High-efficiency Capture and Recovery of Anionic Perfluoroalkyl Substances from Water Using PVA/PDDA Nanofibrous Membranes with Near-zero Energy Consumption

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■ ABSTRACT

Poly- and perfluoroalkyl substances (PFASs) have caused severe public concerns due to their toxicity and extensive occurrence in the aquatic environment. This study reports a highly porous amine-functionalized membrane for the rapid capture of GenX and other anionic PFASs (e.g., perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA)) from contaminated water with near-zero energy consumption. The optimized membrane, prepared by electrospinning of polydiallydimethylammonium chloride using crosslinked polyvinyl alcohol as a binder, had a high water permeability of ~2700 $Lm^{-2}h^{-1}kPa^{-1}$. This high permeability enabled rapid gravity-driven filtration of contaminated water with a merely 5 cm water head, corresponding to an estimated energy consumption as low as 2.7×10^{-4} kWh/m^3. Meanwhile, the membrane showed highly-efficient capture of GenX (> 97%), PFOS (> 99 %), and PFOA (> 99 %). A large capture capacity of up to 1.2×10^6 $\mu g/m^2$ was demonstrated for GenX. The captured GenX was recovered and concentrated with a small-volume NaCl/methanol solution, which simultaneously regenerated the membrane for its reuse. Over a 12-cycle capture-recovery test, the membrane demonstrated a high GenX recovery ratio of 94% and a volumetric concentration factor of 40. Our study provides a promising strategy for effective capture and recovery of GenX to enable its sustainable control and remediation.

■ INTRODUCTION

Poly- and perfluoroalkyl substances (PFASs), such as perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOA), have been widely detected in the environment and human bodies.[1-6] Because of their extreme persistence and toxicity,[7-9] PFOS and PFOA have been strictly regulated over the past decades[10-12] and have been replaced with short-chain PFASs in a number of industrial and commercial products.[13, 14] In recent years, the ammonium salt of hexafluoropropylene oxide dimer acid (HFPO-DA), commonly known as GenX and used as a main substitute chemical for PFOA, has been under public scrutiny and has been the subject of several major lawsuits related to drinking water contamination.[15-19]

Recent toxicological investigations reveal that GenX has similar or even worse effects on the environment and human health compared to PFOA.[20, 21] Despite the quick actions by governments and manufacturers to curtail its discharge,[22-24] GenX is still continuously detected in the surface water with concentrations from several to hundreds ng/L.[25, 26] Urgent measures are called for to remediate GenX contamination for the protection of drinking water safety and public health.[27, 28] For example, its manufacturer, Chemours, has been mandated a minimal removal of 99% from its industrial waste effluent for subsequent destruction or reuse.[29] Treating GenX contaminated waters with low environmental concentrations yet huge volumes is a daunting challenge. Adsorption is able to remove GenX with some success while conventional sorbents such as activated carbon and resins requires extended treatment time (often due to mass transfer limitations).[30] On the other hand, advanced oxidation[31] or electrochemical treatment[32] are highly energy intensive and are not suitable for treating large volumes of dilute streams. Highly efficient technology for the rapid capture and recovery of GenX is yet to be developed.

This study reports a highly porous nanofibrous membrane with a nano-functionalized amine chemistry to separate GenX via an electricity- and chemical-free filtration. The large porosity of the membrane can enable rapid water transport even under gravity alone while the amine-based functionalization can realize effective removal of GenX due to their high affinity. A subsequent rinsing step was performed to recovery the enriched GenX for further destruction or recycle and to regenerate the exhausted membrane. Our study shows the feasibility of the rapid and effective remediation of water contaminated by GenX and other anionic PFASs using energy-efficient membrane separation.

■ MATERIALS AND METHODS

Chemicals. Polyvinyl alcohol (PVA, n = 1700, i.e., the number of repeating units, TCI), polydiallydimethylammonium chloride solution (PDDA, 20 wt.% in water, Sigma-Aldrich), and deionized (DI) water were used to prepare PVA/PDDA nanofibrous membranes by electrospinning. Acetic acid (99%, Dieckmann), glutaraldehyde (25 wt.% in water, Dieckmann), and hydrochloride acid (37%, VWR) were used to crosslink the membranes. GenX was received in the form of HFPO-DA (97%, Alfa Aesar). PFOA and PFOS were purchased from Sigma-Aldrich. The properties of GenX, PFOA, and PFOS were summarized in Table S1

(*Supporting Information S1*). Methanol (VWR) and sodium chloride (Uni-Chem) were used to prepare the rinsing solution for GenX recovery and membrane regeneration.

Membrane fabrication and characterization. PVA/PDDA nanofibrous membranes were fabricated using an electrospinning setup with three injection nozzles. For each membrane, a polymer dope solution was prepared by dissolving 10 wt.% PVA and 0-3.3 wt.% PDDA in DI water in a 90 °C water bath overnight. The dope solution was cooled to room temperature (~ 25 °C) and transferred to three 10 mL syringes (i.e., a total volume of 30 mL). Electrospinning was performed at a voltage of 18 kV, an injection rate of ~ 0.003 mL/min, a collection distance of 15 cm, and a receiving roller speed of 100 rpm. The electrospun membrane was subsequently immersed in the crosslinking solution containing 96 mL acetic acid, 4 mL glutaraldehyde, and 0.1 mL HCl for a duration of 0.5 h. The crosslinked membrane was then thoroughly rinsed with DI water and stored in a DI water bath before further use. The fabricated membranes were designated as PDDA-0, PDDA-1, PDDA-2, and PDDA-3 according to their PDDA content of 0, 1.0, 2.0, and 3.3%, respectively. Scanning electron microscope (SEM), elemental analyzer, and precision balance were used to characterize the morphology, structure, chemical composition, porosity, and grammage of the membranes (*Supporting Information S2*). At least three independent membrane samples were tested to determine membrane elemental composition, porosity, and grammage.

GenX capture and recovery. The capture of GenX was performed using a gravity-filtration cell with an effective filtration area of 3.3 cm^2 (*Supporting Information S3*). A membrane coupon was placed in the cell followed by DI water rinsing. Subsequently, 2 L solution of GenX at a concentration of 200 µg/L was continuously introduced to the cell to implement gravity-driven filtration at a constant water head of 5.0 cm (corresponding to a hydrostatic pressure of 0.49 kPa). A digital balance connected to a computer datalogging system was used to monitor the weight (and thus the volume) of permeate water collected as a function of time, and permeate samples were collected at specific volumetric intervals. Membrane regeneration and GenX recovery were performed for the PDDA-2 membrane over 12-cycle filtration tests to evaluate the durability of the membrane. At the end of each filtration cycle, the membrane was regenerated and the captured GenX was recovered by rinsing the membrane with a 50 mL mixture of 10 g/L NaCl and methanol with a volume ratio of 30/70%.[33] For comparison, the capture of PFOA and PFOS by the PDDA-2 membrane were also evaluated under identical operational conditions only the volume of feed solution was 4 L. The concentration of PFAS samples were determined by direct injection of samples into a liquid chromatography coupled with mass spectrometry (LC-MS) (*Supporting Information S4*).[34] The details of the calculation for membrane separation performance including water flux and capture ratio of PFAS as well as the recovery of GenX are described in *Supporting Information S5*.

■ RESULTS AND DISCUSSION

Membrane characterization. The electrospun membranes show typical nanofibrous structure consisting of a number of nanofibers, while the addition of PDDA has mild effect on membrane morphology (Figure 1A). Elemental analysis reveals the presence of nitrogen in the PDDA-doped membranes (e.g., 0.3% for PDDA-1 to 0.6% for PDDA-3, Figure 1B), confirming the successful incorporation of PDDA. Membrane grammage (i.e., mass per unit membrane area) decreases with increasing the PDDA content (Figure 1C), which can be attributed to the reduced loading of nanofibers as a result of enhanced electrostatic repulsion.[35] According to the membrane grammage and the pre-determined weight content of PDDA in the membranes (i.e., 0-3.3 wt.%), the PDDA-2 membrane possessed the highest PDDA loading of 4.0 g/m^2 among all the membranes (*Supporting information S6*). All the membranes present high water permeability ranging from 1615 to 3308 $Lm^{-2}h^{-1}kPa^{-1}$ (Figure 1D), which is 3-4 orders of magnitude higher than that of typical gravity-driven membranes.[36] The high water permeability can attributed to the hydrophilic nature of the polymers (e.g., PVA and PDDA) and the high porosity of the membranes (e.g., ~ 90%, Figure 1C). Such high water permeability is beneficial for the rapid water production under gravity-driven condition (e.g., using a 5 cm water head in this study) and therefore enables near-zero energy filtration (e.g., corresponding to a specific energy consumption of 2.7×10^{-4} kWh/m^3, *Supporting Information S7*).

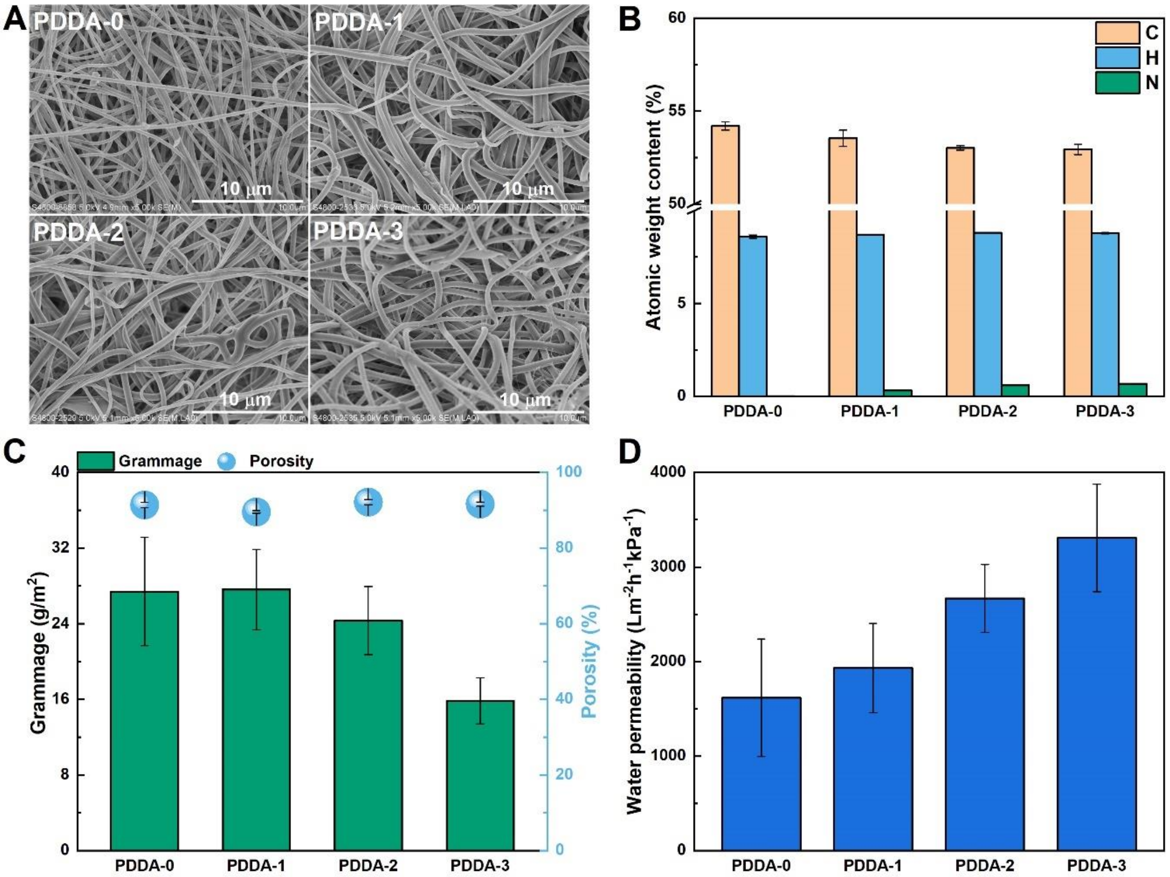

Figure 1. (A) SEM micrographs of membrane morphology, (B) elemental composition of C, H, and N, (C) grammage (i.e., mass per unit membrane area) and porosity, and (D) water permeability for the various PDDA membranes. The error bars represent the standard deviation acquired from at least three independent samples.

PFASs capture. The blank membrane PDDA-0, which contained PVA only, had negligible capture of GenX (Figure 2A). In contrast, the PDDA incorporated membranes (PDDA-1, 2, and 3) had progressive higher capture ratios at higher PDDA loadings, which reveals the critical role of PDDA on the capture of GenX. This effect can be attributed to the electrostatic attractions between the positively charged quaternary amine group in PDDA and negatively charged carboxylic group in GenX.[37] The PDDA-2 membrane presented the highest capture ratio of > 97% with the GenX loading up to 1.2×10^6 μg/m^2, which is partly due to its richest content of PDDA among all the membranes (*Supporting Information S6*). Although PDDA-3 membrane also had high PDDA content, its capture ratio of GenX was lower than that of the PDDA-2 membrane, which may be attributed to the reduced residence time of GenX within the PDDA-3 membrane as a result of its high water permeability (Figure 2D). The capture ratio of GenX by filtration was significantly higher than that by adsorption with same treatment time (Figure S4, *Supporting Information S8*), which can be attributed to the greatly improved mass transfer in the "flow-through" filtration mode. Similar enhancement effects of "flow-through" over "flow-by" have been observed in the context of catalysis.[38] In addition to GenX, the PDDA-2 membrane also presented excellent capture efficiency for PFOS and PFOA (Figure 2B). The capture ratio of PFOS and PFOA remained at ~ 95% with the PFOS or PFOA loading up to 2.4×10^6 μg/m^2. Although PFOS and PFOA has been replaced by short-chain PFAS (e.g., GenX) for decade, their occurrence in the water system are continuously reported.[39, 40] Our results demonstrate the great potential of the PDDA membranes for highly efficient capture of PFASs (e.g., PFOS, PFOA, and GenX) from contaminated water. In addition to the capture of single PFAS, we also evaluated the ability of the PDDA-2 membrane for capturing GenX, PFOA, and PFOS from their mixed solution containing 10 μg/L for each compound (*Supporting Information S9*). For volumetric loading of up to 6000 L/m^2, the membrane showed high capture ratios of ⩾ 99% for all three PFASs (Figure S5). With the increase of volumetric loading, the capture ratio of GenX was reduced and those of PFOS and PFOA remained stable. This result is consistent with Figure 2B and other published literature,[33] which can be attributed to the lower affinity of GenX to amine-based functional groups compared to PFOS and PFOA (*Supporting Information S10*). Nevertheless, the capture ratio of GenX remained at > 92% with a volumetric loading up to ~9000 L/m^2 (Figure S5).

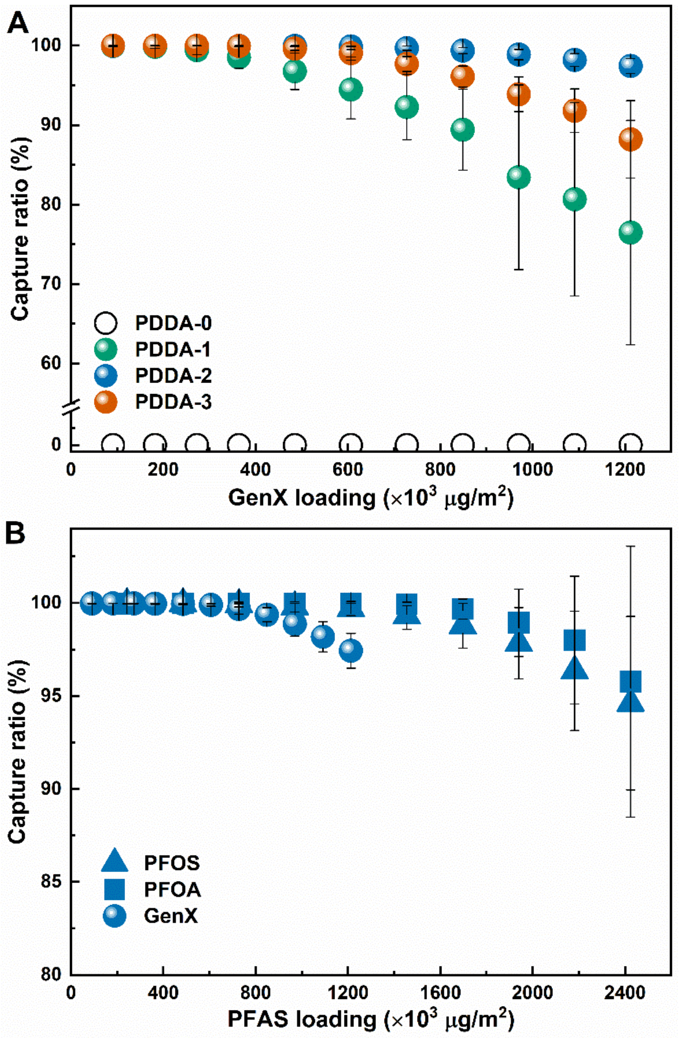

Figure 2. (A) The capture ratio of GenX as a function of GenX loading for the various PDDA membranes, and (B) the capture ratio of PFOS, PFOA, and GenX for the PDDA-2 membrane. Experimental conditions: 2 L feed solution containing 200 µg/L GenX, effective membrane filtration area of 3.3 cm^2, water head of 5.0 cm. For the capture of PFOA and PFOS, the volume of feed solution was 4 L while other experimental conditions were identical. The error bars represent the standard deviation acquired from at least three independent samples.

GenX recovery and membrane regeneration. Following the effective capture of GenX from a contaminated water, its subsequent recovery is also critical for the potential destruction or recycle of the compound as well as the sustainable reuse of the membrane. A simple rinsing using NaCl/methanol solution was performed to simultaneously extract the captured GenX and to regenerate the membrane. As shown in Figure 3, the PDDA-2 membrane maintained high capture ratio of GenX (e.g., > 95%) over the 12 capture-recovery cycles, suggesting the successful regeneration of the membrane. Since the used membrane was regenerated by only 50 mL NaCl/methanol solution after treating 2 L solution of 200 µg/L GenX, a high volumetric concentration factor of 40 was achieved for the GenX recovery. Meanwhile, 94.0% of the GenX in the feed water was recovered from the membrane and concentrated in the small-volume rinsing solution. The highly effective capture, recovery, and concentration of GenX enables its further destruction or reuse in a centralized way, which can significantly reduce the overall treatment cost while improving the efficiency.

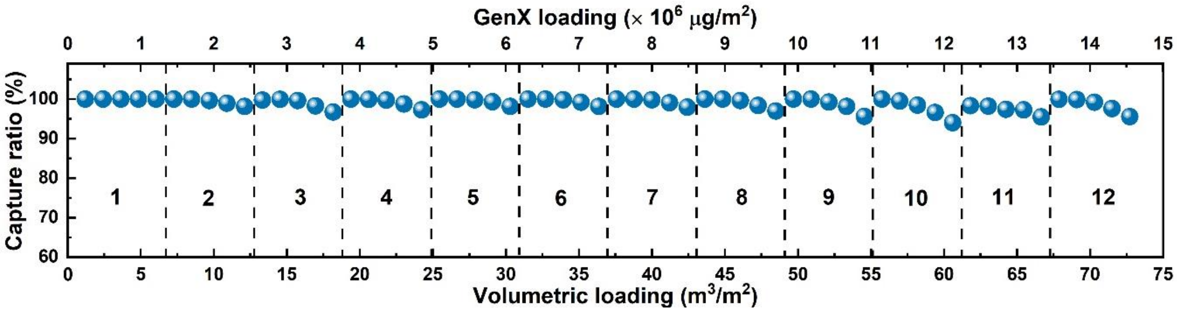

Figure 3. The capture ratio of GenX by PDDA-2 membrane over 12 capture-recovery cycles. Experimental conditions: 2 L feed solution containing 200 µg/L GenX, effective membrane filtration area of 3.3 cm^2, water head of 5.0 cm. At the end of each filtration cycle, 50 mL mixture of 10 g/L NaCl and methanol with a volume ratio of 30/70% was added into the cell to recover the captured GenX and regenerate the membrane. Subsequently, 50 mL DI water was added into the cell to rinse the membrane for next filtration cycle. Total 12-cycle filtration tests were performed.

The global occurrence of GenX poses daunting threats to the environment and public health.[2, 20, 25] A recent study by Joerss et al. revealed the transport of GenX far to the remote Arctic

Ocean,[41] which presents alarming evidence for the wide spread of GenX contamination. Effective control and remediation strategies are urgently called to prevent the further discharge of GenX into the environment as well as to remove it from the contaminated waters. Our study presents a sustainable membrane filtration technology to address this critical issue through (1) the effective capture of GenX from a large-volume contaminated water using a gravity-driven PDDA nanofibrous membrane, and (2) its concentration and recovery for further destruction or recycle (Figure 4). In addition, the rapid filtration process requires no electricity input, which may be used in remote areas or in underdeveloped regions and offers significant advantages over other technologies (e.g., electrochemical degradation).[32] Meanwhile, the sustainable reuse of the membrane also effectively reduces the operational cost.

To control the discharge of GenX from the industrial sources, one can further implement a multi-pass filtration to ensure a high overall capture efficiency of > 99% that has been mandated in some countries.[29] The captured GenX can be recovered for further reuse or treatment within the manufacturing site and thus curtails its transport to the environment. For the removal of GenX from contaminated surface waters, the gravity-driven filtration technology can be integrated into the existing water treatment chains as a polishing step.[36] Alternatively, it can potentially be used as a point-of-use treatment technology for the decontamination of GenX in household (e.g., tap waters or groundwaters).[42] To enable such practical applications, future studies need to systematically investigate membrane separation performance under a wider range of feed water qualities as well as the effect of biofilm growth and membrane fouling on the removal efficiency.[36] The novel filter can also be potentially used for the removal of other anionic PFASs.

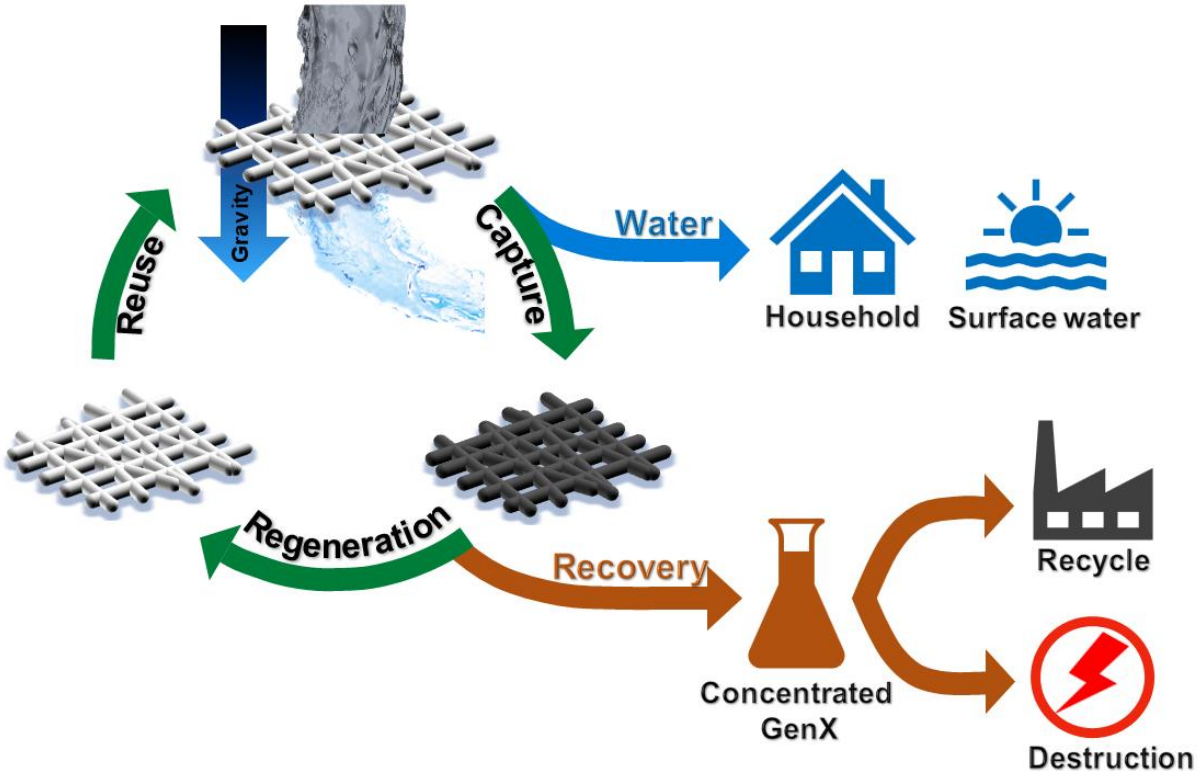

Figure 4. Illustration of the proposed gravity-driven membrane filtration technology for the sustainable capture and treatment of GenX.

■ ASSOCIATED CONTENTS

Supporting Information. S1. Properties of GenX, PFOA, and PFOS; S2. Membrane characterization; S3. Gravity-driven filtration setup; S4. LC-MS analysis for PFASs; S5. Calculation of water permeability, capture ratio, and recovery ratio; S6. PDDA content of the membranes; S7. Specific energy consumption; S8. Comparison between filtration and adsorption; S9. Membrane separation performance for mixed PFASs; S10. Binding affinity of PFASs with PDDA membranes; S11. Effect of organic matter on the removal of GenX; S12.

Comparison between the PDDA-2 membrane and SPE cartridge; S13. Membrane pore size. This material is available free of charge at http://pubs.acs.org.

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Notes

The authors declare no completing financial interest.

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