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Environmental Applications of 3D Printing Polymer Composites for Dredging Operations

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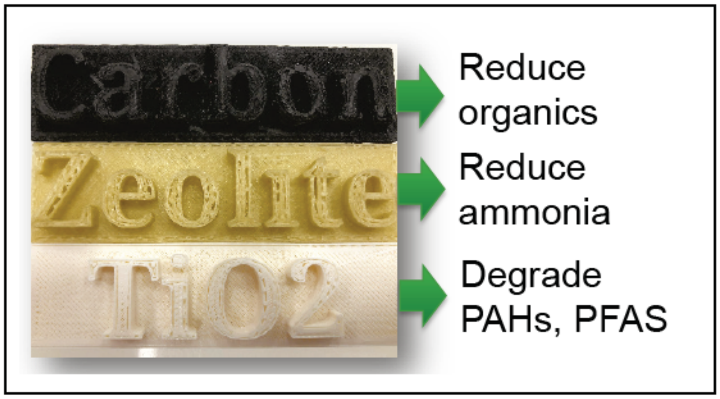

Figure 1. 3D-printed activated carbon, zeolite and photocatalytic titanium dioxide (TiO_2) using polymer binders that may be used for deployable, retrievable and reusable environmental applications to adsorb or degrade contaminants of concern. Graphic by Alan Kennedy.

PURPOSE: This Dredging Operations Environmental Research (DOER) technical note disseminates novel methods to monitor and reduce contaminant mobility and bioavailability in water, sediments, and soils. These method advancements are enabled by additive manufacturing (i.e., three-dimensional [3D] printing) to deploy and retrieve materials that adsorb contaminants that are traditionally applied as unbound powders. Examples of sorbents added as amendments for remediation of contaminated sediments include activated carbon, biochar, biopolymers, zeolite, and sand caps. Figure 1 provides examples of sorbent and photocatalytic particles successfully compounded and 3D printed using polylactic acid as a binder. Additional adsorptive materials may be applicable and photocatalytic materials (Friedmann et al. 2019) may be applied to degrade contaminants of concern into less hazardous forms. This technical note further describes opportunities for U.S. Army Corps of Engineers (USACE) project managers and the water and sediment resource management community to apply 3D printing of polymers containing adsorptive filler materials as a prototyping tool and as an on-site, on-demand manufacturing capability to remediate and monitor contaminants in the environment. This research was funded by DOER project 19-13, titled "3D Printed Design for Remediation and Monitoring of Dredged Material."

BACKGROUND: USACE dredges more than 200 million cubic yards of sediment annually to support vessel navigation to critical U.S. ports and harbors (Verna and Pointon 2003; USACE 2020), a portion of which (e.g., <5%) is considered contaminated (https://odd.el.erdc.dren.mil/; [Suedel et al. 2008]). In addition, USACE manages confined disposal facilities (CDF) containing contaminated sediment (Figure 2A) with discharges to surface waters that fall under jurisdiction of the Clean Water Act (Estes et al. 2010; Schroeder et al. 2008). Other dredged material (DM) discharges include pipeline (Figure 2B) and open water disposal.

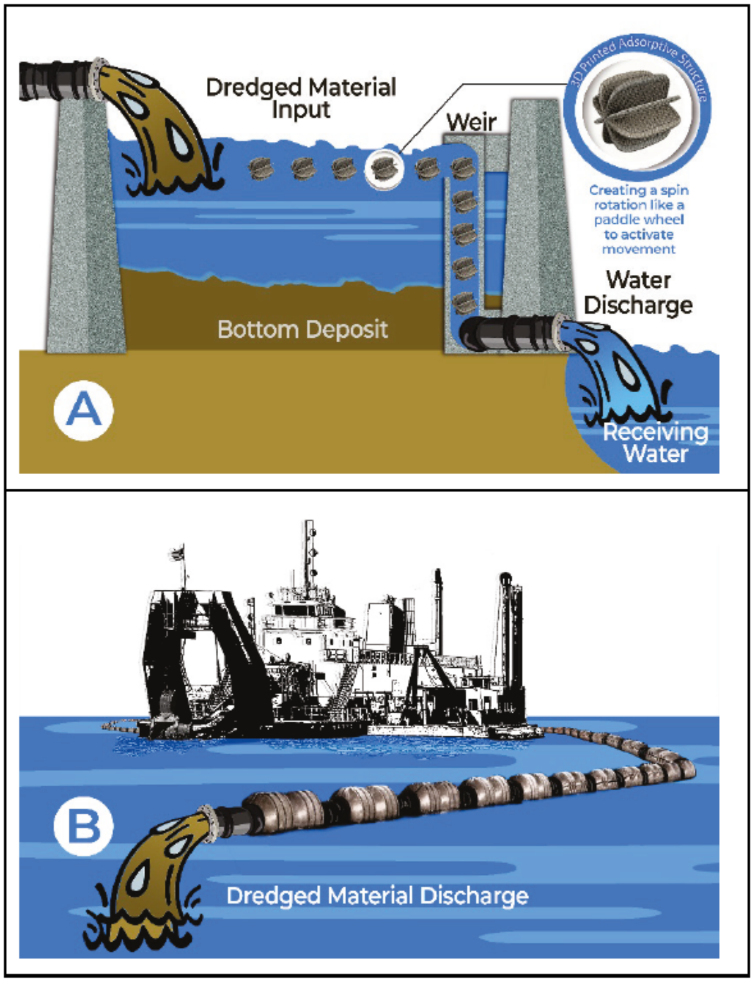

Figure 2. Conceptual model of confined disposal facility containing contaminated sediment discharged to open water (A) and pipeline discharge of dredged material (B). Graphic by Chandra (Pat) Caldwell, USACE, CE-IT.

The diversity of inorganic and organic contaminants in DM and DM discharges is complex and dynamic and therefore requires constant research and development to discover novel, effective, feasible, and low-cost solutions. There is no single solution for legacy contaminants in DM, which include but are not limited to diverse chemical classes such as metals, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), pesticides and organotins (USEPA 1972; Schmidt et al. 2017; Santschi et al. 2001). Further, technologies are needed for emerging contaminants of concern (CoC), including perfluorinated compounds that encompass both hydrophobic and hydrophilic properties (Stroo et al. 2017) and have unique fate and transport that defy traditional contaminant monitoring and remediation strategies (Stroo et al. 2017). Addressing and overcoming these challenges require innovative management strategies in the near term.

While various sorbent powders such as activated carbon (AC) and zeolite reduce the environmental mobility and bioavailability of select chemical classes, there is no single solution for the diverse suite of contaminants present in some DMs and DM discharges. For instance, AC adsorbs PCBs (Cho et al. 2007; Cho et al. 2009; Zimmerman et al. 2005), PAHs (Li et al. 2020), munitions (Boddu et al. 2009) and some metals (Jia, Xiao, and Thomas 2002) while zeolite adsorbs radionuclides and ammonia (Norberg-King 1991; Melby et al. 2018; Burgess et al. 2003; Burgess et al. 2004; Miensah et al. 2020). Additional photocatalytic materials such as nano-scale titanium dioxide (TiO2) in the presence of ultra-violet light can degrade certain chemical classes, including PAHs (Yang et al. 2017; Zhang et al. 2008) and perfluorinated compounds (Cho 2011; Gomez-Ruiz et al. 2018; Wang and Zhang 2011). The lack of any one comprehensive sorption technology for the diverse suite of chemical classes in DM is further confounded by additional application and deployment constraints. For instance, sorbent powders must be used in a treatment column with low aqueous flow rates or must be thoroughly mixed into contaminated sediments to allow effective treatment and to ensure the material remains in-place (Cho et al. 2007; Cho et al. 2009; Millward et al. 2005). Further, traditional environmental application of sorbent powders does not allow recovery of the material/technology once amended to environmental media. Sediment amendments will likely be buried and rendered less effective by new sediment composites and contaminant inputs (Gidley et al. 2019). Active adsorptive surface structure technologies consisting of composites of several different materials (e.g., AC, zeolite, TiO2) may provide a solution. Advancements in research and customizable advanced manufacturing capability may provide a path to overcome the aforementioned constraints.

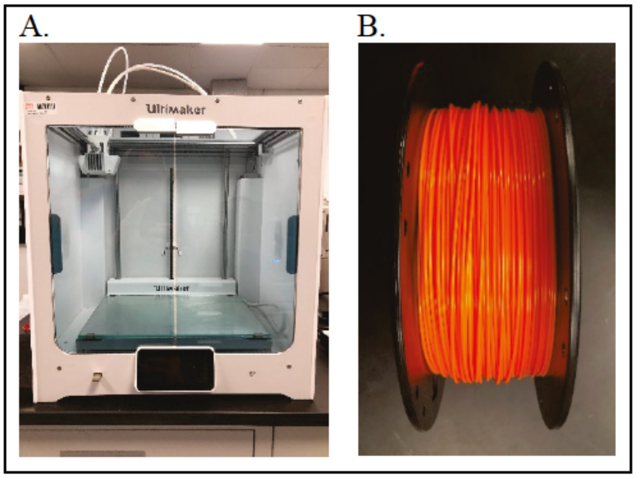

Figure 3. Examples of (A) a commonly available polymer extrusion 3D printer and (B) the polymer filament feedstock. Photographs by Alan Kennedy.

Recent developments in Additive Manufacturing (AM) (including low-cost thermoplastic polymer extrusion 3D printers with user-friendly software interfaces) enable completely customizable, on-site, and on-demand designs of seemingly limitless structural complexity that are unobtainable by conventional means (Campbell et al. 2011). Use of AM may also foster sustainability, as additive layering of multi-functional materials may result in less wasted material than traditional techniques (Friedmann et al. 2019). Fused Filament Fabrication (FFF) is a common, low-cost, highly accessible 3D printing technology (Figure 3A) that involves thermal extrusion of polymer filaments (Figure 3B). While the most common feedstock polymer filaments for FFF printers include polylactic acid (PLA), acrylonitrile butadiene styrene, and polyethylene terephthalate glycol (Figure 3B), considerable interest and research effort is being allocated to diversifying the material feedstock portfolio for novel applications and composites (Williams, Mistree, and Rosen 2011; Meisel Nicholas 2016). This technical note provides an evaluation of printable polymer composites consisting of thermoplastic polymers with high mass percentage loadings of particles that adsorb contaminants.

Provided prior and current research on 3D printing of highly filled polymer composites for various applications, including reinforcement and mechanical improvements (Fallon, McKnight, and Bortner 2019; Nadernezhad et al. 2019; Browne and Pumera 2019; Duty et al. 2018), FFF printers are amenable to printing polymer composites containing fillers that have environmental applications, including AC, zeolite, zero valent iron, hematite, biochar, chitosan, peat, biopolymers, organoclay, and graphene oxide (Cho et al. 2007; Guo, Zhou, and Ma 2006; Libralato et al. 2018). Recent published examples of such environmental applications of AM include printed polymer-zeolite monoliths for air purification (Thakkar et al. 2016), removal of ammonia from water (Kennedy et al. 2021), photocatalytic breakdown of contaminants in wastewater (Martín de Vidales et al. 2019; Friedmann et al. 2019) and containers for passive samplers to monitor contaminants (Kalsoom et al. 2018).

TECHNOLOGY DESCRIPTION: The U.S. Army Engineer Research and Development Center, Environmental Laboratory (ERDC-EL), is transitioning lessons learned from previous polymer-composite research to develop customizable, deployable, retrievable and reusable 3D-printed devices developed specifically for remediation and passive monitoring of contaminants in (1) dredging and CDF effluents; (2) in-place, contaminated DM; and (3) for mission relevant toxicity reduction/identification evaluation laboratory bioassays (Melby et al. 2018; USEPA 2007; Kreitinger, Farrar, and Lotufo 2017) conducted under the jurisdiction of the Clean Water Act or Marine Protection Research and Sanctuaries Act and associated DM testing guidance (USEPA/USACE 1991, 1998). Another relevant application of 3D printers for USACE dredging operations is the ability to produce parts on demand on vessels working offshore, where replacement parts are otherwise unavailable or time consuming to acquire.

The new USACE capability disseminated in this technical note is multi-functional polymer composite devices for remediation and monitoring of DM-specific CoCs that are enabled by AM. The composite may include multiple filler powders (e.g., AC, zeolite, TiO2) to have application across multiple classes of CoCs simultaneously (Friedmann et al. 2019). There are seven general categories of AM technologies (or processes) considered: vat polymerization, powder bed fusion, binder jetting, sheet lamination, material extrusion, and directed energy deposition (Williams, Mistree, and Rosen 2011). However, not all are amenable or feasible for economical 3D printing CoC adsorbing particles. Some technologies were not applicable to printing polymer composites for environmental applications and/or were expensive (i.e., directed energy deposition, powder bed fusion, material jetting, binder jetting) or not readily available or applicable (i.e., sheet lamination). Preliminary efforts with vat polymerization revealed the addition of nano-fillers did not allow consistent photopolymerization, and high filler loadings were not readily obtainable. Following this down selection, only material extrusion remained. Material extrusion, specifically desktop FFF 3D printers, was ultimately selected due to the availability of a diversity of polymer feedstocks, relatively low cost, and availability of a diversity of consumer products and established methods for compounding particle fillers into polymers to generate printable filaments.

While the selection of the appropriate sorbent particles may be intuitive to environmental scientists, selection of an appropriate polymer as a binder for 3D printing is also important. Considerations include the compatibility of the polymer-filler pairing, need for a more hydrophobic or hydrophilic polymer depending on the hydrophobicity of the CoC, and avoidance of thermal degradation of the polymer if the material composite must be processed multiple times (Taubner and Shishoo 2001;

Torres, Robin, and Boutevin 2000). Environmental applications of deployable polymer technologies and devices must consider concerns with polymer biocompatibility, environmental compatibility, hazard and potential for degradation and distribution of microplastics into environmental media (Sutliff et al. 2020; Browne 2015; Browne et al. 2011; Geyer, Jambeck, and Law 2017). For instance, passive sampling technologies may require more hydrophobic and persistent polymers such as polyethylene to adsorb the CoC (Schmidt et al. 2017) that to resist degradation to ensure recovery while other deployable polymer device applications may need to be more hydrophilic for soluble contaminants (Kennedy et al. 2021) or due to a desire for the material to biodegrade following deployment. A common bio-polymer used in 3D printing is PLA, which is being explored for environmentally compatible polymer applications (Lee, Ohkita, and Kitagawa 2004; Cicala et al. 2018; Taubner and Shishoo 2001). PLA also has a relatively large processing window due to its shear thinning behavior across a range of temperatures and 3D print speeds (Lee, Ohkita, and Kitagawa 2004; Cicala et al. 2018); this characteristic is helpful for overcoming challenges with processing filler applications (Shenoy 2013).

While many polymer and polymer composite filaments are currently commercially available, polymer filaments containing adsorbents for environmental CoC are not yet in the marketplace. Thus, researchers need to compound these materials into polymers and extrude into filaments for 3D printing. Compounding is the incorporation, wetting, and mixing of polymer melts with solid fillers or additives, involving breaking up of particle agglomerates followed by distributive and dispersive mixing into the polymer (Shenoy 2013; Huneault, Champagne, and Luciani 1996). The maximum filler concentration may be determined by morphology packing models (Blattmann and Pratsinis 2019; German and Park 2009; Fallon, McKnight, and Bortner 2019). Polymer-filler compounding methods may be broadly categorized as batch or continuous (Shenoy 2013). Batch methods include roller mills and internal mixers while continuous methods include single (Duty et al. 2018) and twin screw (Oksman et al. 2006) extrusion. This work accomplished compounding of environmental adsorbents into polymer by twin screw extrusion (Figure 4), since this method is considered efficient for mixing and fully homogenizing filler particles into polymer (Shenoy 2013; Villmow et al. 2008; Villmow, Kretzschmar, and Pötschke 2010).

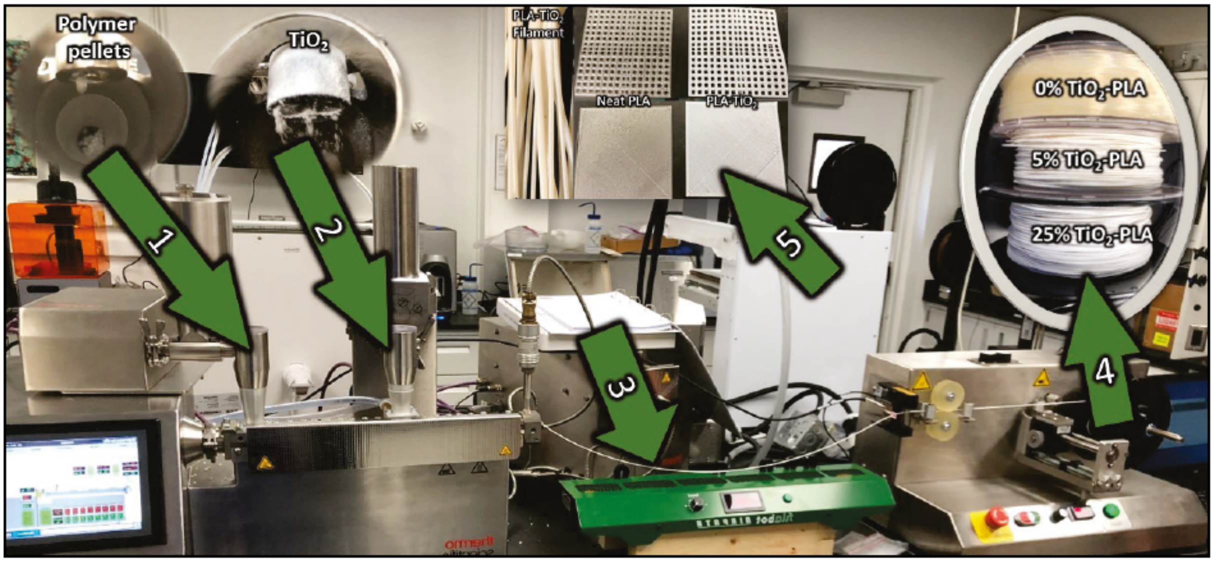

Figure 4. Photograph of the twin screw extruder used to compound particles for adsorbing environmental contaminants into 3D printable thermoplastic polymers. Step 1 (green arrow) shows pure polymer pellets fed into the system. In Step 2, pellets are melted and compounded with the filler (e.g., nano-TiO_2 particles). In Step 3, the extruded polymer-TiO_2 filament is cooled, and in Step 4 it is spooled for 3D printing (different TiO_2 loadings shown). Finally, in Step 5, the TiO_2 polymer composite is 3D printed into different structures for environmental remediation efficacy testing. Photographs and graphic by Alan Kennedy.

Following compounding, the resulting feedstock composite may be 3D printed if it has suitable rheological properties within the thermal processing window of FFF printers. If sole consideration is given to environmental contaminant absorption efficacy and kinetics, then maximizing filler particle loading is desirable; however, this must be considered in concert with the competing objective of creating a 3D printable composite filament within the processing window of a typical FFF machine. Greater filler concentration typically leads to processing issues such as higher polymer viscosity and/or potentially nozzle clogging (Chawla 2012; Gilmer et al. 2018; Fallon, McKnight, and Bortner 2019). The filler size and morphology also play a role as smaller particle sizes (which are an advantage for contaminant adsorption kinetics) and longer aspect ratios both increase viscosity (Shenoy 2013). Various parameters may also be adjusted to increase the processability/printability of the composite, such as extrusion temperature, environmental/localized heating (Luo et al. 2018; Sabyrov, Abilgaziyev, and Ali 2020), print speed (shear rate), layer height, and nozzle size (Duty et al. 2018; Mazzanti, Malagutti, and Mollica 2019; Fallon, McKnight, and Bortner 2019). The authors propose a preliminary flow chart to successfully produce 3D-printed environmental applications (Figure 5).

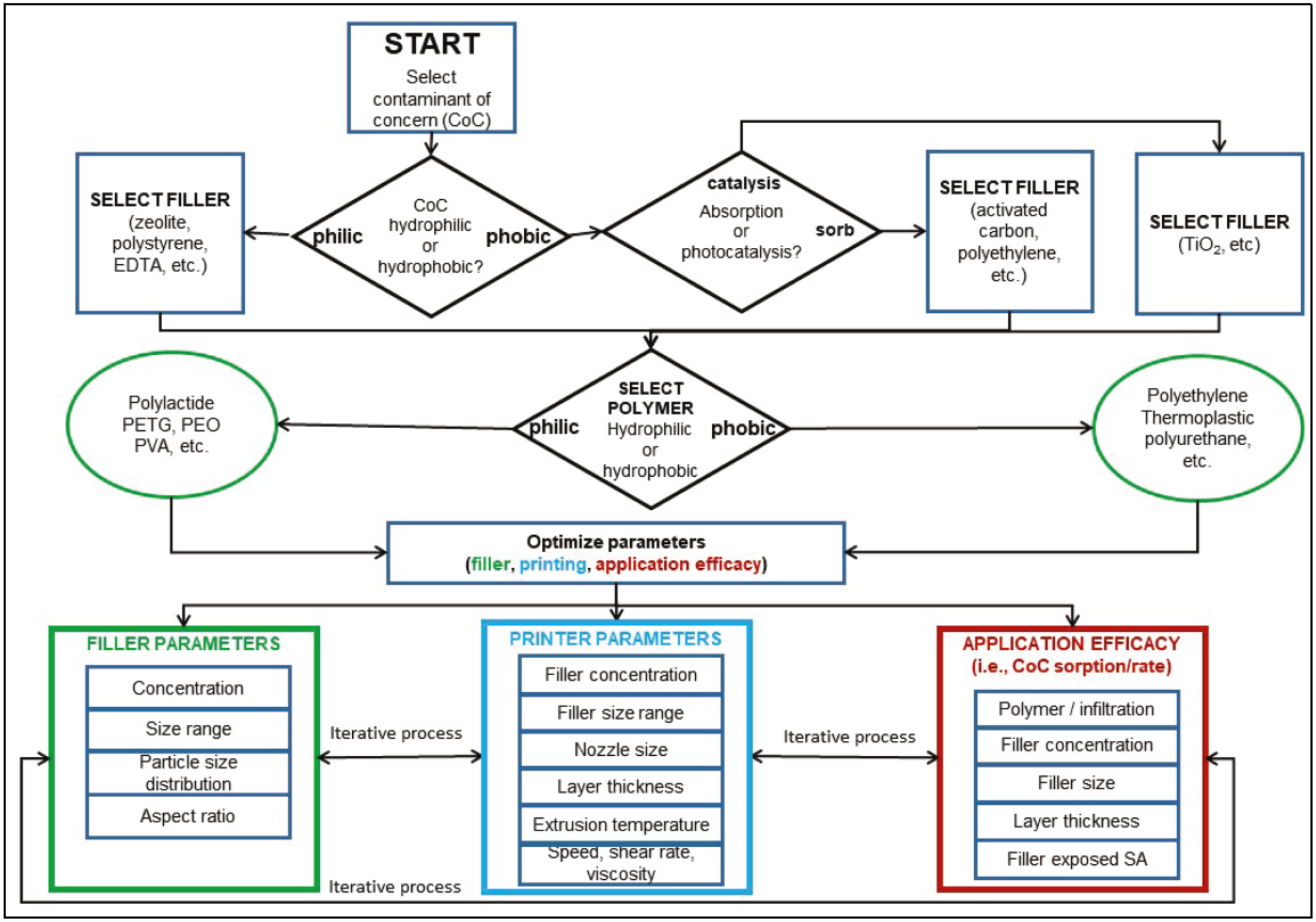

Figure. 5. Preliminary flow chart for additive manufacturing process selection for environmental applications of highly filled polymers. Flow chart by Alan Kennedy.

DISCUSSION AND RECOMMENDATIONS: For environmental applications of 3D printed polymer composites to have efficacy, the adsorbent particles within the polymer binder must have some exposed surface area to the environmental media. This may be accomplished by surface sanding or void space between the particles and the polymer (Zhang et al. 2020; Kennedy et al. 2021). Once the polymer composite is prepared as a feedstock, the benefit to 3D printing is the seemingly limitless, high surface area geometries that can be customized to the USACE site and application. Additional benefits include ability to deploy and retrieve the material from environmental media, which would not be possible if deploying loose powder. Finally, printing the composite material allows targeted delivery of the environmental sorbent (e.g., different water buoyancies, fixed structure at different sediment depths). Example geometries include spheres (e.g., shade balls used in California reservoir (Herman et al. 2017), screens, filters, and other complex geometries. Figure 6 provides some examples of environmental polymer composite geometries that were 3D printed at ERDC-EL for this project.

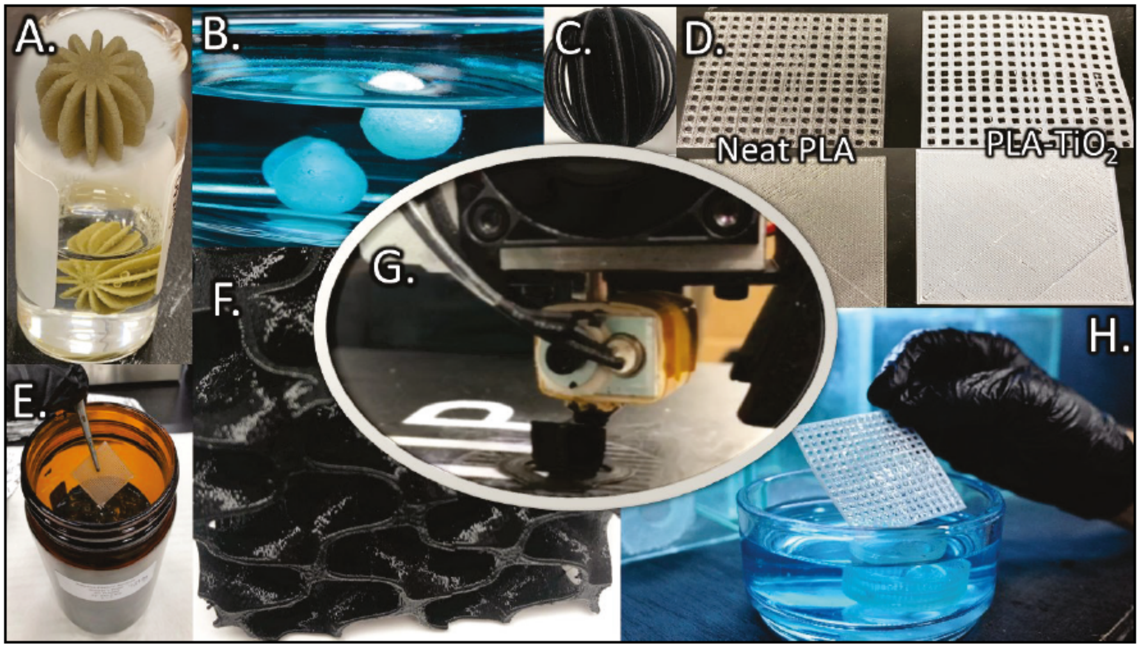

Figure 6. 3D printed applications produced for this project, including: (A) zeolite composite paddlewheels for remediation and passive sampling of emerging contaminants; (B) spheres with different buoyancies; (C) variable surface area structures; (D) test screen structures with and without TiO2 nanoparticles; (E) a polyethylene passive sampling device for sediment; (F) a high surface area structure that cannot be produced by conventional manufacturing; (G) a 3D printer extruding an activated carbon-polymer composite filament; and (H) a photocatalytic test screen structure. Photographs and graphic arts by Alan Kennedy.

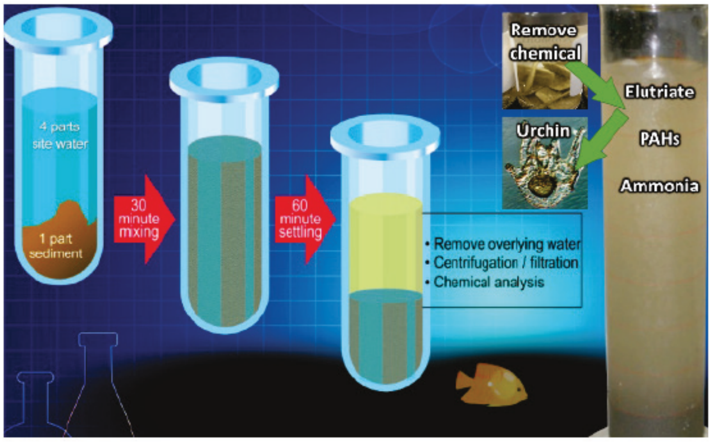

Figure 7. Conceptual model of integrating 3D printed zeolite composites for ammonia toxicity reduction evaluations in elutriate tests. Further description of the need to reduce ammonia in dredging elutriate bioassays in described in (Kennedy, Lotufo, and Steevens 2015). This figure is adapted from that source.

Clearly, numerous applications resulting from the materials 3D printed in Figure 6 can be envisioned to serve the USACE mission and beyond. A non-comprehensive list conceptualized by ERDC-EL includes the following: (1) customizable, deployable, retrievable, and reusable devices to remediate and/or passively monitor contaminants in various environmental media (e.g., water, sediments, soils) and effluents; (2) advancing scalable water treatment technologies for existing and emerging issues (e.g., legacy COCs, aquatic nuisance species, fluorinated compounds); (3) use in toxicity bioassays and novel treatments for toxicity reduction and identification evaluations (Figure 7); and (4) 3D printing novel, obsolete, or hard to obtain parts on demand on USACE or contract dredging vessels to avoid interruptions and delays in dredging operations while waiting for delivery of replacement parts.

This project (DOER-19-13) seeks to identify some of the countless customizable solutions to environmental issues associated with contaminated dredged sediments enabled 3D printing. This includes addressing proof-of-concept research, applied demonstrations, and filling knowledge caps associated with field deployment and scalability.

ADDITIONAL INFORMATION: This technical note was written under the Dredging Operations and Environmental Research (DOER program by Alan Kennedy (*Alan.J.Kennedy@usace.army.mil*, Tel: 601-634-3344, Fax: 601-634-2263), Mark Ballentine (*Mark.L.Ballentine@usace.army.mil*), Andrew McQueen (*Andrew.D.McQueen@usace.army.mil*), and Chris Griggs (*Chris.S.Griggs@usace.army.mil*) of the ERDC-EL.

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