PNNL-37141

Scale-up, Field Testing, and Optimization of Nontoxic, Durable, Economical Coatings for Control of Invasive Mussels at Hydropower Facilities

Final Report for CRADA 527

September 2024

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U.S. DEPARTMENT OF ENERGY

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Pacific Northwest National Laboratory
Richland, Washington 99354

Cooperative Research and Development Agreement (CRADA) Final Report

Report Date: September 30, 2024

In accordance with Requirements set forth in the terms of the CRADA, this document is the CRADA Final Report, including a list of Subject Inventions, to be provided to PNNL Information Release who will forward to the DOE Office of Scientific and Technical Information as part of the commitment to the public to demonstrate results of federally funded research. **PNNL acknowledges that the CRADA parties have been involved in the preparation of the report or reviewed the report.**

Parties to the Agreement:

Battelle Memorial Institute, Operator of Pacific Northwest National Laboratory under its

U.S. Department of Energy Contract No.DE-AC05-76RL01830

AND

Dry Surface Technologies LLC

AND

Lorama Group Inc.

AND

Prometheus Innovations LLC

AND

Taylor Shellfish Company, Inc.

AND

BioBlend Renewables LLC

CRADA number: 527

CRADA Title: Scale-up, Field Testing, and Optimization of Nontoxic, Durable, Economical Coatings for Control of Invasive Mussels at Hydropower Facilities

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Sponsoring DOE Program Office(s):

Office of Technology Transitions

Water Power Technologies Office

Joint Work Statement Funding Table showing DOE funding commitment:

	Funding Amounts			
CRADA Parties	**DOE Funding**	**Funds-In**	***In-kind**	**Total**
Participant(s)	$0	$0	$1,172,000	$1,172,000
DOE Funding to PNNL	$1,115,000	$0	$0	$1,115,000
Total of all Contributions	$1,115,000	$0	$1,172,000	$2,287,000

Provide a list of publications, conference papers, or other public releases of results, developed under this CRADA:

Presentation of research results at the Clean Currents 2023 conference in Cincinnati, OH on October 11, 2023.

Presentation of research results at American Fisheries Society Conference in Honolulu, HI on September 15-19, 2024

MCount software to accurately count mussel larvae in images was open-sourced 10/17/2023 and is available at https://github.com/pnnl/MCount.

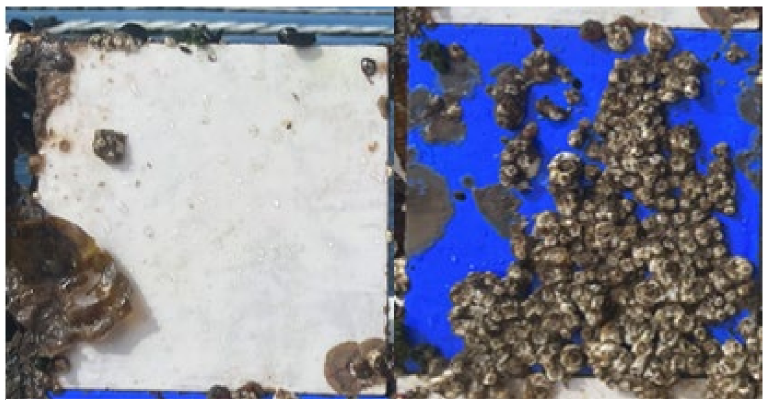
Provide a detailed list of all subject inventions, to include patent applications, copyrights, and trademarks:

This agreement resulted in a new invention disclosure: “A simple one-step process for paintable superhydrophobic lubricant infused composite surface”, IPID 32854-E. Submitted 8/21/2023.

Executive Summary of CRADA Work

In this effort, Pacific Northwest National Laboratory (PNNL) demonstrated the technical maturation of a durable, economical, and nontoxic coating (Superhydrophobic Lubricant Infused Composite, SLIC) that prevents invasive mussels and other unwanted organisms from growing on hydropower and marine structures. The physical characteristics of SLIC were measured using a variety of industry standard methods, just as a commercially available paint would, to show that it can rival other antifouling paints on the market not just in antifouling efficacy, but in structural integrity. SLIC also underwent extensive field testing in diverse field sites across the US to demonstrate that it is effective in varying environments. Engagement with industrial partners showed there is continued interest and need for a cost-effective, durable antifouling coating such as SLIC. Specifically, this project addressed the following technical and commercialization issues:

- Durability and adhesion: Durability demonstrates a coating's capacity to withstand damage from external stressors and/or debris, which should be tested alongside a coating's adhesion capacity. Ideally, a versatile topcoat should be able to adhere well to a variety of existing primers.



Fouling present on SLIC (left) vs Intersleek 1100SR (right) after 12 months of exposure in the Hood Canal. (Photo by Blue Dot Sea Farms).

- Field testing: Long-term field testing in locations with varying environmental factors is imperative to understand how SLIC and similar coatings will operate in real-world conditions over lengthy time exposures.

- Commercialization: SLIC was made in the lab *only* up until this effort, and a commercial partner was needed to help convert the coating into a market-ready product.

In parallel with the commercial maturation of SLIC, the coating has undergone extensive field testing and lab testing for antifouling efficacy as well as physical characterization. The SLIC coating has continued to perform well in each facet previously mentioned, maintaining coating strength similar to or exceeding a leading antifouling paint, and exhibiting exceptional antifouling performance at various field sites over time.

This Phase 2 project built upon the technical success and commercial potential generated during Phase 1 TCF funding by moving the technology from TRL 5 to TRL 6. Commercial licensing of SLIC is expected to follow through one of the project's cost share partners, Dry Surface Technologies, LLC, and they have produced a prototype of SLIC which was subsequently sent to PNNL for characterization.

Acknowledgments

PNNL acknowledges funding from the US Department of Energy's Office of Technology Transitions through the Technology Commercialization Fund. This work is the result of award number TCF-25078.

PNNL also wishes to acknowledge the collaboration and participation of Dry Surface Technologies, Lorama Group, Prometheus Innovations LLC, BioBlend Renewables LLC, Taylor Shellfish Farms, the US Bureau of Reclamation, Blue Dot Sea Farms, and The Mill.

Acronyms and Abbreviations

ASTM: American Society for Testing and Materials
BOR: Bureau of Reclamation
CRADA: Cooperative research and development agreement
DOE: Department of Energy
DST: Dry Surface Technologies, LLC
in: inch
Inc: Incorporated
IP: Intellectual property
LLC: Limited liability corporation
O&M: Operation and maintenance
PNNL: Pacific Northwest National Laboratory
POC: Point of contact
PVC: Polyvinyl chloride
SLIC: Superhydrophobic lubricant-infused composite
TCF: Technology Commercialization Fund
TRL: Technology readiness level

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1.0 Project Summary

This effort focused on technology transfer and commercialization of antifouling coatings with the support of an engaged and collaborative industrial team. Environmental requirements and operational demands call for a nontoxic coating/paint to prevent fouling on hydropower and marine structures. This project built partnerships between PNNL and private companies to optimize, demonstrate, mature, and commercialize a novel PNNL-developed technology that addresses this critical coating need of the hydropower industry.

With focus on commercialization, the project team leveraged prior experience in the Energy I-Corps program. Industrial partners included commercial coating development specialist (Lorama), hydrophobic material manufacturer and paint developer (Dry Surface Technologies), nontoxic biodegradable lubricants manufacturer (BioBlend), and aquatic applications specialist (Prometheus Innovations). Engagement with the US Bureau of Reclamation (BOR), a hydropower operator, throughout the project provided expertise on real-world performance requirements. Taylor Shellfish Farms, Blue Dot Sea farms, and The Mill provided organisms and fouling expertise as well as a perspective of potential broader impacts for the blue economy.

Coated samples were tested to demonstrate performance in a range of environments for key hydropower and marine energy applications. PNNL worked with industrial partners to overcome commercialization barriers and to address manufacturing and regulatory issues.

1.1 Product Innovation, Target Market, Value Relative to Existing Products/Solutions

PNNL's antifouling coating technology is novel and transformative. Prior testing by PNNL and others has shown *no cost-effective technology presently exists in the commercial market* to prevent colonization of hydropower structures, by invasive mussels and other types of damaging organisms that contribute to biofouling. New cost-effective and environmentally acceptable (nontoxic) antifouling coatings are needed for this target market. Coatings applied to hydropower and marine structures are often required to meet legal, regulatory, environmental, social requirements and can affect economic viability of power production through both their capital cost and operations and maintenance expenses.

Growth of organisms on these structures, and the associated weight, drag, and in some case head loss, result in increased loading and reduced power generation, increased operations and maintenance expenses, and decreased life of the structure or system. A coating on these structures that prevents biological fouling would result in significant cost savings, increase reliability, and allow industry to respond to the legal, regulatory, environmental, and social requirements of hydropower energy production.

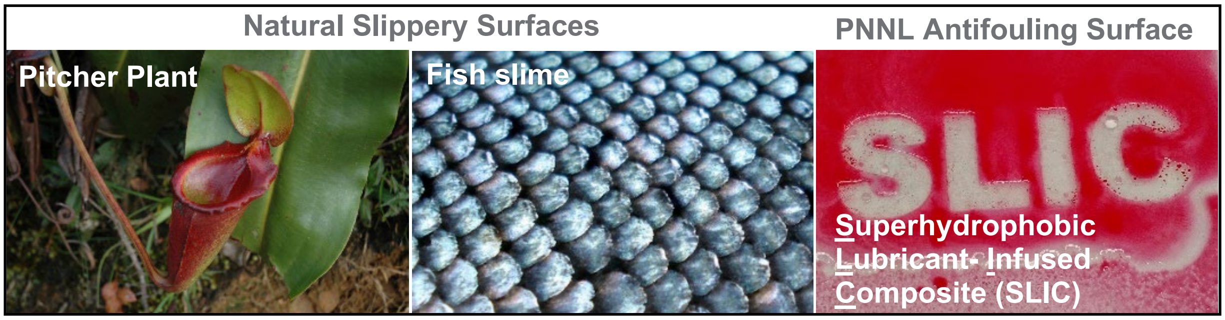

Figure 1: PNNL's antifouling coating is inspired by nature's best fouling prevention strategies. The pitcher plant (left) has a slippery liquid surface at its lip that helps it capture insect prey. Fish scales (center) have a patterned microtexture and surface layer of slippery slime to prevent adhesion. Fine texture on SLIC coating, similar to the self-cleaning lotus leaf, helps to retain and stabilize the slippery liquid at the surface of the coatings.

PNNL has developed and patented a novel coating technology that represents a significant advance in materials engineering. *Superhydrophobic Lubricant Infused Composite (SLIC)* technology uniquely combines multiple antifouling mechanisms to provide improved antifouling performance without using toxic materials. Using an inexpensive nontoxic polymer, with the lowest known adhesion, a sprayable coating has been created that has a "slippery" surface. The slippery surface is the result of an infusion of a nontoxic biodegradable lubricant. This creates a surface that resembles those found in nature (**Figure 1**) and has been shown to be resistant to mussel colonization. In addition to being durable, economical, and nontoxic, the coating is self-healing and has a low-friction surface that maintains efficient hydrodynamics. Results prior to the start of this work showed SLIC to be resistant to fouling from a large range of organisms. A prior Phase 1 TCF effort determined the coating was effective at reducing mussel settling and adhesion. That effort resulted in significant development toward an effective antifouling coating for hard surfaces.

1.2 Competing Technologies and Barriers Impacting Product Acceptance

Many commercially available antifouling paints have at least one of the following shortcomings: they leech toxic compounds into their surrounding environment or are prohibitively costly. Nontoxic antifouling paints have been in increased demand in the past few decades due to the banning of many toxins, but nontoxic technologies are frequently ineffective at antifouling, difficult to mass-produce, and not durable. PNNL's SLIC coating was conceived to address these shortcomings of nontoxic antifouling paints.

Through the Energy I-Corps program, team members extensively explored competing technologies, barriers to acceptance, and pathways to market for this new coating. The Energy I-Corps efforts coupled with insights from Phase 1 and in-depth conversations with the expanded industry team clearly identified the remaining barriers to product commercialization. Phase 2 addressed a different and final set of commercialization challenges including:

- Optimize and demonstrate the durability, operational lifetime, and functionality of SLIC for hydropower and marine energy needs
    - ASTM standard methods were used to characterize SLIC in these areas

- Adjust the coating formulation for scaled-up production and retail
  - Collaborated with Dry Surface technologies, LLC to bring SLIC from the lab into a manufacturing facility
- Demonstrate sustained coating performance under field conditions relevant to hydropower and marine energy
  - Engaged with partners at Blue Dot Sea Farms, BOR, Prometheus, Taylor Shellfish, and The Mill to conduct extended operational testing and performance demonstrations
- Work with industrial partners to identify and provide technical input to address necessary regulatory requirements and certifications
  - This an area of expertise held by the paint industry; PNNL provided technical input, but any additional tests and certifications will be worked into the licensing agreement.

In summary, the team has spent extensive time identifying barriers impacting market penetration and product acceptance by end users. Our industrial partners have helped us address the remaining challenges involved with transitioning SLIC technology into the marketplace.

1.3 Enabling Technology Maturation and Commercial Success

This effort was focused on technology optimization for hydropower/marine energy applications and validation by independent testing (i.e., BOR, Taylor Shellfish, Blue Dot Sea Farms, The Mill, and Prometheus Innovations). Through Phase 2, SLIC transitioned from Technology Readiness Level (TRL)5 to TRL6 and long-term performance data was acquired. The lab and field test data allowed for the optimization of SLIC's formulation to enhance performance (e.g., durability) and packaging, which are key to enabling technology transfer of a mature proven technology to industry and production of a viable commercial product specifically focused for hydropower needs. Phase 2 efforts enabled further technology maturation and technology transfer. Barriers and solutions for technology maturation are detailed above.

For a new coating technology to be commercially viable, it must have utility in a significant primary market or wide range of smaller market segments to attract manufacturer investment and adoption. Fortuitously, the Energy I-Corps interviews also identified many related customer needs and applications that significantly expanded the potential commercial market for the coating, including:

- improved fuel efficiency for ships through drag reduction,
- improved energy efficiency for building heating and cooling units through anti-icing,
- reduced fouling in the piping of geothermal energy plants,
- antifouling for marine energy generation,
- antifouling for fossil fuels extraction,
- antifouling for power plant cooling water systems, and
- reduced maintenance of solar panels resulting from self-cleaning of surfaces.

Industry representatives affirmed that these markets would also benefit from a nontoxic, nonstick, easily applied, and inexpensive durable coating such as SLIC. These larger market segments have provided additional incentives for industry engagement and help support product development for hydropower and MHK needs.

At the end of Phase 2, a prototype commercial product was developed with partner DST and is in the process of being transferred (licensed) to industry.

1.4 Technical Background

Hydropower and marine energy structures inevitably fall prey to biofouling over time (**Figure 2**), and this unwanted accumulation of biofouling causes serious problems including decreased efficiency and increased maintenance costs. Many antifouling paints exist but have a host of problems such as toxicity and cost. Even nontoxic paints on the market have their own issues when it comes to durability, cost, and antifouling efficacy, and SLIC was conceived to solve these issues.

Figure 2: Zebra mussels clogging a pipe within a hydroelectric power plant in Gavins Point, South Dakota. (Photo by Michael Schnetzer, USACE).

SLIC reduces the attachment of organisms found in freshwater and marine environments via low surface energy created by superhydrophobic silica, a hydrophobic polymer, and an environmentally friendly lubricant. Organisms that try to settle on the coating surface will often slide right off, and organisms that do stick to the coating can be easily washed or wiped away which leads to decreased maintenance hours.

Prior to this effort, SLIC was only made in the lab and in small amounts. To be commercially successful, special attention to scale-up was needed in conjunction with industrial partner collaboration.

1.5 Technology Development and Commercialization Efforts

The SLIC coating was developed and demonstrated in laboratory tests at PNNL beginning in 2015. Early lab experiments showed the ability of the lubricant coating to shed water and prevent adhesion of a range of organisms including mussels. SLIC coatings were initially developed for a niche marine application and commercialization was focused on marine antifouling coatings for ships and other vessels. The coating was adapted for use in hydropower and marine hydrokinetic facilities through a fiscal year 2019 TCF Phase 1 award. This effort also entailed extensive testing to demonstrate the ability of the coating to prevent invasive mussel adhesion. In 2020 the project team completed the Energy I-Corps training with the SLIC technology as the focus.

This TCF Phase 2 effort aimed to bridge the gap between laboratory research and industry by elevating SLIC from TRL 5 to TRL 6, entering into licensing talks with industrial partners, and producing SLIC at one of our partners' manufacturing facilities.

2.0 Project Goals and Objectives

This project aimed to mature and de-risk PNNL's SLIC technology and to demonstrate the coating's continued antifouling efficacy in lab and field settings. The following specific objectives and tasks were aimed at maturing a SLIC technology and rapidly removing barriers to commercialization.

Specific Objectives:

- Optimize the current coating formulation for minimal cost, maximum antifouling efficacy, and maximum durability
- Deploy SLIC at various field test sites around the country to demonstrate that SLIC works successfully in a range of environments
- Develop and capture new IP (building on prior IP) to provide secure licensing opportunities
- Engage industrial partners to enable technology transfer and commercialization
- Engage stakeholders to facilitate acceptance and assure environmental compliance of the SLIC technology

2.1 Project Plan

The project addressed the following technical risks:

- Durability and adhesion: Durability demonstrates a coating's capacity to withstand damage from external stressors and/or debris, which should be tested alongside a coating's adhesion to underlying primers. Ideally, a versatile topcoat should be able to adhere well to a variety of existing primers.
- Antifouling efficacy/Lifetime: PNNL developed a novel mussel larvae assay to iteratively test new SLIC formulations, but this information needed to be coupled with long-term field testing for a better overall understanding of SLIC's performance as an antifoulant. Long-term field testing in locations with varying environmental factors is imperative to understand how SLIC and similar coatings will operate in realistic conditions over lengthy time exposures.
- Commercialization: SLIC was made in the lab only up until this effort, and a commercial partner was needed to help convert the coating into a market-ready product.
- Environmental compliance: The market for antifouling coatings is trending strongly towards nontoxic foul release systems. Any new coating must be tested for its overall toxicity.

The project was formulated to minimize technical and commercial risks. Commercial risks were mitigated by using inexpensive, common paint components and traditional application methods to keep costs low and aligned with existing expenses. Commercialization risks were also mitigated by engaging potential industrial partners early and on a regular basis as well as having a highly experienced technology licensing/commercialization professional on the team. The project collaborated with BOR, Taylor Shellfish, Blue Dot Sea Farms, and The Mill, which provided early engagement with the largest future users to facilitate acceptance and early adoption of a new antifouling coating. Creating a new solution to mitigate fouling without the use of toxic materials is challenging. To maximize the probability of success we combined multiple proven antifouling mechanisms into a coating that is damage resistant and has self-healing capabilities.

2.2 Team and Resources

This effort integrated a multidisciplinary project team of experts supported with world class scientific facilities. It included major hydropower operators (BOR) who understand and routinely address fouling problems on hydropower structures and other marine industrial partners that regularly deal with heavy marine fouling (Taylor Shellfish, Blue Dot Sea Farms, The Mill). This effort was supported by a team of cost share partners that represent a significant portion of the commercial ecosystem for coatings for hydropower and marine applications. For example, Dry Surface Technologies and BioBlend supply raw materials to feed into PNNL's coating formulation. Lorama and Prometheus Innovations have experience bringing novel coatings to market. Team members and their roles included:

Lorama Group, Inc. is a leading manufacturer and distributor of colorants and additives in the paints and coatings industry. They specialize in manufacturing and distribution of custom coating solutions. Lorama focused on optimizing PNNL's coating formulation for commercialization, addressing issues related to storage (shelf-life), distribution, and application.

Prometheus Innovations, LLC is a science and technology consultancy with diverse expertise in robotics, renewable energy, and fish protection. Prometheus is a former DOE Fish Protection Innovation Prize award winner that is developing technology to remotely monitor the integrity of fish protection netting.

Dry Surface Technologies, LLC makes custom formulations of superhydrophobic additives under the name Barrian in both powder and liquid forms to improve the performance of a wide range of paints, coatings, and other materials. They harness the power of superhydrophobic technology to protect surfaces from water, ice, and corrosion. These challenges are faced by energy producers operating in extreme environmental conditions. Dry Surface Technologies provided sample materials to integrate with PNNL's coating technology.

BioBlend Renewables LLC is a leading producer of environmentally friendly lubricants used for a variety of applications. BioBlend provided lubricants for testing.

US Bureau of Reclamation (BOR) is the second largest operator of hydropower dams in the US. PNNL collaborated with BOR for field testing of coated samples at Parker Dam on the Colorado River and leveraged BOR staff expertise and experience with antifouling coatings.

Taylor Shellfish Farms, Inc. has been farming in the waters of the Puget Sound for 130 years. They use nets, ropes and other flexible structures to sustainably raise oysters, clams, and mussels. Taylor provided mussels for laboratory testing, insight as a commercial user of aquaculture hardware, and field testing locations.

Blue Dot Sea Farms operates a shellfish and seaweed farm located off the northern coast of the Hood Canal. The site is exposed to heavy tidal flow and offers exposure to a diverse set of fouling organisms for field testing.

The Mill, based in Florida, deploys and tests new technologies for the marine environment. They offered to deploy SLIC samples for field testing at one of their sites in Miami, FL.

3.0 Research Results

3.1 Laboratory Characterization of Coating

One main technical objective of this effort was to characterize the current formulation of SLIC with industry standard methods used by paint manufacturers. The abrasion resistance and adhesion of SLIC were measured and reported.

3.1.1 Abrasion

Ships and power generating structures are often damaged by debris and vessels that come into contact with them. Any coating applied to these structures must be able to resist these forces or else the coating's structural integrity will be compromised. The abrasion resistance of SLIC and a commercial competitor, Intersleek 1100SR, was measured according to ASTM D4060-19 Standard Test Method For Abrasion Resistance Of Organic Coatings By The Taber Abraser. Per the standard methodology, samples of each coating were applied onto 3 in x 3 in acrylic squares (five replicates each) using a 10 mL pipette. Samples were placed into an Elcometer 1720 Abraser (Elcometer USA, USA) and sandpaper (120 grit) was cut into 4 cm x 7.62 cm rectangles and fastened to the universal sample tool (Elcometer USA, USA). Parameters for the abrasion cycles were as follows: stroke length – 45 mm, speed – 54 cycles/minute, mass on sample tool – 500 g, number of total cycles – 1000. Every 100 cycles, the machine was stopped, and the sandpaper on the universal sample tool and the sample coating's surface were brushed with a 4-inch paint brush and a Kimwipe respectively. The mass of the coating plus substrate was recorded before and after 1000 abrasion cycles, and the percent mass retention was reported. SLIC lost slightly more mass during the abrasion cycles when compared to Intersleek 1100SR with a 1% difference in mass retention (**Figure 3**). The abrasion resistance of SLIC was determined to be sufficient at this time and can easily be raised in the future by adding more silica particles if need be.

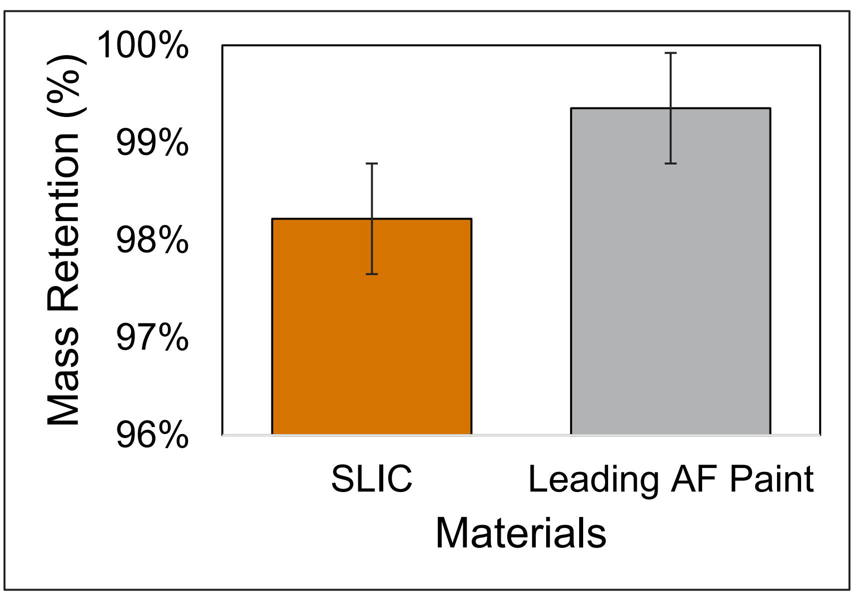

Figure 3: Mass retention of SLIC and Intersleek 1100SR coated on acrylic coupons after 1000 abrasion cycles. Sample size for each material was n = 5 and error is expressed as standard deviation.

3.1.2 Adhesion

Protective coatings are effective only when they are securely attached to their underlying substrate. Reduced adhesion can lead to partial delamination, or worse, total detachment of the coating from the underlying substrate, which is why the adhesive forces between a coating and its substrate must be measured. For extended lifetime, antifouling coatings are often coated over a primer to increase adhesion to the substrate, allowing the antifouling coatings to stay attached far longer than if they were painted directly on a substrate alone. There are a wide variety of aquatic primers on the market today, and a top-performing antifouling coat should be able to adhere to a variety of them. The adhesion strength of SLIC to different primers (Primer 1: Rustlok, Primer 2: Sigmaprime 700 + Sigmaglide 790, Primer 3: Sigmaprime 700, Primer 4: Amercoat 235, Primer 5: Hempel 17630, Primer 6: EP Prime 1000, and Primer 7: Intergard 264 + Intersleek 731) was measured against the adhesion of Intersleek 1100SR to its own primer system (Intergard 264 + Intersleek 731) according to ASTM C1583 Standard Test Method for Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair and Overlay Materials by Direct Tension (Pull-off Method) using a PosiTest AT-A adhesion tester (DeFelsko, USA). Primers were applied to steel substrates according to their respective technical data sheets and overcoat times were appropriately followed. SLIC and Intersleek 1100SR were applied to primed 3 in x 3 in, sanded steel squares (three replicates each) using a 3/8-inch camel hair brush. 50 mm aluminum dollies (DeFelsko, USA) were sanded with 50 grit sandpaper prior to application of cyanoacrylate glue. Permabond POP Primer (Chemical Concepts, USA) was sprayed on the surface of the samples 1-2 min prior to adhesion of the dollies to the coatings. Permabond 268 cyanoacrylate (Chemical Concepts, USA) glue was applied to the bottom of the aluminum dollies until they were entirely covered (~40 drops), and then the dollies were placed onto the samples with light pressure applied to ensure complete glue coverage. The glue was allowed to cure for a minimum of two days, after which a 50 mm serrated cutting tool (DeFelsko, USA) was used to separate the glued coating perimeter from the rest of the coating and the PosiTest adhesion test was executed on the prepared samples. SLIC adhered better to 6 out of 7 tested primers better than Intersleek 1100SR adhered to its own primer system (**Figure 4**).

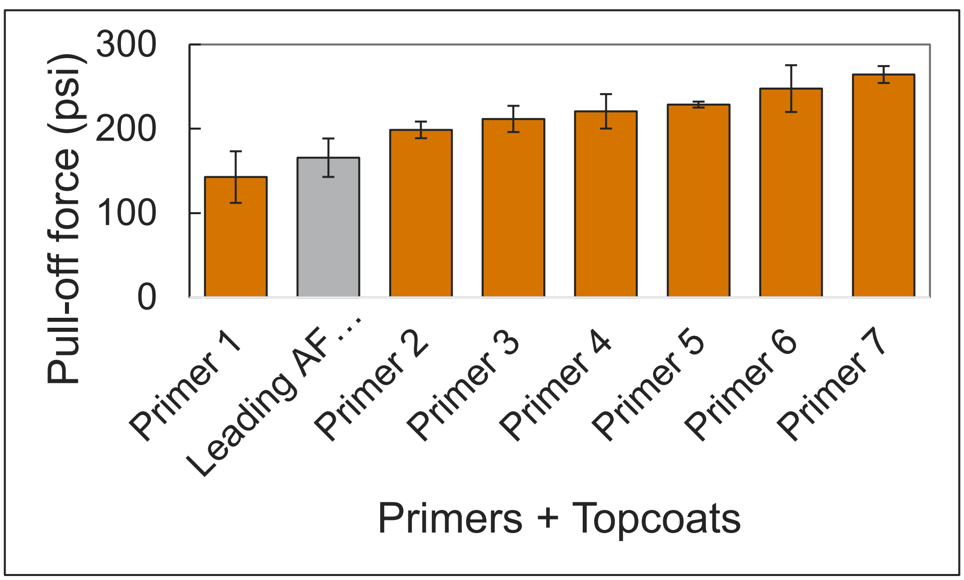

Figure 4: Pull-off force of SLIC on various primers (orange bars) and Intersleek 1100SR on its own primer system (gray bar). Primer identities are described in the section above. Sample size for each material was n = 3 and error is expressed as standard deviation.

3.2 Antifouling Performance Testing

Another main technical objective of this effort was to supplement laboratory antifouling testing with field test data. PNNL's novel mussel larvae assay was used to downselect the best SLIC variants and measure them against an industry leading antifouling paint. The best SLIC variants were then tested in the field at various sites across the country.

3.2.1 Mussel Larvae Testing

Biofouling quantification techniques exist but they often rely on underperforming photo analyzing software. PNNL sought to fix this gap in quantification methods by creating a mussel larvae assay where samples are placed in an aquarium and exposed to mussel larvae over a period of a week (at which point the larvae have settled). The samples are then photographed and subsequently analyzed by counting each individual mussel larva. If a sample can reduce mussel settlement at the larval stage, then those larvae cannot grow into adulthood, and adult mussels will not have to be cleaned off of the sample at a later date. The antifouling efficacy of SLIC was measured against Intersleek 1100SR using PNNL's novel mussel larvae assay. SLIC and Intersleek were coated on 1 in x 1 in acrylic squares (three replicates) and were placed into a 76-liter aquarium alongside other standard materials. The aquarium was kept at 30 ppt salt concentration and ~450,000 mussel larvae (*Mytilus galloprovincialis*) were added into the tank. Larvae were fed 4 mL of Reef Phytoplankton every other day. Coated acrylic squares were spaced out evenly throughout the tank and were exposed to mussel larvae for one week before data collection (**Figure 5**). SLIC exhibited a *7x* reduction in mussel larvae settlement when compared to the leading antifouling paint, Intersleek 1100 SR.

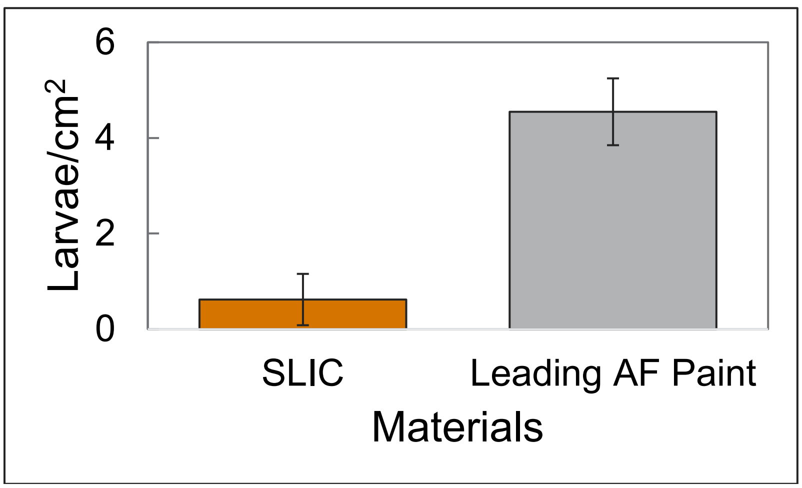

Figure 5: Mussel larvae assay performed on SLIC and Intersleek 1100SR (Leading AF Paint). Both topcoats were coated on 1 in x 1 in acrylic coupons. Sample size for each material was n = 3 and error is expressed as standard deviation.

3.2.2 M-Count

Quantification of biofouling is a complex task involving the counting of organisms that can be broadly defined by two categories: isolated individual organisms, and tightly packed groups of organisms. Manual quantification methods are time-consuming, and existing automatic machine learning-based methods are poorly suited to being utilized by coding non-experts and often lack the ability to detect both categories of biofouling in one workflow. To satisfy the need for a machine learning-based application that is free, user-friendly, and well-suited to the quantification of biofouling, PNNL developed a mussel larvae counting application, M-Count, with a graphical user interface (GUI) that uses a YOLOv8 model for individual organism detection and a thresholding algorithm for grouped organism detection. M-Count was developed for, and demonstrated on, the quantification of mussel larvae on fouled sample surface images obtained from a previously developed mussel larvae fouling assay. Mussel larvae are an important fouling organism because adult mussel colonies form via the settlement of larvae, making the settlement or repellence of mussel larvae a good indicator of a surface's antifouling performance.

M-Count is nearly as accurate as manual counting but accomplishes the task over 60× faster and with exact consistency. Operation of M-Count requires very little coding knowledge and generates an organized spreadsheet containing detected mussel larva quantities for each image in a selected folder (**Figure 6**). M-Count can be trained to detect any type of object in images and is well-suited to marine biological quantification of fouling, which often contains individuals and amorphous films that require parallel and unique detection processes. The M-Count application was open-sourced on 10/17/2023 and is available at https://github.com/pnnl/MCount.

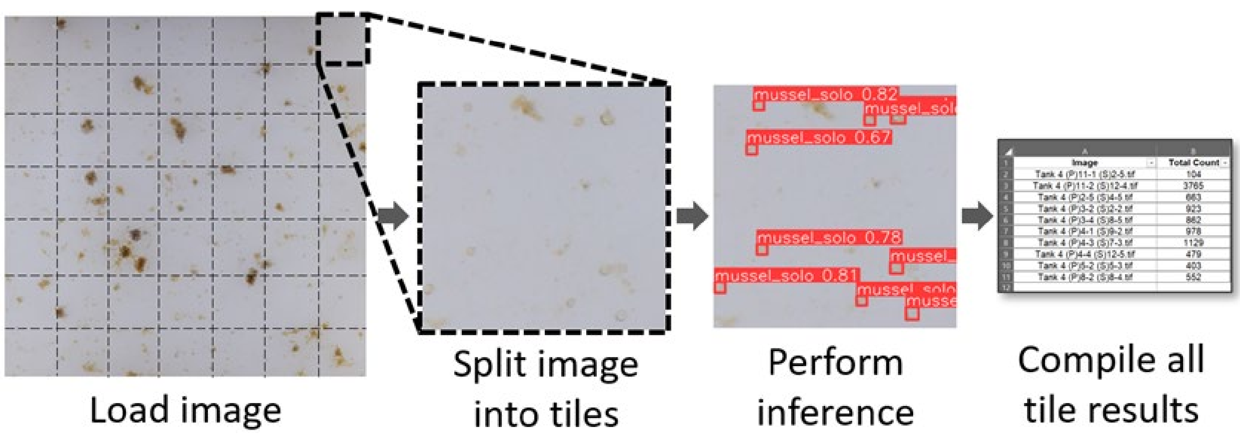

Figure 6: Automatic process flow for the object detection and quantification of non-clumped mussel larvae using YOLOv8 detection in M-Count.

3.2.3 Field Testing

Although lab tests involving SLIC yielded positive performance results, they needed to be supplemented with field test data to capture every aspect of SLIC, including real-world performance and coating lifetime. Several cost-share partners and outside collaborators agreed to test SLIC samples in various test sites across the US, giving SLIC an opportunity to combat fouling in a variety of aquatic environments.

3.2.3.1 Hood Canal, WA

Blue Dot Sea Farms deployed SLIC samples coated on 8 in x 8 in steel plates mounted on a PVC cage off one of their floating oyster farms in the north of Hood Canal, WA. Samples were exposed to moderate saltwater tidal flow for 1 year and were subsequently photographed. SLIC outperformed all controls, including Intersleek 1100SR (**Figure 6**).

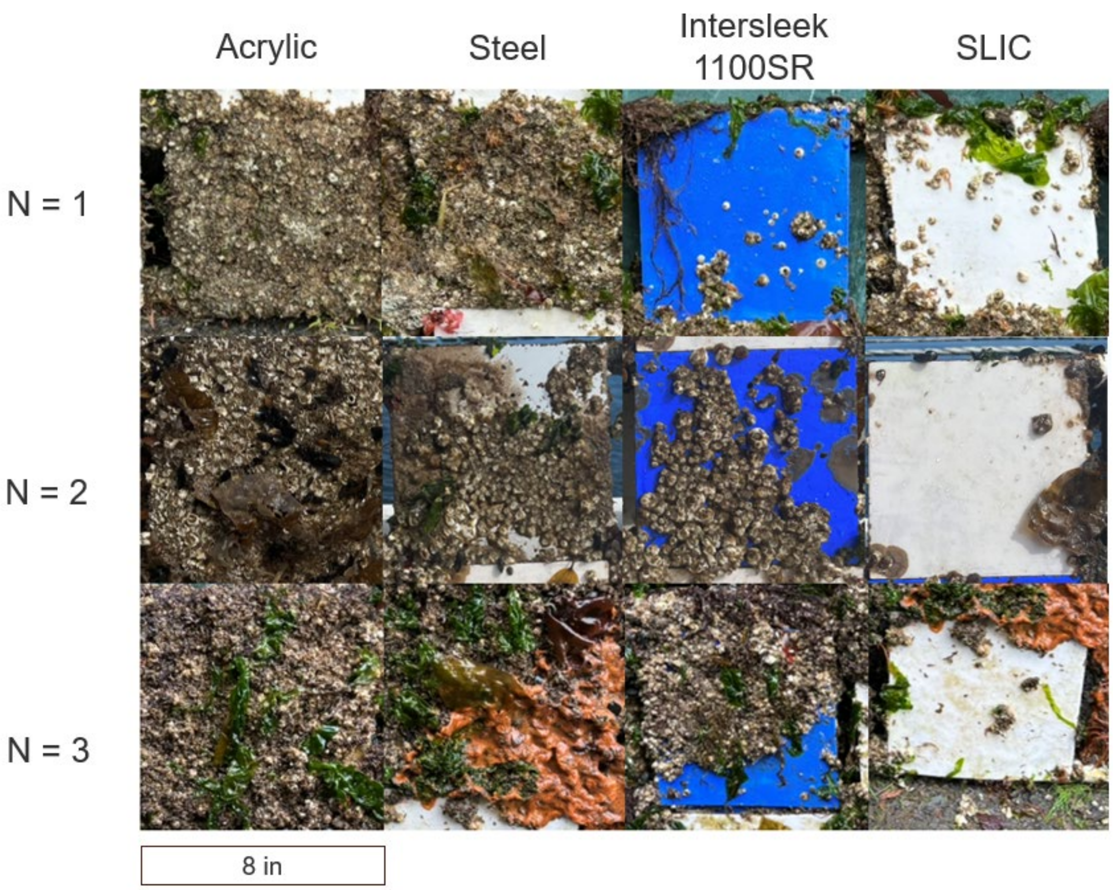

Figure 7: SLIC coated on 8 in x 8 in steel coupons compared to acrylic, steel, and Intersleek 1100SR after 1 year of marine exposure in the Hood Canal, WA.

3.2.3.2 Totten Inlet, WA

Taylor Shellfish Farms deployed an 18-inch buoy coated with SLIC and Intersleek 970 off one of their floating shellfish farms in Totten Inlet, WA. The buoy was exposed to moderate-to-high saltwater flow for 1 year and was subsequently photographed. The SLIC wedge of the buoy coated with SLIC outperformed the wedge coated with Intersleek 970 with minimal fouling after the exposure period (**Figure 7**).

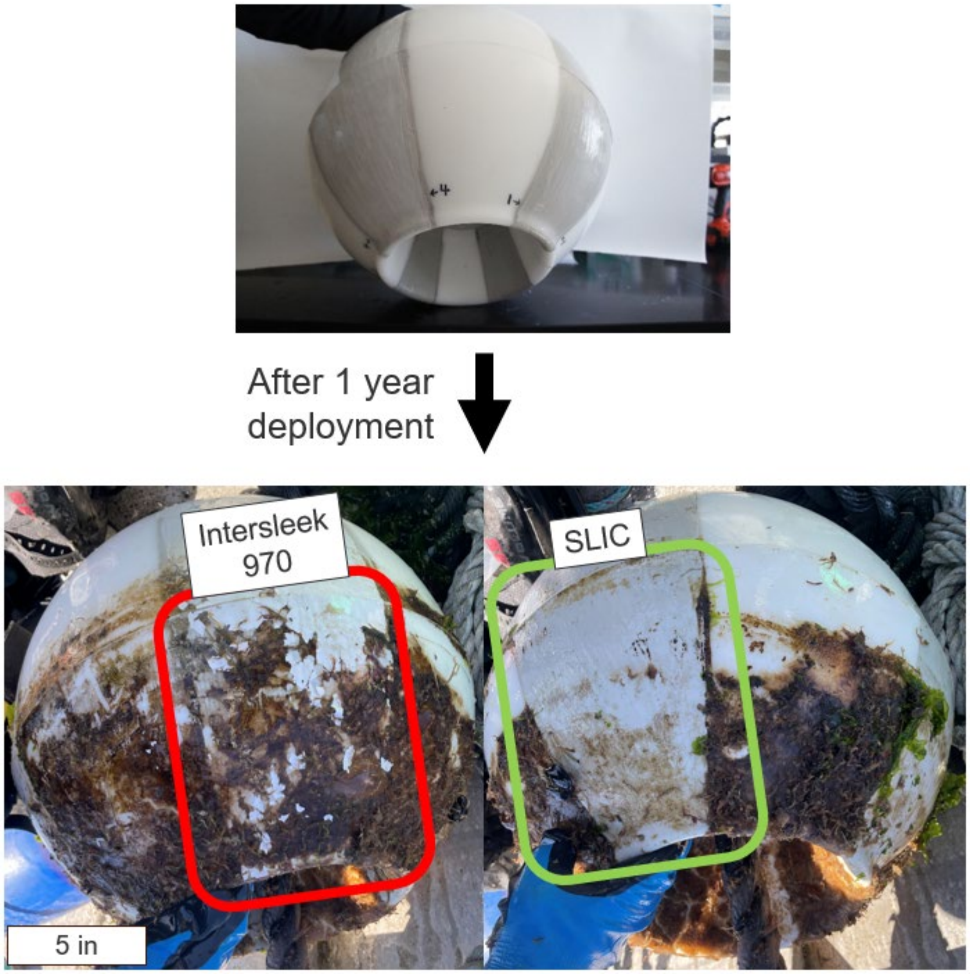

Figure 8: SLIC and Interleek 970 coated on an 18-inch polyethyene buoy after 1 year of marine exposure in Totten Inlet, WA.

3.2.3.3 Miami, FL

The Mill deployed SLIC samples coated on 3 in x 3 in steel plates mounted on a PVC frame off one of their testing sites in Miami, FL. Samples were exposed to low velocity saltwater flow for 1 month and 3 months and were subsequently photographed. Both SLIC and steel samples fouled, but the fouling on SLIC was easily cleaned off with low flow from a common garden hose and restored to near-perfect condition (**Figure 9**). It was also noted that there was a <u>*2x*</u> reduction in the force required to remove barnacles from SLIC vs steel with a force gauge.

Figure 9: SLIC compared to bare steel 3 in x 3 in coupons after 1 month and 3 months of exposure to marine fouling in Miami, FL. Samples were photographed before and after they were cleaned with a common garden hose.

3.2.3.4 Parker Dam, CA

Collaborators at the BOR deployed older SLIC samples (field testing delayed due to COVID) that were mounted on a trash rack in a quasi-static flow area of the Parker Dam, CA.[1] Samples were coated on 3 in x 6 in steel coupons and exposed to freshwater fouling (including quagga mussels) for 6 months, then were subsequently photographed. SLIC samples in the quasi-static flow area were reported to have 0% fouling, were easily cleaned, and free of quagga mussels (**Figure 10A**). It was noted that bubbles were seen under the coating of some SLIC samples possibly from delamination, but this issue has since been solved in successive iterations of the SLIC system. Competitor paints (**Figure 10**(**B-E)**) from various institutions/companies were submerged for varied lengths of time (B: Adaptive Surface Technologies, 3 years, C: North Dakota State University, 5 years, D: Jotun, 9 years, E: Intersleek 970). Paints B, C, and D experienced heavy fouling by quagga mussels and other organisms.

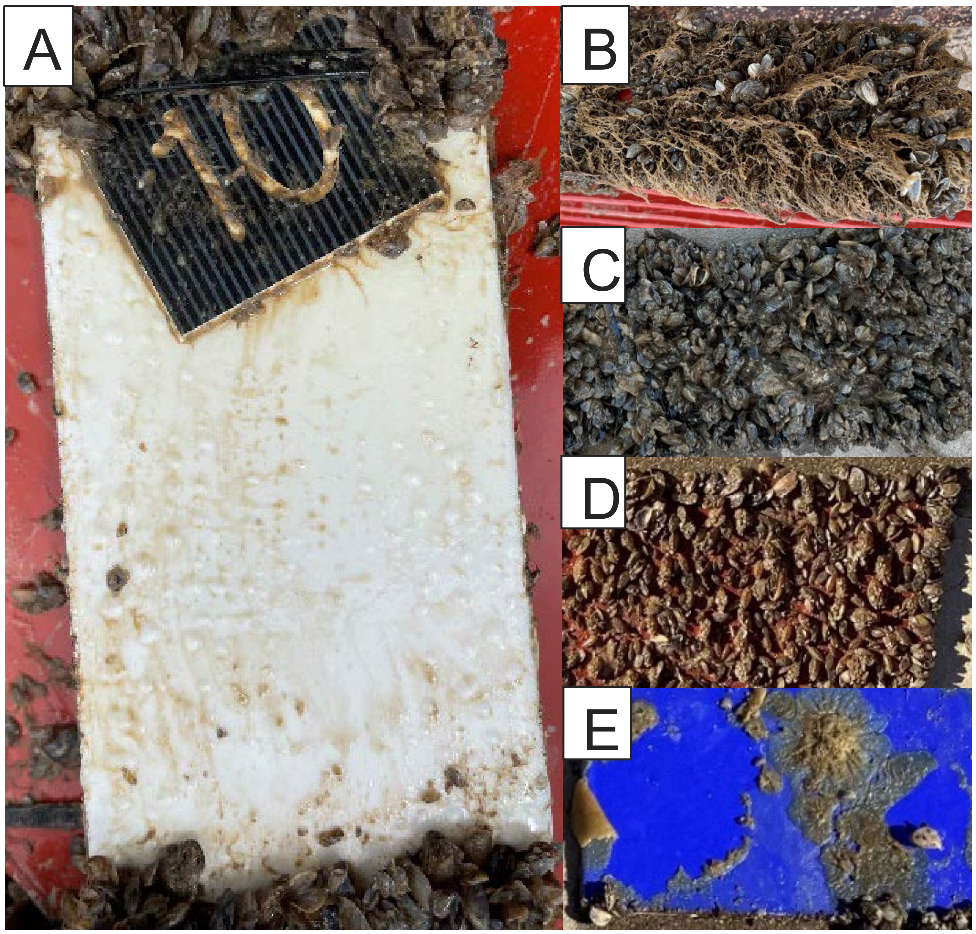

Figure 10(A-E): Antifouling coatings on 3 in x 6 in steel coupons submerged at Parker Dam, CA for various periods of time. (A) SLIC after 6 months of exposure to quagga mussels compared with (B-E) competitor paints (described above) at varying time points. Samples were placed on trash racks in a quasi-static flow area of the dam. (Photos by Carter Gulsvig and Allen Skaja, BOR).

[1] "Development and Field Research on Next Generation Coatings for Mussel Mitigation on Infrastructure" *Bureau of Reclamation*, **2022**, Carter Gulsvig and Allen Skaja

4.0 Commercialization

The main goal for this work was to mature and advance the SLIC coating so it can be transferred to industry for commercialization and introduction to the market. This effort strategically de-risked several key areas of concern. Close coordination with commercial partners enabled us to make substantial progress towards these goals. Dry Surface Technologies dedicated their technical effort to understanding and meeting commercial requirements for the coating. Lorama Group supplied valuable information about blending with pigments and other additives that would be desirable or required for market introduction. Prometheus Innovations focused their effort on additives to SLIC that would provide enhanced functionality such and reflectivity of sonar. Finally, we are grateful for the valuable input from Taylor Shellfish Farms, who provided both expert knowledge of mussel development and behavior, but also provided mussel larvae for testing.

As a result of the work conducted in this effort, PNNL has strengthened its position to license the SLIC intellectual property. Throughout the course of the project, multiple commercial entities contacted PNNL to learn more about the technology and several have agreed to participate in future development, testing, and deployment. Dry Surface Technologies worked with PNNL to create a SLIC prototype produced at their manufacturing site (**Figure 11**), marking major progress towards a version of SLIC suitable for commercial scale production. It is expected that a license to commercialize will follow.

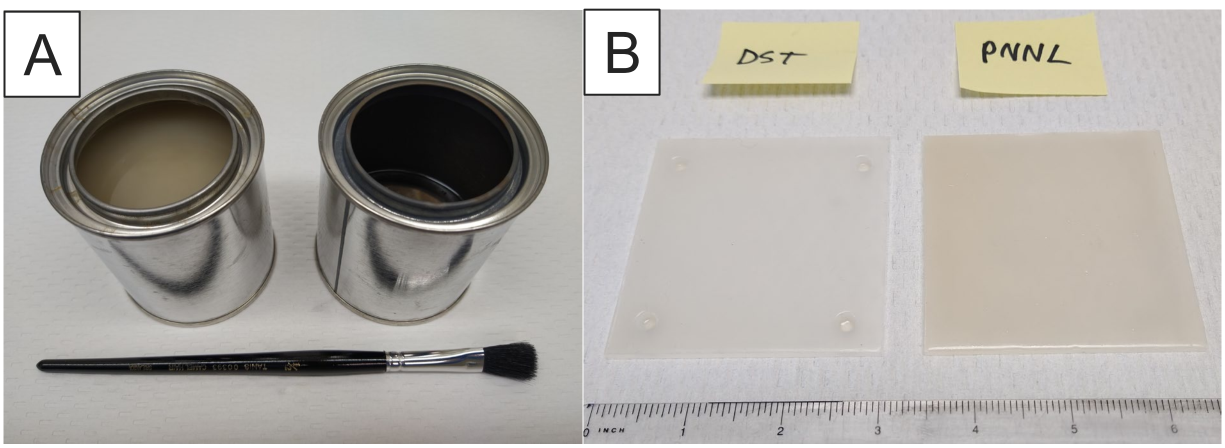

Figure 11(A, B): SLIC made by DST and PNNL. (A) First SLIC prototype coating made outside of PNNL at DST's manufacturig facility and (B) SLIC prototype made by DST compared with PNNL SLIC on 3 in x 3 in acrylic squares.

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