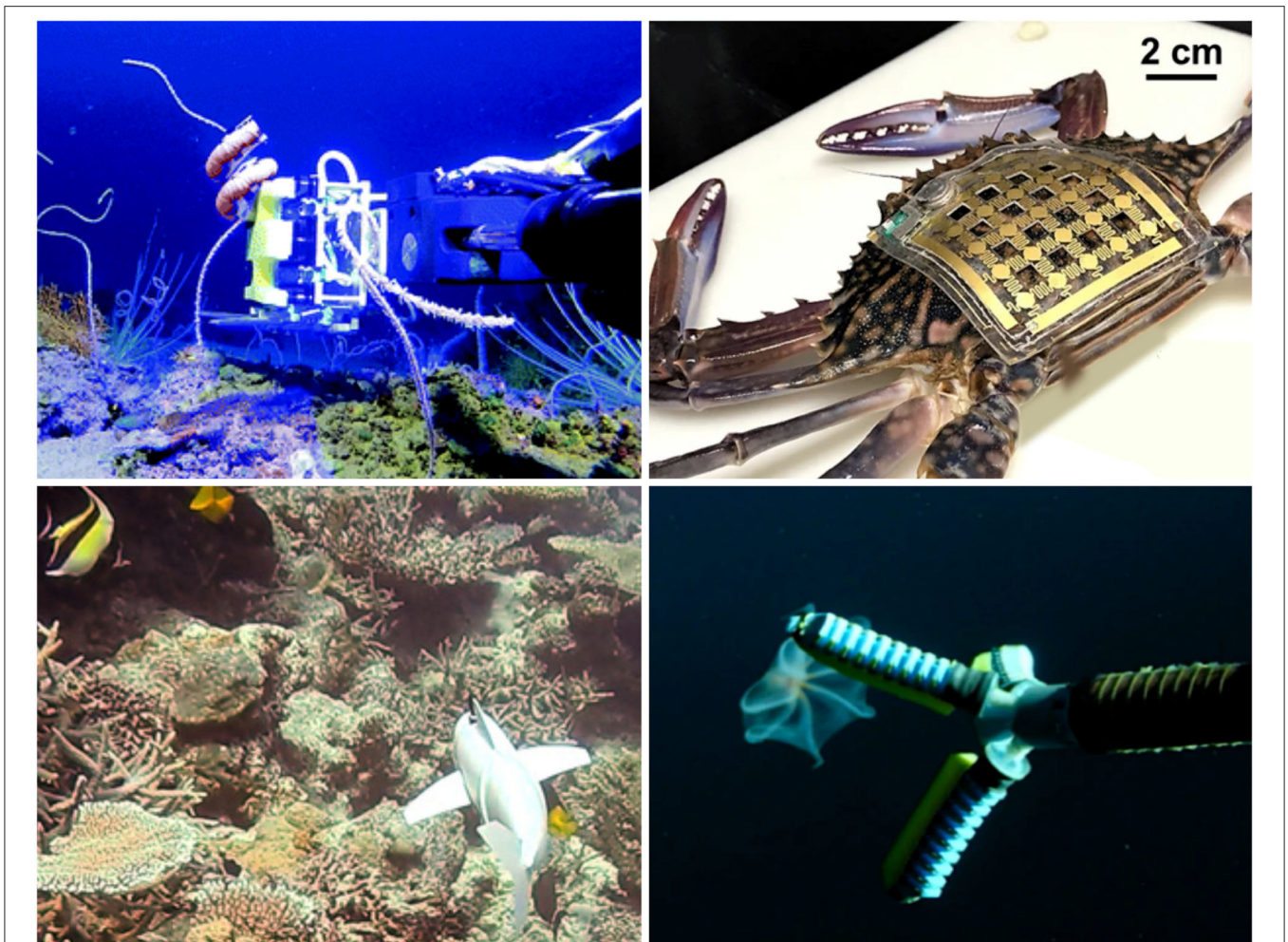


FIGURE 1 | Real world deployment of soft systems, i.e., settings where highly biodegradable elastomers can be employed. The image shows two different designs of soft robotics grippers for delicate sampling (Galloway et al., 2016-top right- and Phillips et al., 2018 -bottom right-), compliant marine sensory skin (Nassar et al., 2018), an underwater soft fish robot (Katzschmann et al., 2018).

TABLE 1 | Sample Database. Samples made of elastomers and plant based ingredients.

Bio component	Elastomer concentration %	E43	Ecoflex™ 00-10	ECOFLEX™ 00-30	Ecoflex™ 00-50
pure		E43PU^{SEM}	E10PU^{SEM}	E30PU	E50PU
potato starch	50	E43PS50^{SEM}	E10PS50^{SEM}	E30PS50	E50PS50
potato starch	60	E43PS60	E10PS60	E30PS60	E50PS60
potato starch	70	E43PS70	E10PS70	E30PS70	E50PS70
pre-treated p.starch	50	E43PPS50^{SEM}			E50PPS50
pre-treated p.starch	60	E43PPS60			
cocoa powder	50	E43CO50		E30CO50	
cocoa powder	60			E30CO60	
cocoa powder	70			E30CO70	
Water Degradation Test					
potato starch	50	E43PS50W ^{SEM}	E10PS50W ^{SEM}		

Concentrations and materials used are specified in the table. Each cell contains the ID of the sample. Superscript ^{SEM} indicates the samples, whose images were acquired with SEM. Bold font ID represents samples on which we performed tensile tests.

soft robotics (Giorgio-Serchi and Weymouth, 2017; Armanini et al., 2021; Bujard et al., 2021), because they have a density very close to that of water and an elasticity vaguely similar to that of living tissue. This last aspect is actually key to bio-inspiration, which often drives the design of soft robots. Examples of soft devices (**Figure 1**) that have been already deployed in the ocean are SoFi (Katzschmann et al., 2018), different kind of grippers, suitable for delicate sampling (Galloway et al., 2016; Phillips et al., 2018) and compliant sensory skin (Shaikh et al., 2019).

Beholding the pervasive pollution (Worm et al., 2017), of the world's ocean, it is inevitable to link the increasing number of deployed devices (Editorial, 2020) with the danger of aggravated contamination of the natural environment. The dilemma, arising from the contrast between the need of more oceanographic data and the need to reduce plastic pollution (Borja et al., 2020), made the research on biocomposites manufacturing (Bayer et al., 2014; Ceseracciu et al., 2015; Barron and Sparks, 2020; Lott et al., 2021) and transient electronics (Yin et al., 2014; Fu et al., 2015, 2016; Guna et al., 2016) thrive. Therefore, experimental projects contemplated bio-compatible materials for coral healing (Contardi et al., 2020), disposable soft robots (Rossiter et al., 2016, 2017) and observing platforms (Novelli et al., 2017), such as moorings (Pillsbury, 1993) and data retrieving autonomous systems (AOML Communications to Physical Oceanography, 2020), to reduce the economical and environmental cost of oceanographic cruises.

This study investigates the properties of soft biomaterials as substitutes of plastic components for devices meant to be left adrift at sea, which, with the call for increasing the deployments of autonomous platforms, is likely to grow.

The following sections describe the data and methods of our experiments to create highly biodegradable biocomposites. The article continues with the synthesis of the main results and the discussion. The conclusions derived from the experiments are at the end of the manuscript.

2. DATA AND METHODS

2.1. Experimental Design

Biocomposite, as intended in this study, is a material resulting from the mixing of a base elastomer and a plant fiber. By creating and testing different kinds of biocomposites in the lab, we want to demonstrate that their microstructure insures a level of biodegradability higher than the base polymer used, and that their physical properties ensure a mechanical performance similar to the one of commonly used plastic in the marine environment.

To characterize the effect of natural fibers in elastomeric blends, we created samples with different ratios of vegetable powder and elastomer. We selected two base polymers widely used in soft robotics: E43 and Ecoflex™.

E43 (silicone/polysiloxane rubber) is a mono-component elastomer. Ecoflex™ (Polybutylene Adipate Terephthalate-PBAT-) is a bi-component elastomer. As vegetable ingredients we used potato starch, produced by Sigma-Aldrich¹ and cocoa powder. If needed, we added heptane as a solvent to help amalgamating the mixture (starch + elastomer). Some samples were created using pre-treated potato starch, i.e., dried for 2 h at 50 degrees E43PPS50, E43PPS60, E50PPS60. The final blends database is synthesized in **Table 1**. Blends including cocoa powder easily stick to the Petri dish; to avoid it, we used Teflon coated containers.

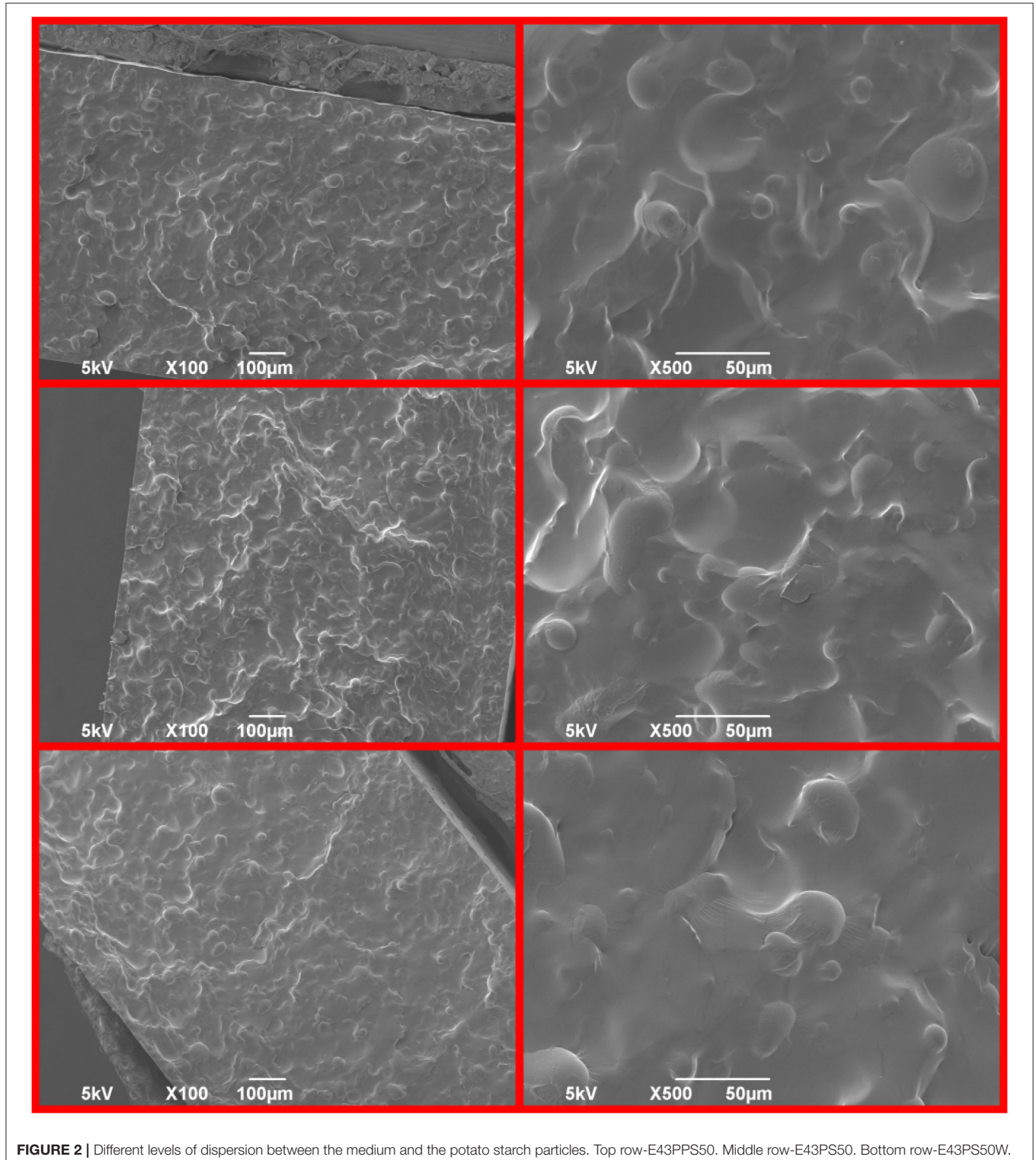
After curing time, we selected two samples with different base polymers, E43 and Ecoflex™ 00-10 (E43PS50 and E10PS50), both blended with potato starch, 50% concentration, and we immersed them in distilled water (MilliQ) at 50°C for 19 h (obtaining E43PS50W

¹<https://www.sigmaaldrich.com/IT/en/sds/sial/s4251>

and E10PS50W), to observe any possible degradation happening in such time span. We then measured and cataloged the microscopic and mechanical properties of the materials.

2.2. SEM Imaging

We used the most significant and integer samples to acquire SEM (Scanning Electron Microscope) images (**Table 1** superscript^{SEM}). For SEM imaging the samples have to be



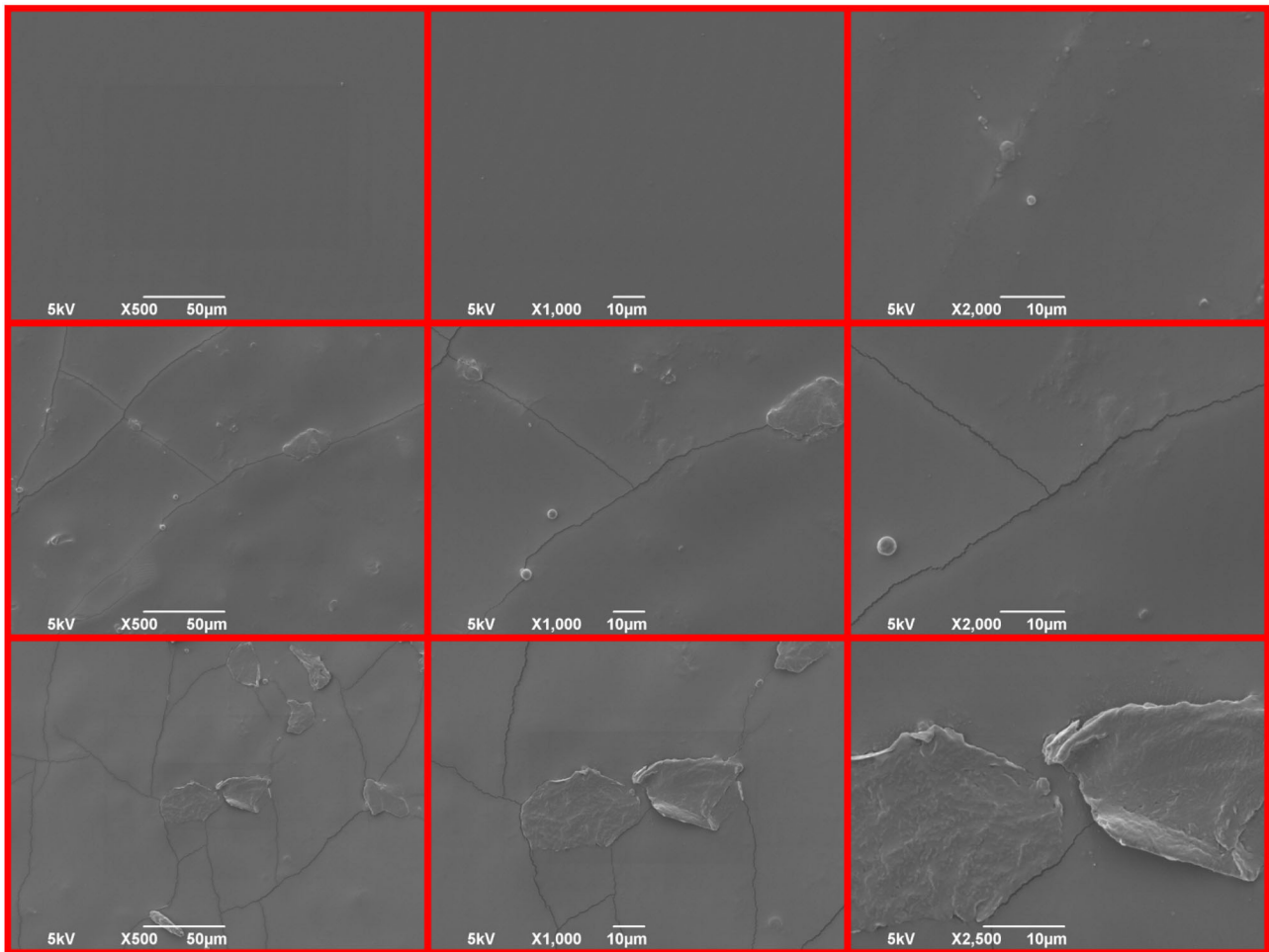


FIGURE 3 | Top row of images shows the flat surface of pure E43. The middle row shows the presence of some flaking and cracks on the flat surface of sample E43PS50. The bottom row presents the flat surface (that has been immersed in water) of sample E43PS50W with bigger and frequent flaking.

prepared to rest on small place holders, covered with carbon tape, to enhance conductivity. To obtain a clean cross section, the sample needs to be placed in liquid nitrogen, then snapped and placed on the carbon tape. We also placed top and bottom sides on the carbon tape. We covered the sides of the samples on the carbon tape with silver paste to improve conductivity. We used the sputter coater to cover the samples with gold powder. The main results are reported in **Figures 2–4** and discussed in the next section.

2.3. Mechanical Tests and Properties

To prepare the 19 samples (**Table 1** in bold) for tensile tests, we cut a dog-bone shaped specimen. The strain rate of the tensile test was 50 mm/min and we followed the ASTM D 882 method. The test consists in applying a controlled tensile force on every sample and register the modulus, maximum load, tensile stress at yield, tensile strain at yield, tensile stress at maximum load, the main results are graphically reported in **Figure 5** and **Table 2**.

3. RESULTS AND DISCUSSION

3.1. Cured Samples Observation

E10PS70 presented sparse visible crystals homogeneously distributed in the sample. E30PS70 instead had ramified crystals. E50PS70 was very brittle with dense ramifications. E43PS70 showed little sparse crystals. E43PS60 had sparse little dot-crystals. Also E43PPS60 displayed sparse little dot-crystals, even finer than E43PS60. E43PS50 showed sparse little dot-crystals, less frequent than E43PS60, but similar in size.

3.2. Scanning Electron Microscope Imaging

The cross section of sample E43PS50W clearly displayed dispersion between potato starch particles ($\sim 34.5\mu\text{m}$ diameter) and the medium (E43), see **Figure 2**. Also, the samples blended with pre-dried starch had finer conglomerates, $\sim 15.6\mu\text{m}$ diameter, of starch visible in the cross section (**Figure 2**). Instead E43PS50 had lumps of starch which reached $\sim 37\mu\text{m}$ diameter with an average of $\sim 27\mu\text{m}$. As noted in Stasiak et al. (2013) the

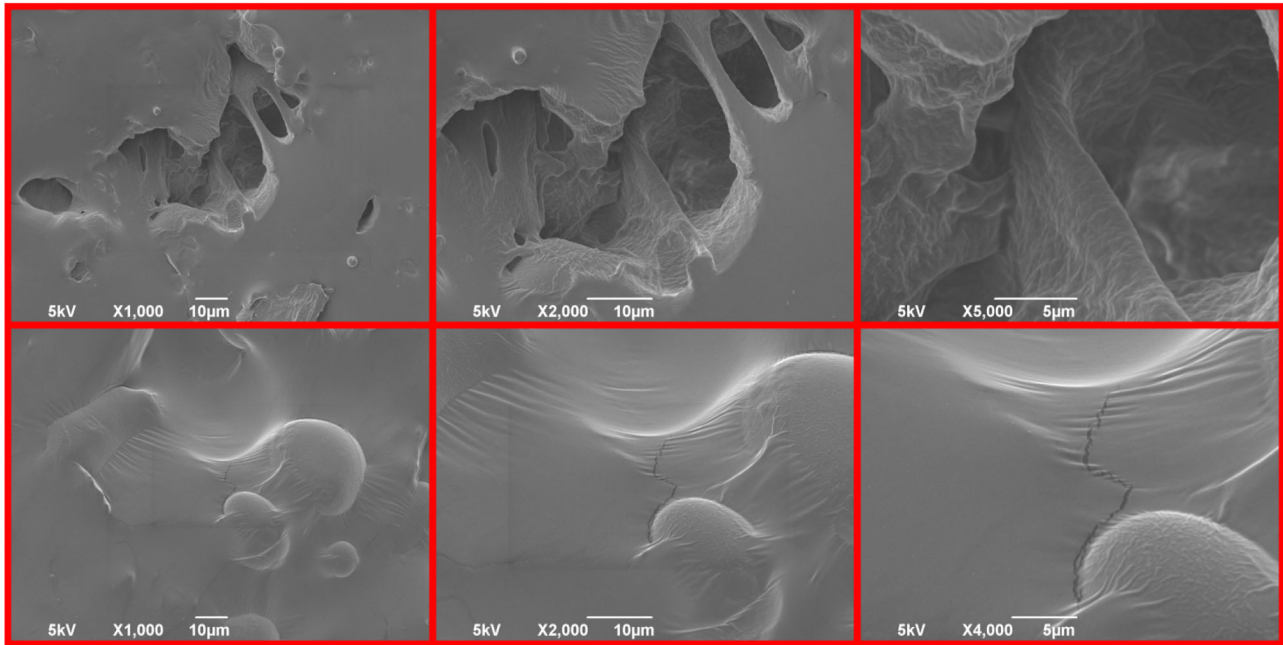


FIGURE 4 | Sample E43PS50W. Observed cracks and caves ascribable to the degradation of the material in the water.

potato starch produced by Sigma-Aldrich should be considered “wet”; pre-drying the starch in the oven likely removed the humidity in the powder and the bigger clumps disintegrated, allowing the starch to disperse in the medium with finer lumps. The surface on the submerged side of the sample had frequent flaking (**Figure 3**), 2 to 7 flakes, probably due to the degradation process in water. Samples that were not left in the water (E43PS50, E43PPS50) showed less flaking, 0 to 3 flakes, on the flat surfaces (view from above) (**Figure 3**). The flakes in E43PS50 are $21.7\mu\text{m}$ wide, $26.9\mu\text{m}$ long. The flakes in E43PPS50 reach up to $38.1\mu\text{m}$ width, $41.5\mu\text{m}$ length. E43PS50W presents “cave” structures, which are $29.4\mu\text{m}$ wide and $60.1\mu\text{m}$ long, probably the result of degradation happening in the water (**Figure 4**). All the materials containing starch displayed several cracks on both sides probably due to evaporation processes. The width of the cracks present in the samples E43PS50 and E43PPS50 is similar, i.e., $0.7\mu\text{m}$, sometimes $0.6\mu\text{m}$ for E43PPS50. E43PS50W instead had smaller cracks, $0.4\mu\text{m}$ wide, positioned at the base of those lumps of starch that had visible wrinkling at the attachment with the medium.

3.3. Tensile Tests

Observing the graphs emerges that E43 and EcoflexTM 00-10 are the materials most affected by the presence of plant fibers in the blend, which reduces their maximum tensile strain to 30% with respect to the pure base elastomer. In fact, in the E43 (EcoflexTM 00-10) case, the maximum tensile strain changes from 8.42 (12.77) mm to 2.68 (3.82) mm when blended with 50% potato starch powder. In the case of E43 the pre-dried potato starch did not seem to affect consistently the mechanical properties of the elastomers, i.e., with 50% concentration of starch the

specimen broke slightly before the same blend with untreated potato starch. In the sample with 60% concentration of starch, the opposite happened. Instead, EcoflexTM 00-30 and EcoflexTM 00-50 showed better resistance to stress, even when blended with potato starch, which reduced their maximum tensile strain to 65–70%, with respect to the pure base elastomer. In EcoflexTM 00-30 (EcoflexTM 00-50), the maximum tensile strain changes from 11.77 (9.29) mm to 7.69 (6.55) mm when blended with 50% potato starch powder. The reduction of tensile strain for the samples containing potato starch has been observed also by Contardi et al. (2021). Their samples, created with the same technique had starch/E43 weight ratio of 60:40, similarly to our E43PS60 and E43PPS60. The biocomposites broke at 1.5 mm/mm in both studies. Moreover, their study demonstrated as 45% of the surface of the biocomposite began to degrade after 6 months at sea. Mechanical performances decrease gradually with the increase of potato starch in the blend, there is not such a dramatic loss of resistance to stress, as it happens instead in the E43 and EcoflexTM 00-10 specimens. The influence of the pre-dried potato starch on the mechanical performance of the material remains unclear. In the EcoflexTM 00-50 specimen the blend with pre-dried potato starch performed very poorly in comparison with the same blend with untreated starch (**Figure 5**).

3.4. Discussion

The interface between a vegetable fiber and a polymer matrix is the transition zone where two materials combine mechanically, physically and/or chemically (Zhou et al., 2016). Plant fibers are naturally hydrophilic whilst polymers are hydrophobic rendering the two materials not compatible. Some of the

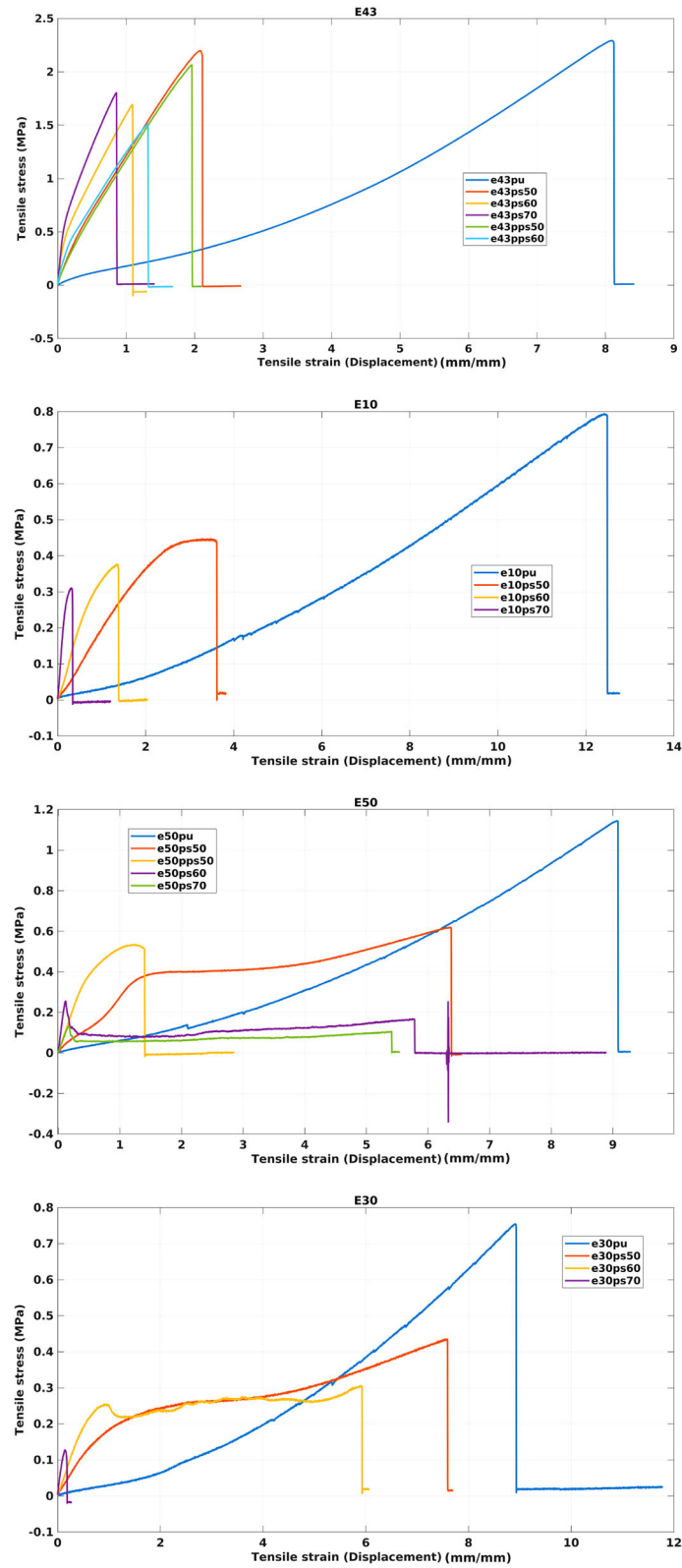


FIGURE 5 | Stress-strain curves of the specimen in bold in **Table 1**. Ecoflex™ 00-30 and Ecoflex™ 00-50, 50% blends maintain most of tensile strain resistance of the pure elastomers.

TABLE 2 | Mechanical traction tests results.

Specimen label	Modulus (Segment) [MPa]	Modulus (Final) [MPa]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [mm/mm]	Thickness [mm]
e43pu	0.36	0.42	2.29	8.090	1.809
e43ps50	1.96	1.57	2.20	2.074	1.232
e43ps60	5.10	4.79	1.69	1.089	0.758
e43ps70	6.63	6.63	1.80	0.856	1.128
e43pps50	1.93	1.39	2.07	1.956	0.975
e43pps60	2.90	2.34	1.51	1.312	1.031
Mean	3.14	2.86	1.93	2.563	1.155
Standard deviation	2.31	2.36	0.31	2.75	0.360
e10pu	0.03	0.09	0.79	12.422	1.311
e10ps50	0.17	0.21	0.45	3.288	0.901
e10ps60	0.30	0.48	0.38	1.358	0.929
e10ps70	1.53	1.75	0.31	0.316	1.05400
Mean	0.50	0.63	0.48	4.346	1.04875
Standard deviation	0.69	0.76	0.22	5.52	0.19
e30pu	0.05	0.14	0.75	8.908	1.201
e30ps50	0.25	0.23	0.44	7.542	0.973
e30ps60	0.34	0.43	0.31	5.894	0.908
e30ps70	1.17	1.42	0.13	0.140	0.961
Mean	0.45	0.56	0.41	5.621	1.01075
Standard deviation	0.49	0.59	0.26	3.86	0.13
e50pu	0.20		1.15	9.065	1.27900
e50ps50	0.41		0.62	6.344	1.12400
e50pps50	0.89		0.53	1.229	0.92800
e50ps60	2.75		0.26	0.120	1.00600
e50ps70	0.84		0.13	0.166	1.14400
Mean	1.02		0.54	3.385	1.09620
Standard deviation	1.01		0.39	4.08	0.14

The table reports specimen label, modulus (segment) [MPa], modulus (final) [MPa], tensile stress at maximum load [MPa], tensile strain (extension) at maximum load [mm/mm], thickness [mm].

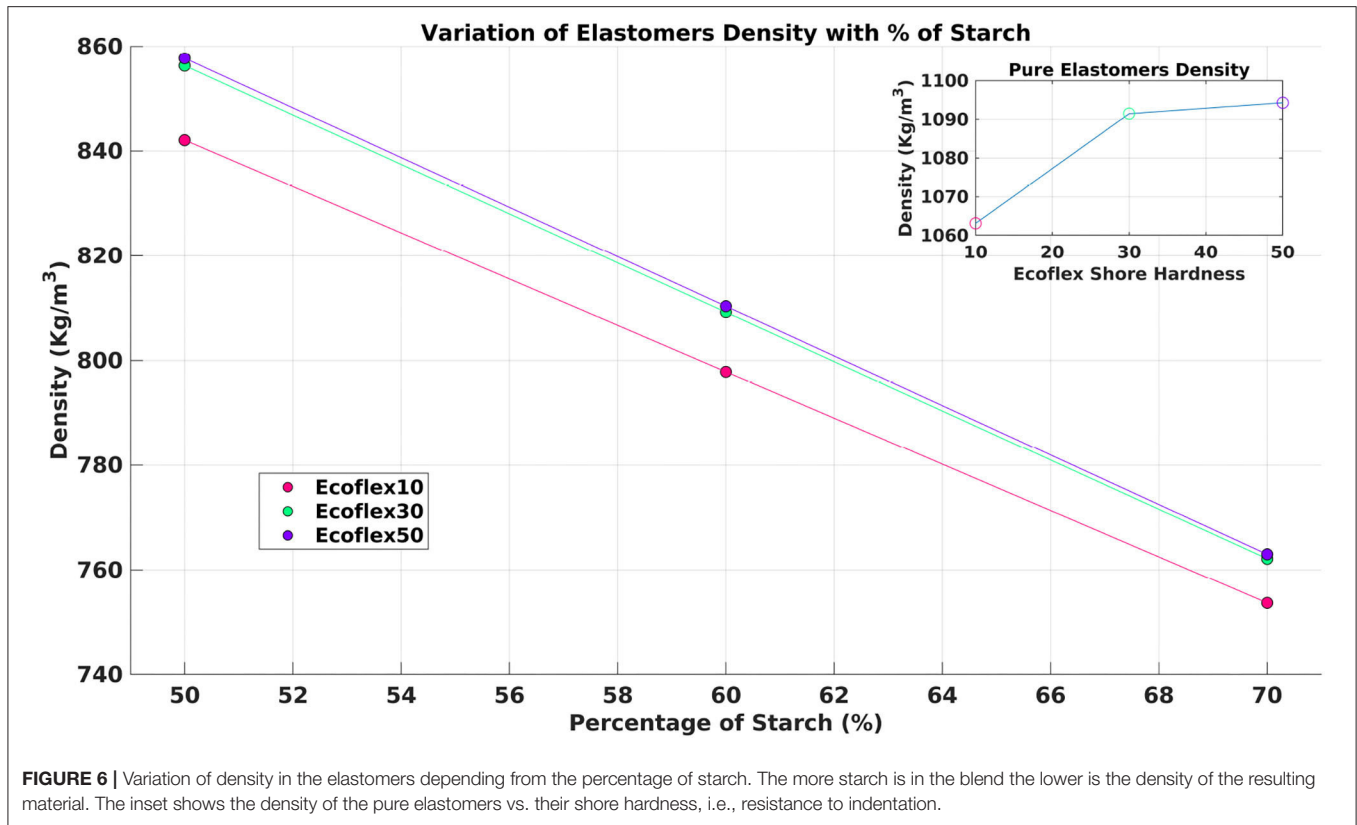
techniques to improve the interface bonding include heat treatment to evaporate the water absorbed from the environment by the vegetable fiber, this would improve the bonding with the hydrophobic silicones. SEM images show indeed finer conglomerates, hence better blending at a microscopic level, mechanically the biocomposite E43PPS60 had an improved tensile strain (Extension) at Maximum Load [mm/mm] from 1.089 to 1.312. Cocoa powder is more hydrophobic than potato starch and contains between 8 and 26% of fat (Gibson and Newsham, 2018), rendering it hardly hydro-soluble. Therefore, the samples containing cocoa powder were amalgamated poorly and showed dense ramifications, making them hard to manipulate.

The more homogeneous and continuous is the blend the better the loads distribute across the matrix. Curves in **Figure 5** are stress-strain curves of E43, EcoflexTM 00-10, ECOFLEXTM, 00-30 EcoflexTM 00-50. Elastomers often report J-shaped stress strain curves, resembling the same curves of biological material, e.g., skin, organ tissue, cartilage. This means that at a lower stress correspond to a higher elongation, but the more tensile force is exerted on the specimen the stiffer it becomes, until

the rupture point. The j-shaped curves can be recognized in the pure elastomers. The stretching characteristics of the elastomeric network are not dominant in the case of the biocomposites (Ceseracciu et al., 2015). In fact the latter materials display a more linear behavior and a reduced rupture strain.

Bearing in mind the scope of our study, it is important to consider that in soft robotics, bioinspiration is an important component, hence the capability of a material to mimic the movement of an animal tissue is important to reproduce locomotion techniques, to ensure resilience and allow dexterity, for instance in the marine environment. For this reason elastomers such as E43 and EcoflexTM are often employed in soft robotics. The Young's modulus of chosen elastomers is in the same order of magnitude of the one of biological materials, i.e., $10^5 - 10^6$ Pa (Rus and Tolley, 2015).

Another important characteristic for aquatic soft robots is buoyancy. The potato starch that we used had a moisture content equal to 18–21%. According to Stasiak et al. (2013) wet (~20% moisture content) packed potato starch has a density equal to 621 Kg/m³, dropping to 283 Kg/m³ for aerated powders. The density of EcoflexTM 00-30 (00-50, 00-10) is equal to 1091.47



Kg/m³ (1094.31 Kg/m³, 1063.12 Kg/m³)², see **Figure 6**. The starch lowers the density of the resulting biocomposite, creating a bigger buoyancy margin, which would play to the advantage of a richer payload.

During the creation of the samples, it became clear that potato starch was better than cocoa powder to create highly biodegradable elastomers. The specimen that contained cocoa were very brittle and too frail to handle. Therefore, we did not include those samples in the SEM imaging or the mechanical tests.

Visually the elastomer-potato starch blends showed little crystals which increased in number and size the more percentage of the blend was constituted by starch. Eventually these crystals became ramifications, e.g., E30PS70, which indicated a very brittle sample.

At the microscopical level, dispersion between the medium and the vegetable fibers is clearly visible. Pre-drying the potato starch leads to a blend with finer conglomerates (~15.6 μm instead of ~37 μm diameter).

The samples that were in contact with distilled water for 19 h displayed early signs of degradation, i.e., frequent flaking (up to 7) and cracking on the flat surface (**Figure 3**), and cave structures (~60.1 μm length) and fractures in the cross section (**Figure 4**). E43PS50W showed visible wrinkling at the base of the starch lump with small cracks (~0.4 μm width).

Mechanical tests revealed that, among the four elastomers that we chose as basis for our experiments (E43, EcoflexTM

00-10, EcoflexTM 00-30, EcoflexTM 00-50), EcoflexTM 00-30 and EcoflexTM 00-50 best maintained the mechanical properties even when blended with 50% potato starch. The maximum tensile strain decreased to 30% in E43 and EcoflexTM 00-10, but only to 65-70% in EcoflexTM 00-30 and EcoflexTM 00-50.

Although silicones are not found in nature, studies demonstrate that their degradation is harmless for the environment, yielding benign silica, water, and CO₂ (Graiver et al., 2003). Polysiloxane is more sensitive to hydrolysis and abiotic degradation, whilst PBAT is subject to biodegradation and shows excellent mechanical properties (Jian et al., 2020). Also, the Executive Summary (Brandt et al., 2012) on Silicon-Chemistry Carbon Balance states that the use of silicones in Europe, USA and Japan led to a reduction of green house emissions of 54 tons of CO₂. Highly biodegradable elastomers can improve the bio-compatibility of elastomers in order to reduce the carbon footprint of marine sciences. Their mechanical and physical properties well compare to those of commonly used elastomers, e.g., resilience, easy manufacturing, versatility. Nevertheless, their environmental impact can be minimized especially for devices that are likely to be lost, or are meant to be left at sea.

4. CONCLUSIONS AND FUTURE PERSPECTIVES

This manuscript shows that a 50% vegetable fiber presence in the blend led to visible degradation after 19 h in distilled water.

²https://www.smooth-on.com/tb/files/ECOFLEX_SERIES_TB.pdf

Moreover, potato starch preserves the mechanical properties of the base elastomer better than cocoa powder, which, instead, made the samples very fragile and challenging to handle. Not all base elastomers maintain their resistance to strain when blended with vegetable fibers. Nevertheless, EcoflexTM 00-30 and EcoflexTM 00-50, 50% blends, reduced their maximum tensile strain to 70% of their capacity, making them good candidates for *in-situ* experiments.

Further analyses could include biodegradability assessment of a material, e.g., Biochemical Oxygen Demand (BOD Test): carried out immersing the material in water (e.g., Mediterranean Water) and measuring the Dissolved Organic Carbon (DOC) and CO₂ content in the air gap in the bottle to evaluate respiration processes in the sample. Nevertheless, BOD tests can not reproduce faithfully the environmental conditions that a given instrument is going to encounter in his life at sea. These conditions are hard to recreate in a laboratory, given the lack of data in the deep sea, such as the variability rates, pressure, salinity, currents, microbiological interaction, light penetration, etc... For this reason real world studies, such as that conducted by Contardi et al. are extremely valuable.

In the real world highly biodegradable elastomers can be already employed to manufacture anchor parachutes and marine sensory skin, as well as short term observations drifters, used to tune the wind settings in coastal and lagoon models. Moreover, the lower density of highly biodegradable elastomers would leave space for a larger payload.

When the soft materials play an important role in the propulsion of a device, the change in density given by highly biodegradable materials can not be neglected. This might constitute a limitation to their use. Moreover, the lifetime of biodegradable instruments would be inevitably shorter than that of traditional observing platforms. Rendering the use of highly biodegradable elastomers more suitable for short term observations or as part of a spatially mindful deployment.

Biocomposites can already be used in traditional oceanographic platforms. However, international exchange the recent employment of soft robotics in the marine environment disclosed a whole new realm, where bio-elastomers can annihilate the footprint of marine observations.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/**Supplementary Material**, further inquiries can be directed to the corresponding author.

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AUTHOR CONTRIBUTIONS

SA organized the international exchange between the University of Edinburgh and the Italian Institute of Technology, designed and performed the experiments, and wrote the present manuscript. MC performed the SEM imaging and conceived the water degradation experiment. MZ performed the mechanical tests. IB organized the international exchange and instructed SA on how to make biodegradable elastomers. FG-S helped with the writing and editing of the manuscript. AS led the acquisition of the financial support for the project leading to this publication. All authors contributed to the article and approved the submitted version.

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SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/fmars.2021.761307/full#supplementary-material>

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