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Cyclosiloxane Hybrid Polymer as a Robust Transparent Eco-friendly Anti-fouling Coating

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Abstract

Anti-fouling coating is the most widely used approach to mitigate the negative impacts caused by marine biofouling. Here, the present study demonstrated cyclosiloxane-hybrid polymer (CHP) as a possible anti-fouling coating for submerged surfaces in seawater. The smooth surface of CHP has a low R_{tm} value of 0.79 nm, reducing the available anchoring point for marine micro-organisms and the extracellular polymeric substances attachment, hence preventing biofilm formation. CHP coating showed generic anti-fouling against micro-organisms of different size and shape. The pseudo-barnacle adhesion tests showed that CHP has a strength of 0.06 MPa, which is lower than that of most coating materials, such as Sylgard 184. Moreover, the CHP coating had an adhesion strength of 1.85 MPa, 2.94 times higher than that of Sylgard 184. After 15 days of immersion, no significant decrease in adhesion strength of CHP could be observed while 33% decrease was observed for Sylgard 184. The robust CHP coating retained its adhesion strength to fiber reinforced epoxy and high transparency after 15 days of immersion in diatom suspension. The present study provides an ecofriendly solution for marine biofouling, and a candidate for further advanced anti-fouling coating design. After 24 h of immersion in concentrated *Navicula exigua* suspension, few cells could be observed on the surface of CHP, whereas much more cells attached to the surfaces of Sylgard 184 and E51.

Keywords: Cyclosiloxane hybrid polymer; Anti-fouling; Coating; Low surface energy; Low surface roughness.
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1. Introduction

The accumulation, settlement and eventual colonization of marine organisms on the submerged surfaces during marine biofouling process has posed serious negative effects to both domestic and naval industries.[1] The undesirable effects, such as surface deterioration, fuel consumption, loss of maneuverability of the vessels, and invasion of non-native marine species to new environments, cost billions of dollars per year in rectification.[2] Anti-fouling (AF) coatings have become the most widely used method to fight marine biofouling since being proposed in the early 20th century. Biocide-based AF coatings and fouling release coatings (FRCs) are the commonly employed approaches adopted by the global marine coatings market.[3,4] The early developed biocide-based coatings, such as tributyl tins were widely used before they were banned by the International Maritime Organization in 2001 due to the toxicity and the detrimental environmental impacts.[5,6] The alternative approach of copper containing biocides is also being regulated due to the poor environment benignity.[7] Non-toxic AF coatings are highly in demand to fight marine biofouling and relieve the pressure on the negative environmental impacts posed by the conventional coating materials. Artificial surfaces mimicking the microtopography of shark skin[8] or crab shell[9] were proven to be a green approach to deter biofouling through impeding microbial attachment.[10] However, to reproduce such surfaces at the magnitude that is suitable for practical application as generic AF surfaces is a huge challenge.

Typical fouling process consists of several key steps: formation of a conditioning film, primary colonization (formation of a biofilm), secondary colonization (fixation of a biofilm), and tertiary colonization.[1,10] The sequence might not be the same, but primary colonization or biofilm formation is often being seen as the prerequisite for subsequent fouling

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process.

Commonly used FRCs relied on non-stick or fouling release properties to weaken the adhesion strength between the fouling organisms and the surface to achieve fouling through external stress generated during navigation or mechanical cleaning.[1] Most of the existing FR mechanisms are targeting the removal of the biofilm formed after the colonization of marine organisms, and very often the failure in full depletion of the biofilm could lead to further fouling problems.[1,7] Counterintuitively, the fouling process will be suppressed through the prevention or determent of biofilm formation at the initial stage when marine organism settlement occurs. Extracellular polymeric substances (EPS) secreted by marine micro-organisms (i.e., diatoms) is a critical criterion for the formation of biofilm because it provides a “platform” and “adhesion layer” for the subsequent settlement.[11,12] Hence, it is reasonable to assume by detaching EPS prior the formation of biofilm, fouling process can be effectively prevented. This has inspired us to develop an AF coating aiming at reducing the attachment points for micro-marine organisms, hence to prevent fouling at the biofilm formation stage. In this work, the present study probe into marine biofouling progress and come up with a new theory in anti-fouling coating: The present study demonstrated for the first time the highly crosslinked and highly optically transparent caged structure of silicone-based polysiloxane (CHP) can be used as an anti-fouling coating with the facile application method. The adhesion between the extracellular polymeric substances (EPS) secrete by marine organisms and the coating surface was minimized through the dual action of low surface roughness and low surface energy of CHP, hence inhibit the formation of biofilm – a prerequisite for fouling. Besides, the environmental benignity, robustness and high optically transparency provides an excellent option to novel marine coating development that requires optical transparency, which is still a challenge in the fouling coating advancement in both the scientific and industrial fields.

2. Results and discussion

Figure 1 exhibits the comparison of three different coating materials of (a) CHP, (b) Sylgard 184, and (c) E51 in molecular structures, naturally formed surface topography, contact angles, and surface energy. To overcome the performance bottleneck of PDMS, for example, the poor mechanical property and the low adhesion between the coating and substrate,[13,14] while maintain its AF properties like low surface energy and low modulus,[15,16] the present study chose cyclosiloxahybrid polymer (CHP), an “extremely crosslinked PDMS” network with a smooth surface (roughness<1 nm)[17-19] as the candidate for AF coating. The AF properties of CHP, crosslinked PDMS (Sylgard 184) and epoxy (E51) were compared. CHP and Sylgard 184 are both hydrophobic with similar surface energy and distinguished by the surface roughness. E51 has a smoother (Fig. 1c and Fig. S1) and hydrophilic surface due to the presence of amine and hydroxyl groups. The surface energy of E51 is almost doubled compared to CHP and Sylgard 184 (Figs. 1a-c).

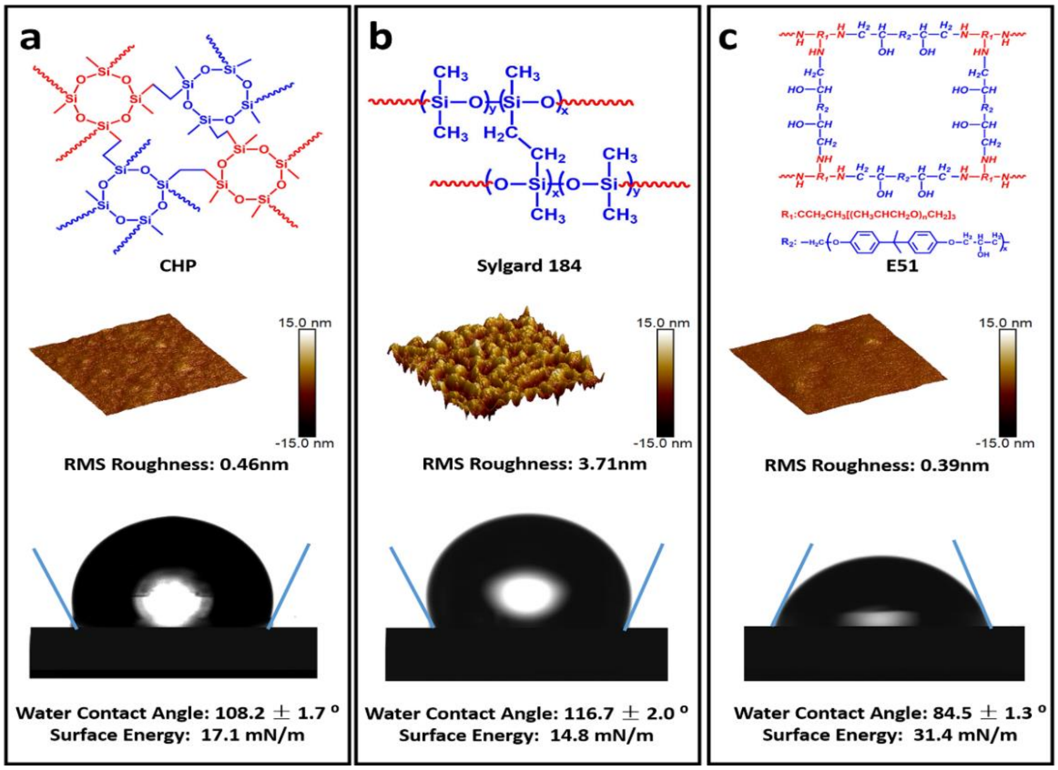

Fig. 1 Comparison of three different coating materials. Molecular structures, naturally formed surface topography, contact angles, and surface energy of (a) CHP, (b) Sylgard 184, and (c) E51. Detailed AFM images and cross section height vs. position curves are available in Fig. S1.

Figures 2a-c compare the number of cells and area coverage for three types of diatoms after wash. To evaluate the generic AF performance against micro-organisms, *Phaeodactylum tricornutum*, *N. Closterium* and *Navicula exigua*, three diatoms of distinct size and shape were used in this study. The number of cells per mm^2 on the PBS buffer solution washed CHP, Sylgard 184 and E51 coated surfaces were counted after immersion in diatom suspensions for 24 h. The number counts of diatoms on CHP surfaces were significantly lower, in the range of less than 15, while the AF performance of Sylgard 184 and E51 were diatom dependant.

CHP, Sylgard coated and bare glass were immersed in marine bacterial suspension for 15 days and washed with PBS buffer solution. The washed surfaces were observed using SEM as shown in Figs. 2d-f. The marine bacteria surface coverage on CHP coating was reduced from almost 100% to barely 3% (Fig. 2h). The AF performance against *Navicula exigua* were compared with various PDMS based polymers and silicone/nanofiller composites, as shown in Fig. 2g.[20-23] CHP coating has surpassed most of the conventional silicone based AF coatings in terms of diatom count per mm^2. Lower diatom count was obtained through the addition of nano-magnetite,[21] however, at the expense of facile preparation process.

Navicula exigua was shortlisted as a diatom representative to investigate the AF mechanism on CHP surface for its high adhesion strength to submerged artificial surfaces in seawater.[20-23] After 24 h of immersion in concentrated *Navicula exigua* suspension, very few cells could be observed on the surface of washed CHP, whereas much more cells were found to attach to the washed surfaces of Sylgard 184 and E51. (Fig. 3a) By following the motion of a single cell, the present study found that the gliding trajectory of an individual *Navicula exigua* near a flat surface is strongly influenced by the surface topology.

The trajectory of *Navicula exigua* cell on the surface of CHP was the longest (Fig. 3b1) while the cell on the surface of Sylgard 184 was hydrodynamically trapped in circular trajectories (Fig. 3b2) during the 240 s period. The stably entrapped cell will serve as the initial point for the development of a conditioning film and the subsequent biofilm.[24-28] *Navicula exigua* cell on the surface of E51 shown similar trajectory as CHP, most probably due to the low surface roughness.

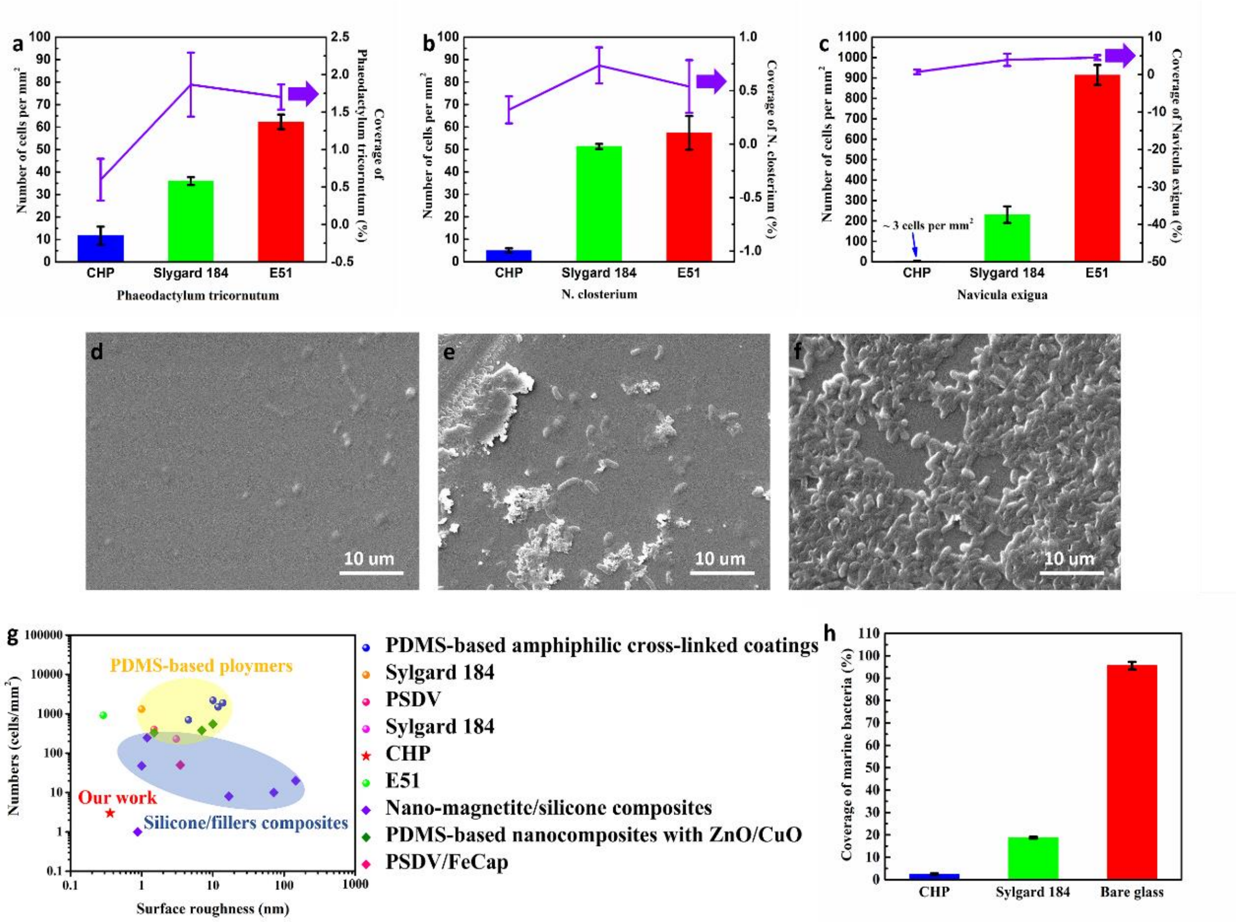

Fig. 2 The anti-fouling properties of CHP, Sylgard 184 and E51 against diatoms and marine bacteria. Coverage and number of cells per mm^2 of (a) *Phaeodactylum tricornutum* and (b) *N. Closterium*, and (c) *Navicula exigua*. Coverage of marine bacteria and SEM pictures of related samples (d) CHP, (e) Sylgard 184, and (f) bare glass, which were pre-treated by immersed in bacterial suspension for 15 days and washed with PBS buffer solution prior surface morphology characterization. (g) Comparison of anti-fouling properties of different materials against diatoms. (h) Coverage of marine bacteria of CHP, Sylgard 184, and bare glass.

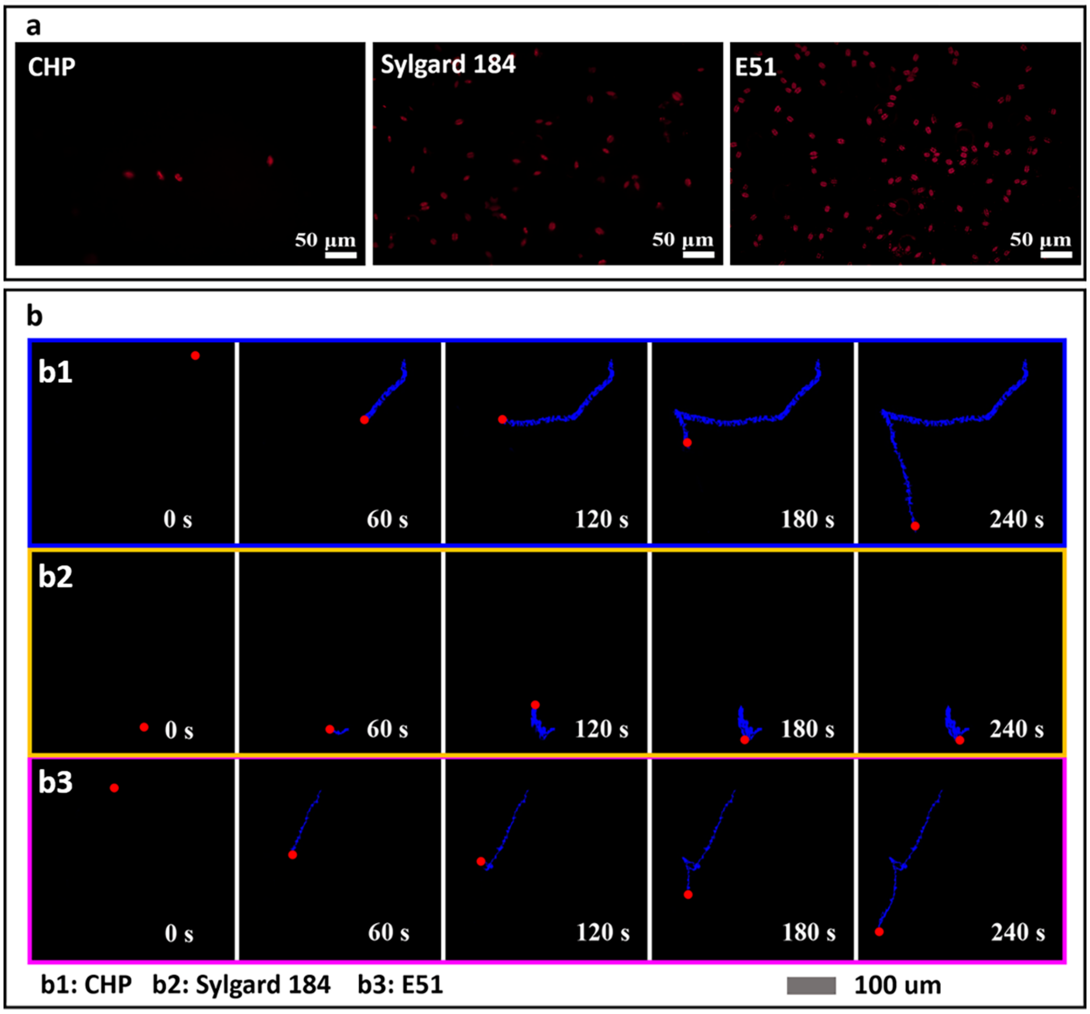

Fig. 3 Fluorescence microscope images of *Navicula exigua*. (a) Fluorescence microscope images of *Navicula exigua* on the surfaces of coatings. (The samples were immersed in concentrated *Navicula exigua* suspension for 24 h before imaging) (b) Trajectory of *Navicula exigua* on the surface of CHP (b1), Sylgard 184 (b2) and E51 (b3). The trajectory was extracted from Movie 1 (CHP), Movie 2 (Sylgard 184), and Movie 3 (E51). The red dot represents the instantaneous location of the *Navicula exigua* and the blue line shows the trajectory of *Navicula exigua* while swimming near surfaces.

However, the adhesion between EPS secreted by *Navicula exigua* and the hydrophilic nature of E51 surface caused the trajectory to be slightly "hinderd", evidenced by the shorter travel path. (Figs. 3b2 and 3b3) Movie recordings for the movement of *Navicula exigua* cells on the three surfaces are available in support information.

Based on the above observations, the AF performance of CHP might be achieved through the simultaneous effects of low surface roughness and low surface energy, which reduced mechanical interlocking and inhibited the secreted EPS from adhering to the surface, respectively. A typical fouling process of a submerged facility starts with the formation of biofilms resulted from the mucilaginous EPS attachment on surfaces. CHP could impede fouling at the initial stage by preventing the adhesion of EPS and diatoms. The present study used R_{tm} (mean of maximum peak to valley height) to represent the surface fluctuation amplitude. The R_{tm} of CHP is 0.79 nm, 1/8 of that of Sylgard 184 (6.29 nm). Low R_{tm} value of CHP resulted from small surface fluctuation, indicating fewer anchoring points available for EPS to firmly fix on the surface. On the contrary, large surface fluctuation allowed easy settlement of EPS on the surface of Sylgard 184 and eventually led to biofilm formation (Figs. 4a1-c1).

The images of stained samples before (Figs. 4a2, b2 and c2) and after rinsing (Figs. 4a3, b3 and c3) reveals the differences in interactions between the diatom and the substrate surfaces. Most of the *Navicula exigua* cells and EPS on the surface of CHP were removed (Figs. 4a2 and a3) while almost all cells remained attached on the surface of E51 (Figs. 4c2 and c3). Although quite a number of *Navicula exigua* cells were removed from the surface of Sylgard 184, clear traces of EPS were observed after rinsing (Figs. 4b2 and b3).

The pseudo-barnacle adhesion strength was obtained to evaluate the effects of physic-mechanical properties on the release of macro-organisms on CHP and Sylgard 184 coated surfaces. CHP has a strength of 0.06 MPa (Fig. S2), which is lower than that of most coating materials, such as Sylgard 184 (0.11 MPa, Fig. S2). The lower adhesion could be attribute to

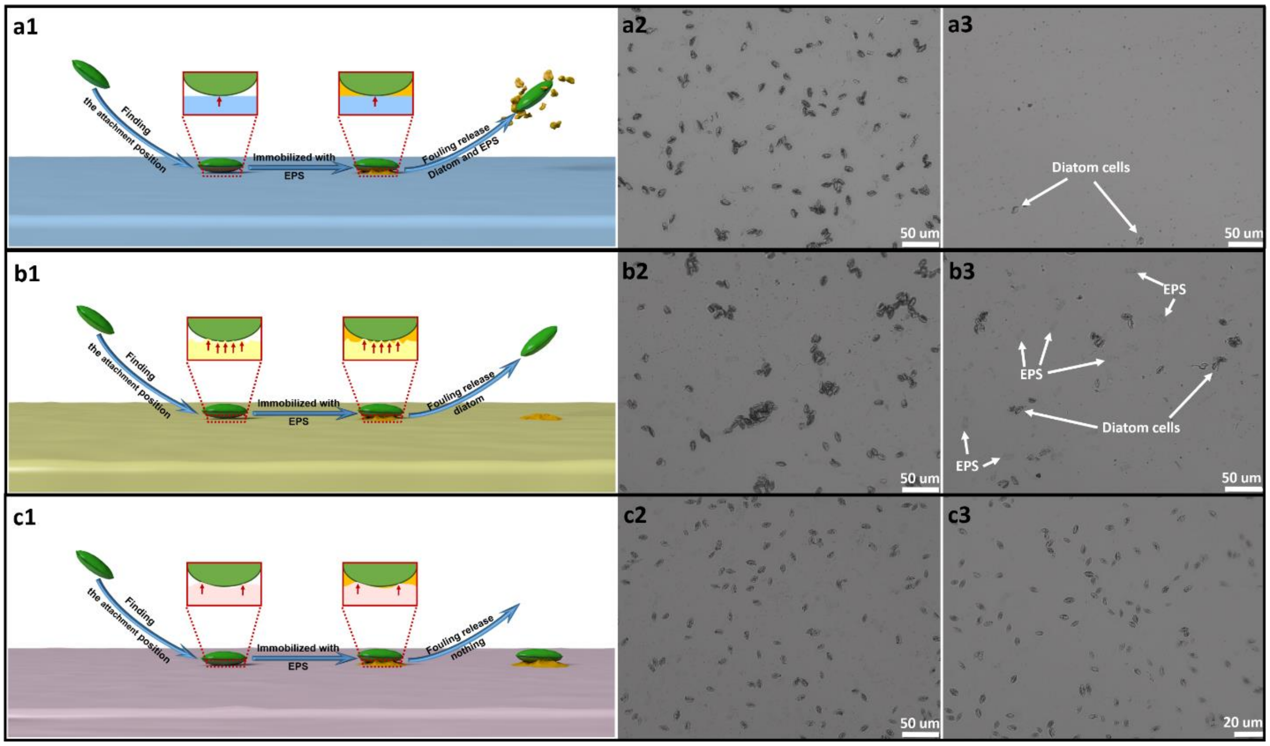

Fig. 4 Proposed anti-fouling mechanism and demonstration by protein-stained coatings after they were immersed in concentrated 24 h. (a: CHP, b: Sylgard 184, c: E51) a1, b1, and c1 are the schematic drawings of anti-fouling mechanism of CHP, Sylgard 184, and E51, respectively. a2, b2, and c2 are microscope images of stained samples which were immersed in concentrated *Navicula exigua* suspensions for 24 h, while a3, b3, and c3 are microscope images of stained samples rinsed by washing bottle.

the well-defined smooth surface of CHP and the highly crosslinked CHP network.

Mechanical properties and surface homogeneity of coatings are of great importance for industrial applications. CHP coating showed a hardness value of 0.23 GPa and a reduced modulus of 1.47 GPa, comparable to the existing epoxy based FRCs. The 5 identical nanoindentation curves obtained at different locations of the surface (Fig. S3) indicated the high homogeneity of CHP coating.

To test the robustness of CHP with respect to the ability to survive under actual service conditions, CHP and Sylgard 184 coated glass fiber epoxy, a widely used composites for sea vehicles and submerged facilities, were subjected in artificial seawater. The pull-off strength of CHP and Sylgard 184 coatings prior immersion were determined to be 1.85 MPa and 0.63 MPa, respectively. After 15 days of immersion, no significant decrease in pull-off strength was detected for CHP while 33% decrease was observed for Sylgard 184. The stable adhesion strength is probably due to the highly crosslinked network with reduced molecular interdiffusion which reduces seawater absorption and expansion, also prevents fouling organisms from penetrating in between the CHP-substrate interface (Fig. S4). The growth curves of N. Closterium populations exposed to bare glass slide and CHP coated glass slide for 15 days did not show any significant difference, indicating CHP coating is non-toxic and eco-friendly (Fig. S5). Besides satisfying the basic requirements for AF application, the CHP coated glass retained excellent transparency (Fig. S6) after placed inside *N. Closterium* suspension for 15 days (Fig. S7) demonstrating potential application for immersed optical instruments.

3. Conclusions

In this work, the present study uses cyclosiloxane based silicone as an eco-friendly AF coating material that prevent fouling at the critical first stage of biofilm formation. The AF mechanism was found to be the combined results of low surface energy and low surface roughness. The smooth surface of the highly crosslinked network significantly reduced anchoring point for micro-organism attachment and facilitate the release of EPS. The marine bacteria left on the coated surfaces shown CHP has a superior AF property, as observed by SEM imaging. Examination of antifouling properties of CHP coating revealed a greater than 90% removal of diatoms for CHP coated substrates. The highly crosslinked network-maintained resistance to seawater absorption and swelling after exposure to culture media for up to 15 days in vitro. The shear adhesion experiments quantitatively revealed that the strength of adhesion between CHP and the substrate remained intact after 15 days of immersion. The non-toxic, robust and highly transparent coating layer effectively resist micro-organism adhesion, demonstrating significant potential in anti-fouling solution for submerged optical surfaces.

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Conflict of Interest

The authors declare no conflict of interest.

Supporting information

Applicable.

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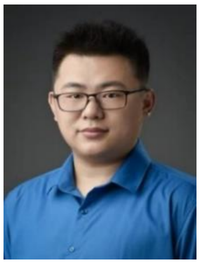

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