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Core-shell structured nets for biofouling control in aquaculture

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ABSTRACT

This study demonstrates a robust, flexible interpenetrated composite based on 3D spined fabrics as core material and polydimethylsiloxane (PDMS) as shell material. The penetration of the shell component into the core material enables the mechanical interlocking at the micro and macro scale, providing mechanical stability and at the same time, introducing hydrophobic surface properties. Pure PDMS is a well-known biofouling-release material, showing drawbacks with respect to mechanical strength and adhesion-to-substrate, which can be overcome by the presented approach. Nowadays, antifouling strategies for aquaculture nets are realized by using biocide-containing coatings to avoid the attachment of organisms or repel them. Up to now, there are no coatings available on the market that provide adequate biofouling protection for aquaculture nets during the whole production cycle of the cultured stock. Even biocidal coatings exhibit a limited efficiency and need to be regularly cleaned, causing a substantial loss of the coating and increased emissions of biocides into seawater. This proof-of-concept study covers the scope from the design and production of the composite up to the first field tests in the Baltic Sea. The presented approach enabled by material science facilitates a fundamentally different approach in biofouling management and contributes to sustainable aquaculture.

1. Introduction

The rapid build-up of marine organisms on submerged surfaces, the so-called biofouling, is an ever-present process in aquatic habitats that poses a significant problem in the aquaculture industry. Unlike capture fisheries, aquaculture production was continually growing in the past three decades. In 2018 it amounted to 114.5 million tonnes of live weight, which corresponds to the farm-gate sale value of USD 263.6 billion and approximately 46 % of the global fish production (FAO, 2020). The twisted nylon netting used for nets forming the stock enclosure is a highly suitable substrate for the adhesion of marine organisms. It consists of an assembly of fibers bearing specific roughness features and thereby facilitating the settlement of organisms by forming strong adhesion, e.g., by mechanical interlocking of the secreted adhesive inside irregularities of the surface (Crisp et al., 1985; Walker, 1980). In general, the extent of biofouling growth and its diversity depends on geographic location and season, while within one site, such factors as depth of immersion, availability of light, and substrate orientation (vertical or horizontal) play an essential role (De Nys and Guenther, 2009; Fitridge et al., 2012). Furthermore, the growth of biofouling organisms is supported by the surrounding waters enriched in organic and inorganic wastes produced by the cultivated stock (De Nys and Guenther, 2009).

Despite its fast expansion and tremendous importance in providing food, conventional techniques applied in aquacultures to suppress micro- and macroorganisms' settlement on the nets are far from ideal. The state-of-the-art method is the impregnation of nets with copper-containing antifouling paints that produce toxic surroundings near the surface to repel or kill potential biofouling organisms. This leads to large annual emissions of copper compounds that can exert a negative impact on the farmed stock and the surrounding ecosystem (Al-Bairuty et al., 2013; Braithwaite and McEvoy, 2004; Burridge et al., 2010; Farkas and Skarbøvik, 2020; Simpson et al., 2013; Skarbøvik et al., 2017). Besides being environmentally hazardous, copper-containing coatings commonly used in the aquaculture industry exhibit limited efficiency against biofouling and are not able to prevent biofouling for the entire production cycle (Bloecher et al., 2015; Guenther et al., 2010). Therefore, they often need to be complemented with periodic cleaning procedures (Bloecher and Floerl, 2020). On the other hand, if nets are unprotected and not regularly cleaned, large accumulations of

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biofouling (several kilos per m^2) are formed within a couple of weeks (Braithwaite and McEvoy, 2004). Fouling on nets leads to three main problems:

1) Decreased mesh size disrupts water exchange. The lack of fresh, oxygen- and nutrient-rich water inside enclosures, as well as impeded removal of waste (unconsumed feed and fish faeces) can affect the health of the cultured stock (Braithwaite and McEvoy, 2004; Cottee and Petersan, 2009; De Nys and Guenther, 2009).
2) Biofouling can increase the weight of the netting by as much as two-hundred-fold and the drag force five-fold. The heavy weight of biofouling can result in net failure and a complete loss of the cultured fish stock. In addition to the economic loss, this can also disturb the ecological balance and natural gene pool of the surrounding ecosystem (Cottee and Petersan, 2009; Izquierdo-Gómez et al., 2017; Izquierdo-Gomez and Sanchez-Jerez, 2016; Milne, 1970).
3) Biofouling communities can host pathogenic microorganisms and promote disease transmission between wild and cultured stocks (Bannister et al., 2019; Braithwaite and McEvoy, 2004; Cottee and Petersan, 2009; De Nys and Guenther, 2009; Fitridge et al., 2012).

Aquaculture operations are subjected to strict regulations regarding food quality and environmental standards (Maroni, 2000; Wardle and Morris, 2017). Considering the rapid growth of the aquaculture industry and the correspondingly increasing release of copper into the sea, copper-containing coatings may be banned in the future. In 2016 the discharge of copper from fish farming to Norwegian coastal rivers was estimated to be 1088 tonnes (Skarbøvik et al., 2017). According to the RID (Riverine Inputs and Direct Discharges) report in 2020, the direct discharge from aquacultures in Norway has increased steadily during the last ten years (Farkas and Skarbøvik, 2020). Therefore, environmentally benign and easily applicable coating systems are required to minimize breeding costs and protect the ecosystems.

Polydimethylsiloxane (PDMS) is a well-known material as a non-toxic alternative to biocide-containing antifouling coatings for ships (Braithwaite and McEvoy, 2004). Due to its low surface free energy (SFE) and resulting "non-stickiness," PDMS has excellent potential for fouling-release coatings, i.e., coatings that do not prevent marine organisms' settlement but significantly reduce their adhesion strength to the coated surface. Thus, it allows the release of biofouling by mechanical cleaning or by hydrodynamic shear forces generated during the operation of the ship (Hu et al., 2020). However, this same feature (low SFE) leads to the low adhesion-to-substrate, driving scientists to develop composite materials exploiting PDMS properties on the surface while mechanical stability and adhesion to the substrate are provided by another matrix material (Gapeeva et al., 2017; Qiu et al., 2018). In our approach, the problem of low adhesion-to-substrate is solved due to the penetration of PDMS inside the net's core, resulting in mechanical interlocking with the net's yarn. In addition, the elasticity of PDMS allows the net to remain flexible.

In this study, aquaculture nets with a core-shell structure (hereafter named "CleaNet") were fabricated by a dip-coating process. Thereby, commercially available nylon aquaculture nets were impregnated with a PDMS/hexane solution. Containing no copper or other biocides, CleaNet possesses promising fouling-release and easy-to-clean properties, long-term stability, and mechanical durability, which are essential properties contributing to sustainable aquaculture. We should highlight the facile fabrication approach, which is easy-to-handle and applicable at an industrial scale.

A key feature of the method described in this work is the modification of the viscosity of PDMS by using a solvent (e.g., hexane). The two crucial advantages of using diluted PDMS are 1) the drastic weight reduction of the coated aquaculture nets because of reduced coating thickness and 2) the significant enhancement of the mechanical stability and durability of the coating due to the penetration of PDMS into the net's yarn and the formation of an interpenetrating core-shell structure. These features make CleaNet highly suitable for periodic cleaning, which becomes indispensable in today's aquaculture operation (Camps et al., 2014).

2. Material and methods

2.1. Materials and sample preparation

The rubber-like one-component silicone PDMS Elastosil E43 was purchased from Wacker Chemie AG (Munich, Germany). It is based on an acetic acid-curing system and starts curing when exposed to air moisture. The solvent hexane (isomers) was purchased from Carl Roth GmbH + Co. KG (Karlsruhe, Germany). The nylon aquaculture nets (half mesh size 30 mm) were provided by Mechanische Netzfabrik Walter Kremmin GmbH & Co. KG (Oldenburg, Germany).

For the modification of the aquaculture nets, PDMS was diluted in hexane with a PDMS-to-hexane weight ratio of 1-to-1. The aquaculture nets were immersed into the PDMS/hexane solution for 2 min. In the first minute, the nets were manually compressed to ensure a sufficient impregnation by PDMS. Afterward, the nets remained in the solution for 1 min to achieve a smooth shell structure. After the dip-coating process, the impregnated nets were unfolded, hung, and dried under atmospheric conditions at room temperature. The nets impregnated with undiluted PDMS were prepared in the same manner but without the use of solvent.

2.2. Sample characterization and field test

To investigate the nets impregnated with PDMS, the Zeiss Ultra Plus scanning electron microscopy (SEM), equipped with an energy-dispersive X-ray (EDX) detector (Carl Zeiss AG, Oberkochen, Germany) at an acceleration voltage of 10 keV was performed on the cross-section of the net yarn. To prepare the samples for SEM and EDX measurements, all the net samples were cut into an appropriate size with a sharp blade to create a smooth cross-section. The samples were sputtered with gold for 90 s using a sputter coater (Bal-Tec AG, Pfäffikon, Switzerland).

To evaluate the weight increase of the nets after impregnation with diluted and undiluted PDMS, three samples of each variation were prepared and weighed before and after the impregnation process using an analytical balance (Kern & Sohn GmbH, Balingen, Germany).

To evaluate the fouling-release/easy-to-clean properties of CleaNet, three net samples in the dimension of 15cm × 15cm were impregnated with a PDMS/hexane solution. Three uncoated net samples were used as a reference. All the samples were randomly fixed on an AlMg3 frame with cable ties. Those were immersed at 1 m depth at a local fish farm (Baltic Sea, Kiel, Germany) from July 2019 to May 2020 for 46 weeks. The samples were cleaned after 17-week of immersion (manual cleaning) and after 46-week of immersion by a high-pressure water jet (Kärcher Pressure Washer, Alfred Kärcher Vertriebs-GmbH, Winnenden, Germany). The net appearance was recorded weekly by a camera (Olympus TG-4, Olympus Corporation, Tokyo, Japan).

3. Results and discussion

Fig. 1 shows SEM-micrographs and corresponding EDX-maps of a cross-section of the CleaNet's yarn. As silicon (Si) is only contained in PDMS, in EDX-maps, the silicon signal (in red) illustrates the PDMS distribution while the carbon signal (in green) refers to the net's yarn. The net's yarn was completely covered and protected by the PDMS shell with a thickness in the micrometer range. Simultaneously, due to the low viscosity of the diluted PDMS, in the core area, the coating penetrated the net's yarn, resulting in mechanical interlocking between the net's yarn and PDMS. Diluting PDMS is essential to obtain the 3D PDMS-Nylon net interlocking structure. The SEM-micrographs of a cross-section of the net's yarn impregnated with undiluted PDMS is illustrated in Fig. A.1. Without diluting PDMS, only a thick and superficial

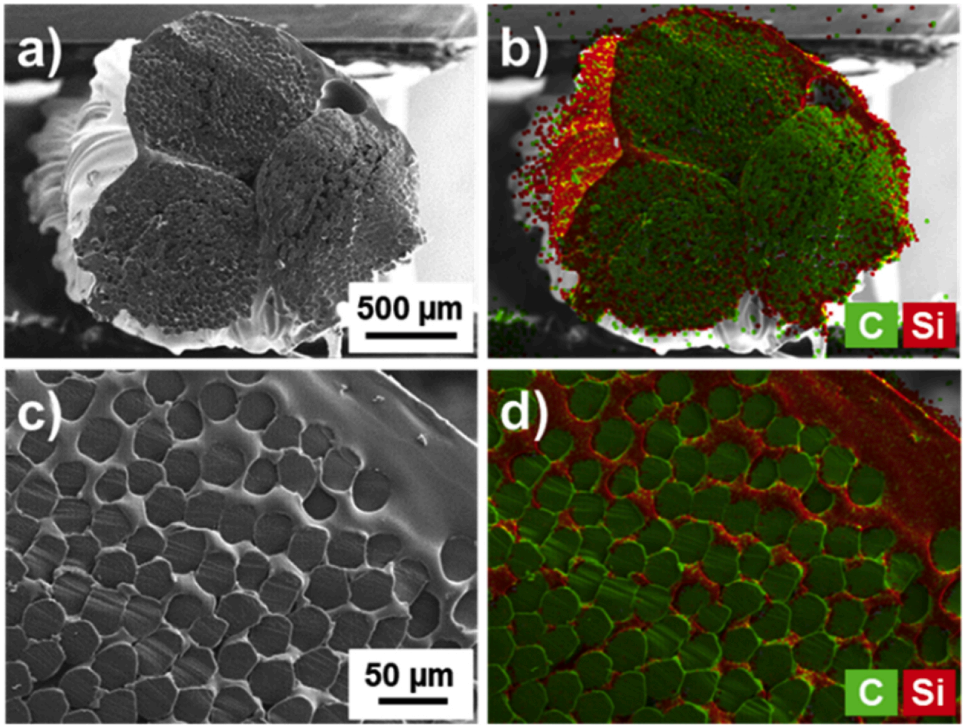

Fig. 1. SEM-micrographs (a, c) and corresponding EDX-maps (b, d) of a cross-section of the CleaNet's yarn. Si signal in red and C signal in green refer to PDMS and nylon net, respectively (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

PDMS corona was formed, surrounding the net's yarn, and nearly no penetration of PDMS into the net's yarn was found due to the high viscosity of PDMS.

To the best of our knowledge, it is for the first time that the interaction between the net's yarn and the PDMS coating was investigated and shown in detail. Although without diluting PDMS, a protective layer surrounding the net's yarn can also be formed, local damage can easily cause the coating to peel off and fail adhesively due to the lack of mechanical interlocking between PDMS and net's yarn. In fact, the long-term mechanical stability and durability of an antifouling coating for aquaculture nets is still an unsolved and challenging problem, especially when the nets are subjected to high mechanical forces (e.g., during high-pressure water cleaning). In contrast to our approach, most of the currently used antifouling coatings for aquaculture nets are self-polishing, i.e., containing seawater-soluble biocidal compounds. Thus, a high-pressure water cleaning results in severe abrasion damage of the copper-based coating and can remove up to 30 % of the coating only during the first cleaning event (Bloecher et al., 2019; Camps et al., 2014).

Besides the just discussed aspect, Swain and Shinjo showed that the weight of a net coated with a commercial copper-based paint increased by 400 % compared to an untreated net due to the cuprous oxide particles contained in the coating (Swain and Shinjo, 2014). In our study, we found that the weight increase of the net coated with undiluted PDMS was approx. 250 % compared to an untreated net, while the weight of the CleaNet (i.e., impregnated with diluted PDMS) increased only by 50 %. The significant weight reduction of the coated net by using diluted PDMS is a significant advantage since it allows to minimize transportation and labor costs and reduces the risk of a net fracture.

Another important aspect is that open-sea net cages and mooring systems often have to withstand adverse environmental conditions, such as strong currents or storms. Therefore, factors contributing to increased drag need to be taken into account. The drag of unfouled nets and nets coated with commercially available copper-based paints was found to be related to the surface roughness (Swain and Shinjo, 2014). In the case of the CleaNet, however, the net surface becomes very smooth due to the formation of a continuous PDMS shell (for details see Supplementary Materials, Fig. A.2), which is beneficial in terms of drag reduction. In addition, increased coating thickness can also influence the drag since it leads to a decrease in the nets' open area. For instance, by using undiluted PDMS, the diameter of the net's yarn after treatment increased by approx. 50 % due to the formation of a thick PDMS shell. In contrast, the impregnation of nets with diluted PDMS has nearly no influence on the diameter of the yarn as only a thin PDMS shell with a typical thickness in the micrometer range (~ 50 μm) is formed.

In order to evaluate the fouling-release and easy-to-clean properties of the nets, biofouling tests were conducted. Both the CleaNet and the uncoated nets were immersed at 1 m depth in a local fish farm (Baltic Sea, Kiel, Germany). Fig. 2 shows exemplary time sections from the immersion test. The experiment started in July 2019, which is the breeding season of mussels, one of the main fouling species on aquaculture nets. After 4 weeks of immersion, the uncoated net was completely covered by young mussels while there were much fewer mussel attachments on the CleaNet (for details see Supplementary Materials, Fig. A.3). A lower degree of biofouling in the case of the CleaNet can be attributed to the fouling-release properties of PDMS. Whenever a shear force (i.e., seawater current) is applied to the net surface, the attached mussels can be easily removed from the surface of the CleaNet. In addition, the PDMS shell sealed the knots and smoothed the surface of the CleaNet which can result in changed flow characteristics around it, thus creating a less favorable environment for biofouling organisms (Baum et al., 2017; Hodson et al., 2000; Swain and Shinjo, 2014).

After 17 weeks of immersion, judging by the appearance in Fig. 2, both the CleaNet and uncoated nets were completely covered by adult mussels. However, in the case of the CleaNet, the mussels were attached to the frame and not to the coated net, as can be clearly seen in Fig. 3b (for details see Supplementary Materials, Fig. A.4, Video A.1). Therefore, the mussels could be easily taken off from the CleaNet as they were actually not adhering to the net, revealing a perfectly clean net surface (Fig. 3d). In contrast, the adhesion of the mussels on the uncoated nets was strong. When mussels were subjected to a mechanical force (i.e., manual removal), the cohesive failure within the byssus of the mussels

Fig. 2. Images of the CleaNet and uncoated aquaculture nets immersed in a local fish farm (Baltic Sea, Kiel, Germany) over time. Both of the nets were hand cleaned after 17 weeks of immersion and with a high-pressure water cleaner after 46 weeks of immersion. For CleaNet after 17 weeks of immersion, the mussels were attached to the frame and not to the coated net, as can be clearly seen in Fig. 3b (for details see Supplementary Materials, Fig. A.4, Video A.1).

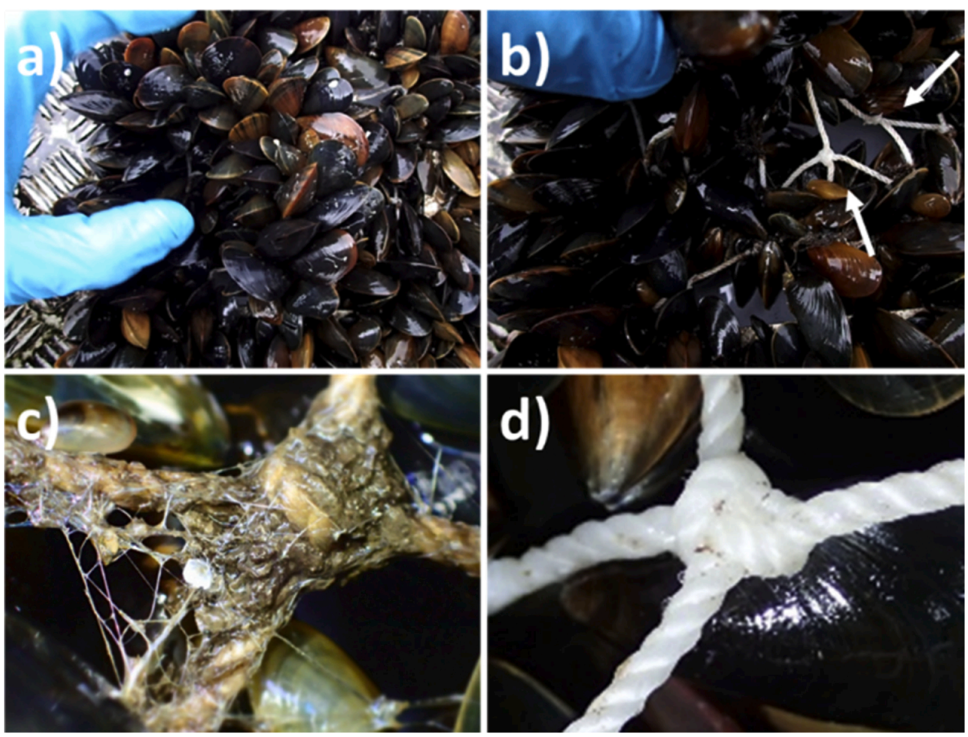

Fig. 3. Images of the uncoated aquaculture nets (a, c) and CleaNet (b, d) after 17 weeks immersion at the local fish farm (Baltic Sea, Kiel, Germany). In the case of the CleaNet, the mussels were not attached to the net and could be easily taken off, revealing a perfectly clean net surface. Mussels' attachment to the uncoated net was strong and their removal led to cohesive failure within the byssus fibers.

occurred as the adhesion strength exceeded the mechanical fracture strength of the mussels' byssus. After removing the mussels from the uncoated nets (Fig. 3c), the residues from mussels (i.e., byssus and secretions) remained on the nets and the colour of the net turned to brown.

All the mussels covering and attaching to the nets were removed manually after 17 weeks of immersion. As already mentioned, the removal of mussels from the CleaNet was easy and fast in contrast to uncoated nets. As shown in Fig. 2, there was almost no biofouling on the CleaNet while the uncoated net was still fully covered with algae, bryozoa, and mussels' byssus after removing the mussels. According to the literature, some organisms can regenerate and proliferate, if they manage to remain on the surface (Bloecher et al., 2019). As demonstrated by Carl et al., hydroid *Ectopleura larynx* uses different methods to maintain their attachment to the uncoated net (i.e., winding of the hydrophyton around threads, growth of the hydrophyton between loose nylon filaments and threads, incorporation of nylon filaments into the chitinous perisarc) (Carl et al., 2011). This type of growth hinders the complete removal of hydroids during the cleaning of nets, and some damaged hydroids may remain on the nets. As shown by Guenther et al., damaged hydroid *E. larynx,* which was not completely removed from the nets, can regenerate its polyps and regrow (Guenther et al., 2010). Moreover, once the nets have been cleaned, hydroids may grow faster than before. Similar concerns have been raised for bryozoa, that re-attachment rate on the surface with fragments of colonial bryozoas can be as high as 100 % (Hopkins et al., 2011). This explains why after 20 weeks of immersion, there were more algae and bryozoa on the uncoated net compared to the CleaNet.

From the 17th week to the 46th week (November 2019 to May 2020), the main fouling species are algae and bryozoa. After 46 weeks of immersion, the occlusion of the uncoated net aperture reached 60 %. It was shown that biofouling organisms covering >60 % of the surface area of a net could cause the net occlusion of up to 100 % (Floerl et al., 2016). With only 20 %, the CleaNet showed much less occlusion of the aperture compared to the uncoated net, without the need to clean it at this condition. This is a promising result since, as previously mentioned, periodic cleaning is required even for copper-based coatings. Currently, net cleaning on Norwegian salmon farms in biofouling-prone regions is conducted every two weeks or more frequently, depending on biofouling pressure (Bannister et al., 2019; Bloecher et al., 2015; Floerl et al., 2016). However, net cleaning is labor-intensive and costly (Camps et al., 2014). For a typical salmon farm with eight uncoated production nets, net cleaning per production cycle (18 months) costs US$ 420 000 (without including personnel costs) (Camps et al., 2014). Since the likelihood of biofouling regrowth is lower on the CleaNet, the net cleaning frequency can be further reduced, and thereby minimizing cleaning costs.

In order to evaluate the fouling-release and easy-to-clean properties and the mechanical stability of the nets, a high-pressure water jet was used to clean the nets (100 bar). As can be seen from Fig. 2, the water pressure was not sufficient to remove all the attached fouling from the uncoated net. This result is in accordance with a previous study, where a pressure of 200–400 bar was needed to remove the fouling from uncoated nets and nets coated with copper-based paints (Floerl et al., 2016). In contrast, the biofouling from the CleaNet surface could be easily removed even at a pressure lower than 100 bar.

As discussed above, for commercially available copper-based coatings, pressure-water cleaning can remove up to 30 % of the coating during the first cleaning, which together with leaching, results in up to 85 % loss of the coating during single service life, causing an annual release of more than 1000 tonnes of copper-compounds into Norwegian waters alone (Bloecher et al., 2019; Skarbøvik et al., 2017). For the uncoated net, the pressure-cleaning caused damage to the nets, which dramatically reduced the service life of the nets. In contrast, the gentler high-pressure water cleaning of the CleaNet did not damage the PDMS core-shell structure and extends its lifetime (for details see Supplementary Materials, Fig. A.5).

4. Conclusion

In summary, nowadays, biofouling control in aquaculture is realized by the application of biocide-containing self-polishing coatings combined with frequent periodic cleaning, which leads to increased biocide emissions into seawater. To make the biofouling management in aquaculture sustainable and environmentally benign, mechanically robust fouling-release systems are needed. Therefore, core-shell structured composite nets (CleaNet) with promising fouling-release and mechanical properties were fabricated by impregnating nylon aquaculture nets with diluted polydimethylsiloxane (PDMS). The mechanical interlocking due to the extensive penetration of PDMS into the net's yarn (confirmed by SEM/EDX investigations) contributed to the long-term mechanical stability of the PDMS shell. Simultaneously, hydrophobic and thereby fouling-release surface properties were introduced to the aquaculture net. The CleaNet composite was successfully tested in a local fish farm for approximately one year and has shown promising fouling-release and easy-to-clean properties with great potential for a ground-breaking change towards sustainable aquaculture.

CRediT authorship contribution statement

Haoyi Qiu: Conceptualization, Methodology, Formal analysis, Investigation, Data curation, Writing - original draft, Writing - review & editing, Visualization. **Anna Gapeeva:** Conceptualization, Methodology, Validation, Writing - original draft, Writing - review & editing. **Sören Kaps:** Conceptualization, Data curation, Writing - review & editing, Supervision. **Rainer Adelung:** Conceptualization, Resources, Writing - review & editing, Supervision, Funding acquisition. **Martina Baum:** Conceptualization, Validation, Resources, Writing - review & editing, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors 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 material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.aqrep.2021.100781.

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