Results of an Ocean Trial of the Symbiotic Machine for Ocean uRanium Extraction

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ABSTRACT: Amidoxime-based adsorbents have become highly promising for seawater uranium extraction. However, current deployment schemes are stand-alone, intermittent operation systems that have significant practical and economic challenges. This paper presents two 1:10 scale prototypes of a Symbiotic Machine for Ocean uRanium Extraction (SMORE) which pairs with an existing offshore structure. This pairing reduces mooring and deployment costs while enabling continuous, autonomous uranium extraction. Utilizing a shell enclosure to decouple the mechanical and chemical requirements of the adsorbent, one design concept prototyped continuously moves the shells through the water while the other keeps them stationary. Water flow in the shells on each prototype was determined using the measurement of radium adsorbed by MnO2 impregnated acrylic fibers contained within each enclosure. The results from a nine-week ocean trial show that while movement of the shells through the water may not have an effect on uranium adsorption by the fibers encased, it could help reduce biofouling if above a certain threshold speed (resulting in increased uptake), while also allowing for the incorporation of design elements to further mitigate biofouling such as bristle brushes and UV lamps. The trace metal uptake by the A18 adsorbents in this trial also varied greatly from previous marine deployments, suggesting that uranium uptake may depend greatly upon the seawater concentrations of other elements such as vanadium and copper. The results from this study will be used to inform future work on the seawater uranium production cost from a full-scale SMORE system.

INTRODUCTION

With global conventional reserves of terrestrial uranium estimated to be depleted in a little over a century,¹ mining of uranium is expected to shift to lower quality sites, leading to higher extraction costs and greater environmental impacts. Fortunately, the ocean contains approximately 4.5 billion tonnes of uranium,² nearly 500 times more than land, and offers an alternative to land-based mining to meet nuclear fuel demand.

The method currently studied by a nation-wide consortium of national laboratory and university partners involves the passive recovery of uranium using polymer-based adsorbents. After initial marine deployment, the polymers are eluted to remove metal ions, including uranium. Following elution, an alkali wash is used to regenerate the polymer, freeing its functional groups, and allowing it to be redeployed in the ocean for reuse. To produce yellowcake (U3O8), the solution undergoes a purification and precipitation process similar to that typically applied to uranium ore.

Previous economic analyses have identified adsorbent production and mooring as the most expensive components of the recovery process.³⁴ Picard et al.⁵ designed a system which targeted cost reductions in the deployment, mooring, and recovery of the adsorbent by coupling the uranium harvester with an existing offshore structure, particularly an offshore wind turbine. In their design, a platform at the base of the 5 MW wind tower supports a belt of adsorbent that cycles through the seawater and through an elution plant located on the platform. The system was sized to collect 1.2 tonnes of uranium per year, a sufficient amount to supply a 5 MW nuclear power plant. A recent independent cost-analysis compared this symbiotic deployment to a reference scheme in which the adsorbent polymer was braided into a buoyant net and deployed as a kelp-field across the ocean floor. This system would need costly servicing by boats for deployment, elution, and redeployment.³⁵ The results of the comparative study showed that the symbiotic deployment proposed by Picard et al.⁵ could achieve a cost savings of 30% compared to uranium produced from the kelp-field like deployment system.⁶

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Recent work indicates that uranium-adsorbing materials with the optimal chemical properties for high adsorbing capacity have inherently low tensile strength and durability, suggesting that the adsorbent may not be strong enough to be woven into a belt as described in the design by Picard et al. The Symbiotic Machine for Ocean Uranium Extraction (SMORE) was developed to overcome this durability issue, utilizing shell enclosures to decouple the chemical and mechanical requirements of the machine. This paper discusses the results of an ocean trial of two SMORE prototypes examining the design followed by a description of the experiment and test site. In addition to the mechanical testing of SMORE, results are shown from the deployment of uranium adsorbent fibers on the test system. While most marine testing of uranium recovery from seawater has been performed under controlled environments, environmental parameters in this study were monitored but not controlled.

SYMBIOTIC MACHINE FOR OCEAN URANIUM EXTRACTION (SMORE) PROTOTYPES

Decoupling of the mechanical and chemical requirements of the offshore uranium harvesting machine was accomplished by a two-part system, shown in Figure 1 (a), comprised of a hard permeable outer shell that serves as the protective element for uranium adsorbent material in its interior. The outer shell has sufficient mechanical strength and durability for use in an offshore environment and chemical resilience against elution treatments, while the adsorbent material is designed to have high adsorbing capacity.

SMORE utilizes shell enclosures strung along high strength mooring rope, resembling conventional ball-chain belts. The belts are strung together to create a net using cross-members which add rigidity and reduce the likelihood of tangling (Figure 1(b)). Large rollers are used to move the nets down the entire length of the turbine. Multiple subsystems are employed (Figure 1(c) and (d)) to achieve a higher device uptime given the lower probability that unforeseen circumstances or complications will cause simultaneous failure of all subsystems. Each subsystem is comprised of an adsorbent ball-chain net, rollers to actuate the net, and tanks, into which the ball-chain net can be rolled for the elution and regeneration processes.

Two designs of SMORE were prototyped at a 1:10 physical scale for prolonged ocean testing (Figure 2(a)) to investigate if movement of the shells through the water column, inducing more seawater flow to the fiber adsorbents encased, would increase the uranium adsorbed. Previous work observed that flow velocities of >5.52 cm/s minimize mass-transfer resistances and maximize adsorbent capacities. These velocities occurred frequently at the ocean test site.

Haji et al. describes the design, fabrication, and assembly of the stationary and dynamic systems. Aside from the bottom support, net, and motor assembly, the dynamic system was analogous to the stationary system. In the case of the dynamic system, the shell enclosure net was moved continuously in a complete loop at a rate of approximately 12 cm/s.
ADSORBENT PREPARATION, DEPLOYMENT, AND SAMPLING

The prototypes utilized the AI8 adsorbent developed at Oak Ridge National Laboratory (ORNL). Details on the adsorbent type, preparation of the fibers, and chemical binding can be found in Das et al.,16 where adsorbent AI11 is identical to AI8 used in this study. These adsorbent fibers consist of hollow-gear-shaped, high-surface-area polyethylene synthesized by radiation-induced graft polymerization to attach a hydrophilic functional group and an amidoxime ligand, which affords the uranium affinity.17 The AI8 adsorbent uses vinylphosphonic acid as the grafting comonomer and amidoxime as the uranium binding ligand. The prepared fibers were dried at 50 °C to achieve a stable weight.

Each enclosure contained “mini braids”, which were preweighed, small masses (80−100 mg) of adsorbent fiber cut from a common braid prepared by ORNL (Figure 3(c)). To determine uranium adsorption as a function of time, samples were collected at 24 h and subsequently every 7 days after deployment for 56 days.

Once collected, the samples were first rinsed in deionized water to remove loosely held sediment, salts, and biological growth. The fibers were then dried at 50°C for 48 h to achieve a steady weight. They were then weighed to compare initial and final weights. The weight before deployment allowed for the determination of adsorption capacity as a function of the adsorbent mass. Weight after retrieval included biofouling (the growth of organisms on the fiber), however it did not account for any loss of fiber incurred during the deployment.

The adsorbent samples were then sent to the Marine Sciences Lab at Pacific Northwest National Laboratory (PNNL) for uranium and other trace element analysis. At PNNL, fibers were digested in 10 mL of high-purity (Optima, Fisher Scientific) 50% aqua regia acid mixture (3:1; hydrochloric:nitric acids) for 3 h on a heating block at 85 °C. A Thermo Scientific ICapQ inductively coupled mass spectrometer (ICP-MS) was used to analyze for trace metals and based on a standard calibration curve. Adsorption (uptake) was determined based on the mass of the recovered elements per mass of adsorbent (g of element adsorbed per kg of dry adsorbent).

Seawater samples were also collected at the ocean testing site at 6 m depth for trace metal analysis. These were collected in HDPE acid cleaned bottles and acidified with nitric acid.
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(Ultrex II J.T. Baker Ultrapure) to 1% and then sent to PNRL for analysis. There samples were diluted in 10% HNO₃ and analyzed using a Thermo ICapQ ICP-MS as described in Gill et al.12

In a full-scale SMORE system, the uranium and other trace metals would be eluted from the Al₈ fibres using a concentrated bicarbonate elution solution15 or another method not yet developed. The fibers would then be regenerated with a sodium hydroxide rinse, after which the fibers may be reused for subsequent uranium extraction campaigns.18,19 The fibers were not reused during this study and therefore the uranium was not desorbed using this process.

**SHELL ENCLOSURE NET**

For both of the prototypes, four lengths of shells were combined to make a single net. The stationary system was comprised of 508 shells while 852 shells were used for the dynamics system. Shells were also designed at a 1:10 physical scale to fit with the prototype. The shell spacing and net dimensions were dictated by the mechanical design of the rollers that engaged with the shells to move them through the water column. Two shell enclosures were tested in this ocean trial (Figure 3(a) and (b)). Each of the stationary and dynamic nets included nine of each design that contained uranium adsorbing fibers. The remaining shells were necessary to ensure proper operation of the mechanical system but did not contain the adsorbent.

On the stationary net, the shells with the adsorbent fiber were placed mid-depth of the system (≈3 m). Adsorbent fibers were also placed in two nylon mesh bags on the stationary net (Figure 3(d)) to serve as a control against those in the shell enclosures. The bags had large (5 mm) mesh sizes to allow water flow but prevent larger organisms (fish) from entering the enclosures as they have previously been observed to nibble on fibers deployed in the ocean.20 Due to the fragility of the fibers in their current form, they are not deployable without a protective enclosure.

**OCEAN TEST MEASUREMENTS**

The prototypes were mounted to a wooden float at the Massachusetts Maritime Academy (MMA) in Buzzards Bay, MA in a low-tide water depth of ≈7 m. Although fairly close to shore, the tides varied up to 1.8 m and the wind generated waves up to 0.9 m high. Additionally, the flow velocity could be extremely strong due to proximity to the Cape Cod Canal, which has currents of up to 2.6 m/s at peak tidal ebb and flow. Physical ocean water properties were monitored between August 10, 2016 and December 18, 2016. Sensors measuring current velocity, temperature, conductivity, and light intensity were deployed on a nearby stationary piling (Figure 2(b)).

Temperature has been shown to have a significant impact on the uranium uptake by the adsorbent where increased water temperatures have been found to have a positive correlation with uranium adsorption.20−22 For the Al₈ adsorbent, a temperature difference of 5 °C can result in a 50% change in uranium uptake.23 Temperature was measured at the piling and on the shell enclosure net using ONSET Tidbit Water temperature loggers. Given that salinity directly indicates the amount of uranium present in the seawater by a well-defined relationship to 238U concentration,24 a Xylem EXO-2 Sonde collected salinity measurements from October 4, 2016 to December 13, 2016. Light has been observed to drastically affect biofouling, the growth of marine organisms, on the adsorbent, impeding uranium uptake by as much as 30%.25 To gather quantifiable data related to biofouling, HOBO Pendant Light/Temperature Loggers measured light at three depths on the piling.

Lastly, previous work has shown that the uranium adsorbed by the adsorbent fibers is dependent on the water flow rate for velocities less than 5.52 cm/s.15 For this reason, current was measured at three depths using Tilt Current Meters from Lowell Instruments. All instruments were calibrated according to manufacturer instructions.

**WATER FLOW MEASUREMENT**

One of the experimental objectives was to determine if increased water flow could be achieved by continuously moving the shell enclosures through the ocean and if that translated to an increase in uranium uptake of the fibers the shells encased. A novel method using the collection and measurement of radium adsorbed onto MnO₂ impregnated acrylic fibers was used to quantify the volume of water passing through each of the different types of enclosures. The details of this method are described in Haji et al.26 In summary, the MnO₂ impregnated acrylic fibers, which adsorb radium, were placed in each of the different types of enclosures in the ocean for approximately 6.25 h. At the same time, seawater was pumped from below the ocean surface at the test site to fill a 120 L container that was then pumped at 1−2 L/min through a control cartridge containing MnO₂ impregnated acrylic fibers. After seawater exposure, the fibers were ashed at 820 °C. The ash was then sealed in epoxy resin while all daughters of 226Ra grew into equilibrium. The samples were then counted for 228Ra using γ-spectrometry by its photopeak at 352 keV.

The known volume of water filtered through the cartridge and the amount of radium adsorbed by the fiber in the cartridge was used to determine a relationship between the radium adsorbed and total volume of seawater to come in contact with the adsorbent fiber.

**RESULTS AND DISCUSSION**

The prototypes were deployed for a total of 56 days, from October 18, 2016 to December 13, 2016. The results describe biofouling, water flow rate, and uranium uptake differences between the stationary and dynamic systems, as well as the physical properties and temporal changes of the seawater at the test site.

**Sensor Data.** Sensors monitored salinity, light, temperature, and currents for the majority of the prototype deployment. As expected, the light intensity dropped off significantly with depth (Figure 4(a)) and is most pronounced in the beginning of September with a difference of about 88% between the upper and lower light sensors. The difference is least pronounced in December with only about 59% disparity between the upper and lower light sensors.

Seasonal variations were also observed in the temperature data (Figure 4(b)). The short-term temperature differences were linked to the tidal and diurnal cycles. Because the incremental adsorption of uranium decreases over time, it is likely that the colder temperatures, which occurred toward the end of the deployment, had minimal impact. The temperature
ranged from a high of 17 °C down to a low of 5.8 °C during the deployment.

The salinity of the ocean test site also varied with tides (Figure 4(c)). Overall, the salinity during the experiment averaged 31.65 ± 0.15 psu, indicating an average 238U concentration of 2.84 ± 0.076 ppb.24

Current meter data does not span the entirety of the deployment due to premature battery failure and programming issues. As seen in Figure 4(d), while there were large short-term variations in the currents (due to tides), overall little seasonal change was observed. In general, the currents peaked at approximately 5 cm/s, with stronger periods of over 10 cm/s. The currents measured were at times much larger than those used to in lab testing of adsorbents, which have been tested in linear velocities up to 8.24 cm/s.15

**Biofouling.** The weight of the fibers after deployment, which accounts for any organism growth, was compared to the weight before deployment to quantify biofouling. Overall, no clear trend was found to indicate that a dynamic system results in less fouling on the fibers. While visually the shell enclosures on the dynamic system showed reduced growth (Figure 5(a) stationary system versus Figure 5(b) dynamic system), this was not reflected in the weight gained by the fibers over the deployment. This may have been due to the fact that the dynamic system was only moving for 37% of the deployment, the majority of which occurred at the end of the trial. Due to the fact that biofouling begins within days of submergence in seawater, the lack of initial movement of the dynamic system may have resulted in similar organism colonization on both systems. All fibers lost material upon initial deployment, of which approximately 5% of the weight loss is attributed to the KOH conditioning process. The additional loss may reflect the fragility of the fibers and possible abrasion during deployment. Some fibers lost weight they had gained toward the end of the experiment (dynamic and stationary design 1) indicating either herbivory or dying of algal matter as temperatures declined. The reduced visual fouling on the shell enclosures is likely due to mechanical rubbing of the enclosures. Further research is needed to identify if it would be beneficial for future SMORE designs to incorporate bristle brushes to clean the shells as they pass, reducing chances of organism growth. Additionally, adding UV LEDs to a point in the adsorbent net’s path could also prevent the formation of biofilm, since UV light has been shown to have strong antibacterial properties.27

However, the variations between the weight gained or lost between the different designs or systems was inconsistent, especially at longer exposure times. Additionally, the dynamic system’s movement occurred during the last two-thirds of the ocean trial, when the water temperature was much colder. Given that the heterotrophic bacterial specific growth rate is positively correlated with temperature,29 the colder water temperature likely inhibited biofouling for all fibers. Thus, the

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**Figure 4.** (a) Light intensity as measured by the top, middle, and bottom light sensors. (b) Temperature as measured from the U24 conductivity logger. (c) Salinity as measured from the Xylem EXO-2 Sonde salinity meter with dashed lines indicating ±2σ where σ is the robust standard deviation. (d) Current as measured from the bottom current meter. The gray rectangle indicates the period of the ocean test, October 18, 2016 to December 13, 2016.
data is not sufficient to deduce whether the continuously moving design would inhibit biofouling of the adsorbent fibers. There exist critical values of current speeds for different species of marine organisms above which fouling biomass is greatly reduced. In general, fouling is not possible at speeds greater than 150 cm/s,28 speeds that would likely damage the adsorbent. Future work should aim to determine adsorbent damage as a function of flow speed and to determine biofouling as a function of immersion time. The results of these studies can be used to determine the speed of movement of the adsorbent net as well as the frequency of other biofouling mitigation. The elution bath may also kill all organisms and hence reduce biofouling, a result that could also be used to determine the frequency of elution.

**Water Flow Rate.** Figure 6 shows the results of the water flow in the shells after correcting for the differences in the amount of radium fibers initially placed in each of the enclosures and adjusting for weight due to ash loss. The results indicate that there was a significant difference in the water passing within the enclosures on the different systems. In particular, the shells on the continuous system had the most water flow, about 57% more water than the shells on the stationary system and 35% more water than the nylon mesh bags.

There was no statistically significant difference between the amount of water passing through shell designs on the same system (Figure 6), suggesting that the design of the shell enclosure has little effect on the amount of water reaching the interior. On the other hand, the difference in water flow to the shell enclosures and the mesh bags on the stationary system varied drastically. Though neither was moving, the bags had approximately 34% more water flow than the stationary shells. This may have been due to the mesh bags being placed at approximately 6 m depth whereas the stationary enclosures were placed at approximately 3 m depth where different eddies could have affected flow. Additionally, the mesh bags have more open space allowing for water flow. Results from a recirculating flume experiment with a linear velocity of 4.8 cm/s showed that a statistically significant difference between the water flow to six shell designs and a control in a recirculating flume did not affect the uranium adsorbed,26 suggesting the uranium uptake of the ocean prototypes will be similarly unaffected by the differences in water flow.

**Uranium Uptake.** To correct for the varying salinity of natural seawater observed over time, all uranium adsorption capacity data was normalized to a salinity of 35 psu given the conservative behavior of uranium in seawater.24,30 As seen in Figure 7, uranium is not the dominant metal adsorbed by the fiber that was analyzed for.

Figure 8 shows the uranium adsorption (g U/kg adsorbent) of the A18 fibers in all the enclosures. The results show that there is very little difference in the uranium adsorbed between the different enclosure or system types. This indicates that the system movement, though it increased water flow to the adsorbent and decreased biofouling on the shells, did not increase the adsorbent uptake of uranium. These results agree with those from a prior study by Haji et al.26 in which a temperature-controlled, recirculating flume experiment of six shell designs found no significant difference in uranium adsorbed by encased fibers, despite drastic differences in water flow rate between shell designs. The results are also in

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**Figure 5.** Biofouling on the shell enclosure net of the (a) stationary system prototype and (b) dynamic system prototype at the end of the ocean test. (c) Percent weight gain or loss in the adsorbent fibers before and after deployment at each sampling. Errors bars indicate ±σ (where σ is the standard deviation) of replicate samples, where not possible they are an average of the percent error.

**Figure 6.** Total volume of seawater to come in contact with MnO2 impregnated acrylic fibers in different enclosure types on different prototype systems as determined by 226Ra count using γ-spectrometry, following the method of Haji et al.26 S1 and S2 refer to shell designs 1 and 2 on the stationary system, respectively. D1 and D2 refer to the shell designs 1 and 2 on the dynamic system, respectively. Bag 1 and 2 refer to the two mesh bags on the stationary system.
agreement with the suggestion by Ladshaw et al.\(^{15}\) that for flow rates >5.52 cm/s the uptake of the adsorbent will no longer increase with increasing velocity.

The maximum uranium adsorbed during this ocean trial was approximately 1.25 g U/kg adsorbent (See Figure S3 in the Supporting Information for details on the total uranium adsorbed by fibers in each enclosure type). This is much lower than has been observed for other ocean tests of the AI8 adsorbent. Specifically, marine testing of the AI8 adsorbent at Broad Key Island (BKI), FL showed a maximum uranium adsorption of over 6.5 g U/kg adsorbent.\(^{31}\) Because uranium adsorption by amidoxime fibers favors warmer temperatures,\(^{21−23}\) part of this difference may be due to the fact that the ambient seawater temperature at the BKI marine testing location was much warmer, ranging from 26 to 31 °C, than the ocean site in this study, which varied from 17 to 5.8 °C. Results from dock tests at the Woods Hole Oceanographic Institution conducted in the same manner as this experiment found the highest adsorption on AI8 fibers to be approximately 2.78 and 2.74 g U/kg adsorbent for experiments averaging 21.7 °C and 18.2 °C respectively.\(^{22}\)

The uranium adsorbed during this study may also have been much less than was previously observed at BKI due to the other metal ions present in the water, specifically vanadium and copper. Vanadium exists at higher molar concentrations than uranium in seawater and has been observed to out-
compete other ions, including uranium, from adsorption, though binding remains unclear. Compared to marine deployments of the A18 adsorbents at BKI (summarized in Table 1), while the vanadium concentration did not differ considerably between the deployments, the average V:U mass ratio adsorbed by the fibers in this study was almost 2.7 times higher than observed at BKI. Figure S4 in the Supporting Information details a time series of the mass ratio of V:U adsorbed for fibers in each enclosure on each system throughout the 56-day deployment. This may be due to the fact that vanadium has been found to load the adsorbent at much higher rates at colder temperatures, with vanadium saturation capacity being almost 14 times higher than the uranium saturation capacity for 8 °C and only about three times higher for 31 °C. Given that the average temperature at the ocean site during adsorbent deployment in this study was 10.9 °C, as compared to 26.6 °C at BKI, it is possible that the adsorbent saturated with vanadium, impeding uranium adsorption.

Marine deployment studies conducted at Woods Hole Oceanographic Institute suggest that copper also greatly impedes uranium adsorption by as much as 54%. The copper concentration during this ocean trial averaged almost twice that measured during the BKI deployment and correlated to a 5-fold difference in average Cu:U mass ratio adsorbed by the fibers. These differences from previous marine deployments suggest that seawater concentrations of other ions may greatly impact uranium uptake and should be investigated further.

Benefits of the SMORE Deployment Strategy. The SMORE deployment strategy allows for the decoupling of the chemical and mechanical requirements of the adsorbent by encapsulating the adsorbent in a hard permeable shell, which protects the fibers inside while also handling any mechanical loads required. A continuously moving SMORE system would allow for the incorporation of elements, such as UV LEDs sections and bristle brushes, that may reduce biofouling on the adsorbent, uranium uptake by fibers in the dynamic versus stationary system did not differ significantly. This was likely due to the fact that the continuously moving system malfunctioned and was stationary for the initial and most crucial point of the fiber adsorption process. A SMORE system still has much potential by allowing for the symbiotic coupling of a uranium harvester with an existing offshore structure, such as an offshore wind turbine, to reduce infrastructure development and possibly reduce cost. Future studies will use the results from this ocean trial to inform analysis of the resulting uranium production cost from a SMORE system.

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Additional information as noted in the text (PDF)

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Notes
The authors declare no competing financial interest.

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