

Membranes

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Robust Aqua Material: A Pressure-Resistant Self-Assembled Membrane for Water Purification

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Abstract: “Aqua materials” that contain water as their major component and are as robust as conventional plastics are highly desirable. Yet, the ability of such systems to withstand harsh conditions, for example, high pressures typical of industrial applications has not been demonstrated. We show that a hydrogel-like membrane self-assembled from an aromatic amphiphile and colloidal Nafion is capable of purifying water from organic molecules, including pharmaceuticals, and heavy metals in a very wide range of concentrations. Remarkably, the membrane can sustain high pressures, retaining its function. The robustness and functionality of the water-based self-assembled array advances the idea that aqua materials can be very strong and suitable for demanding industrial applications.

Self-assembled supramolecular materials that are robust yet adaptive can represent an alternative to conventional materials in applications that benefit from multifunctionality, recyclability, and self-healing.^[1–3] In this regard, a concept of environmentally friendly “aqua materials” (or “aqua plastics”) have been recently introduced,^[4] in which water is employed as a key material component that brings very strong noncovalent interactions.^[5] These interactions result in both robustness and adaptive properties, as demonstrated in mechanically strong supramolecular hydrogels capable of self-healing.^[4] We aimed to show that an aqua material can possess the robustness necessary to sustain harsh conditions that so far required exclusive use of covalent systems, and demonstrate applicability of water-based supramolecular materials for a challenging industrial application. In this respect, membranes used for water purification and desalination are based on crosslinked covalent polymers that possess mechanical and chemical robustness, high porosity, high retention of various contaminants, and ability to operate

under high pressures.^[6–8] Herein we demonstrate that these properties can be achieved using a water-based material obtained by self-assembly of colloidal Nafion on a noncovalent PP2b layer (Figure 1). These components synergistically enhance each other's structure and properties to result in a tough membrane that efficiently purifies water from heavy metals and organic molecules, while resisting high pressures. Such robustness is without precedent for supramolecular water-based materials. The membrane can be easily fabricated and disassembled. The extraordinary robustness and valuable functionality of the water-based self-assembled array demonstrates, for the first time, suitability of aqua materials for a demanding industrial application.

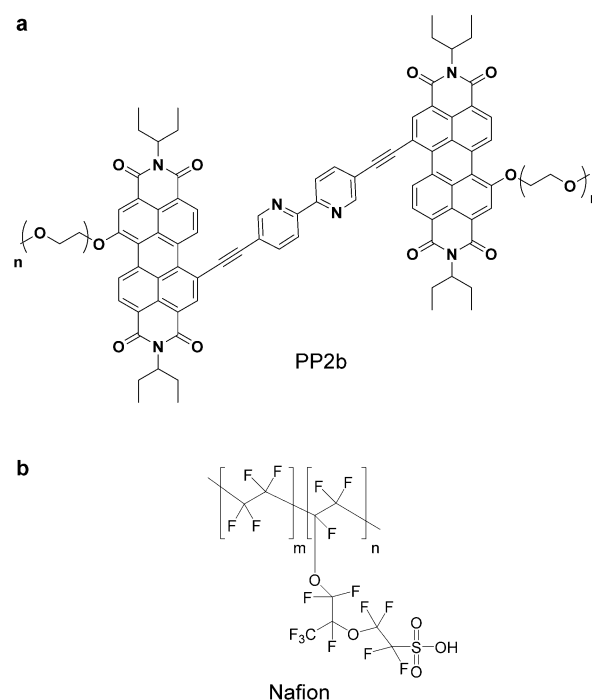


Figure 1. Membrane components. a) PP2b amphiphile building block. b) Nafion polymer structure.

Our choice of PP2b and colloidal Nafion to create a robust water-based material was based on the idea that hydrophobic interactions in both systems may be synergistically strengthened by their contact. Thus, colloidal Nafion particles (40–50 nm in size) should be retained by supramolecular PP2b membrane^[9] and may undergo self-assembly in water interacting via highly hydrophobic fluorocarbon chains, while

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Nafion polyelectrolyte nature is expected to exert significant osmotic pressures on the semipermeable gel-like PP2b layer, increasing its stability through compression.^[10] As a support for membrane fabrication we chose the standard 0.45 μm polyethersulfone (PES) filter since it allows high water fluxes, is low in inorganic extractable ions, and is economical for larger scale applications. A typical membrane fabrication procedure includes deposition of a water/THF (98:2 v/v) solution (0.9 mL) of PP2b (10^{-4} M) over the PES support (hydrophilic, 0.45 μm , 13 mm, effective filtration area 0.95 cm^2), rinsing with water, and gradual increase of trans-membrane pressure to its final value of 2 bar. In the next step, 0.5 mL of Nafion suspension in water (10 % w/w) is deposited on top of a PP2b layer to complete the hybrid structure (Figure 2a). Imaging the membranes cross-section using

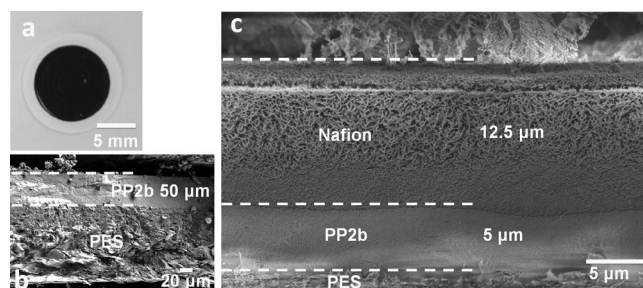


Figure 2. Membrane structure. a) hybrid membrane of PP2b + Nafion (50 mg) deposited on top of the polyethersulfone (PES) support. b) Cryo-SEM image of freshly prepared 5% PP2b PEG13/95% PP2b PEG17 (in PEGX, X designates the number of repeating ethylene glycol units) supramolecular membrane cross section (ca. 1 \times 1 mm) deposited on the PES support without Nafion. c) Cryo-SEM image of freshly prepared 5% PP2b PEG13/95% PP2b PEG17 supramolecular membrane cross section (ca. 1 \times 1 mm) deposited on the PES support with Nafion-hybrid structure. The ratio of the PP2b components (95% PEG17 and 5% PP2b PEG13) is the best to achieve good deposition and reproducible recycling.

cryogenic scanning electron microscopy (cryo-SEM) reveals substantial densification of the PP2b layer in the hybrid structure (ca. 5 μm thick, Figure 2c) compared with a membrane containing only PP2b (ca. 50 μm thick, Figure 2b). The observed compression of the PP2b layer is due to a combination of mechanical compression and the osmotic pressure, resulting from a polyelectrolyte (Nafion) deposition on a semi-permeable membrane (PP2b).^[11] This process is also expressed in the decrease of flux (at 2 bar) from approximately 0.1–0.2 mL min^{-1} (permeance of ca. 30–60 $\text{L h}^{-1}\text{ m}^{-2}\text{ bar}^{-1}$) to 0.01 mL min^{-1} after Nafion deposition (permeance of ca. 3 $\text{L h}^{-1}\text{ m}^{-2}\text{ bar}^{-1}$), consistent with thicker and denser structure. Overall, hydrogel-like material featuring a 3D Nafion network and dense PP2b layer is formed. To further investigate the membrane structure we used energy dispersive X-ray spectroscopy (EDS). By mapping the entire cross section of the dried membrane we observed that the top layer of the hybrid membrane contains Nafion, confirming distinct separation between Nafion and the supramolecular PP2b layer (Supporting information Figure S8). PP2b layers that have been previously employed by us as ultrafiltration

membranes^[9] have lower density, larger pores, and much lower mechanical strength than the ones interacting with Nafion. We note that the densification of PP2b layers has critical importance for the membrane performance (see below).

The removal of toxic heavy metals from wastewater and drinking water is highly desirable,^[12] with the maximum contaminant levels (MCL) ranging from few ppb into hundreds ppb.^[13] To demonstrate removal of heavy metals with our membrane, we focused on salts of the following contaminant metals: lead (Pb), nickel (Ni), cobalt (Co), and cadmium (Cd). These were filtered using the hybrid membrane in a high (hundreds of ppm) and low (up to 1000 ppb) concentration regimes, while the range up to 1000 ppb is more typical for wastewater.^[13–15] The feed solutions and filtrates were analyzed using inductively coupled plasma mass-spectrometry (ICP-MS), see Table 1. Superior removal of heavy

Table 1: Removal efficiencies of Ni^{2+} , Co^{2+} , Pb^{2+} , and Cd^{2+} by the hybrid membrane.

Filtered salt	High concentration		Low concentration	
	Initial metal concentration [ppb]	Filtrate metal concentration [ppb] (metal uptake %)	Initial metal concentration [ppb]	Filtrate metal concentration [ppb] (metal uptake %)
NiSO_4	518 134	4107 (99.21)	1054	11 (98.96)
CoCl_2	543 960	308 (99.94)	1031	0.18 (99.98)
$\text{Pb}(\text{NO}_3)_2$	636 959	4477 (99.29)	588	1–3 (99.5)
CdSO_4	883 138	4857 (99.45)	1075	0.20 (99.98)

metals from water was observed, with metal retention reaching up to 99.98 % in both concentration regimes. Remarkably, even at relatively high metal concentrations the retention efficiency was excellent (above 99 %). Moreover, a mixture containing all the heavy metals (Pb, Cd, Co, and Ni) was also retained (Supporting information, Table S1). Control experiments carried out with Nafion directly deposited on PES support showed low metal retentions, indicating the crucial role of PP2b layer (Table S2). According to EDS, in the absence of PP2b the majority of Nafion passes through the membrane (Figure S9).

To test leaching of heavy metals contaminants retained in the membrane we filtered 5 mL of Pb (588 ppb) and collected five fractions of 1 mL each that were analyzed separately. Results show that all fractions contain 1–3 ppb of Pb, demonstrating reliable metal retention.

The membrane shows excellent retention of Cd (99.5 %) in the presence of NaCl (Table S3), attesting to the selectivity towards heavy metals. Thus, sodium, potassium, and magnesium salts are retained by the membrane to a much lesser extent than the heavy metal salts. Yet, this retention is still substantial as the salts concentrations of a 500 ppm (NaCl, KCl, and MgCl_2 mixture) are reduced by 43 %, 66 %, and 72 % respectively (Table S4). To address the location of metal retention in the membrane we conducted an EDS experiment using a membrane sample after filtration of CdSO_4 . We

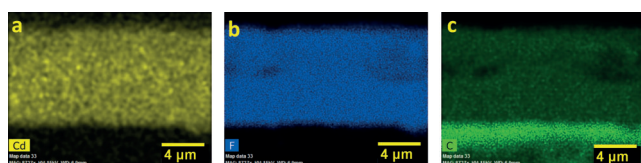


Figure 3. Hybrid membrane cross section EDS (50 mg Nafion, 15 kV). Mapped areas containing a) cadmium, b) fluorine, c) carbon.

observed that Cd is located within the Nafion layer (Figure 3 and Figure S10), and not in the PP2b or PES layers. Furthermore, the Cd distribution is uniform and higher concentration of Cd on top of Nafion is not observed. The hybrid membrane performance related to retention of heavy metals is comparable or superior to commonly used membrane types, such as ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO).^[16]

In the case of Ni, for instance, metal retention is 60–100% using various UF,^[17,18] NF,^[19] and RO^[20] membranes, with much lower initial metal concentration than our high-concentration-regime experiments. In a similar fashion, for the established systems the reported Co retention is 95–100%,^[17,18] while that of Cd is 93–99%.^[21] The reported retentions can only be achieved at specific (optimum) pH values, whereas our membrane does not require any pH adjustment. Furthermore, our membrane exhibits a significant performance advantage when compared with a commercial

membrane comprised solely from Nafion (Nafion 117). In a study conducted on the adsorption of heavy metals by such a membrane, the following metal retentions were found: 96.2% (Ni^{2+}), 90% (Co^{2+}), and 88% (Pb^{2+}) with an initial metal concentration of 1000 ppb.^[22] In the case of this membrane, the metal rejections were found to drop with decreasing concentration and can be as low as 56.7% (Pb^{2+}) when the initial metal concentration is 200 ppb. In our case, the metal retention is high also at very low initial metal concentrations (Supporting information Table S5).

Small organic molecules, such as dyes and drugs, are present in contaminated water,^[23–26] and need to be removed in the process of water treatment. To assess the membrane's performance towards retention of organic dyes we performed filtration experiments with the pH indicator Bromocresol green (BCG) in its two forms: anionic form (blue) and neutral form (yellow). Quantitative removal of both forms was observed (Figure 4a). Cationic and neutral Rhodamine 110 were also quantitatively retained according to UV/Vis spectroscopy (Figure 4b). In the case of Rhodamine 110 we also tested membrane leaching, washing it with 8 mL of water and checking the filtrate by UV/Vis spectroscopy (Figure S11). None of the collected fractions contained Rhodamine. We have also tested the reversible nature of our hybrid membrane. Following the dye filtration, the membranes were disassembled using an organic solvent (60% EtOH/40% water). Adding chloroform to the resultant solution leads to

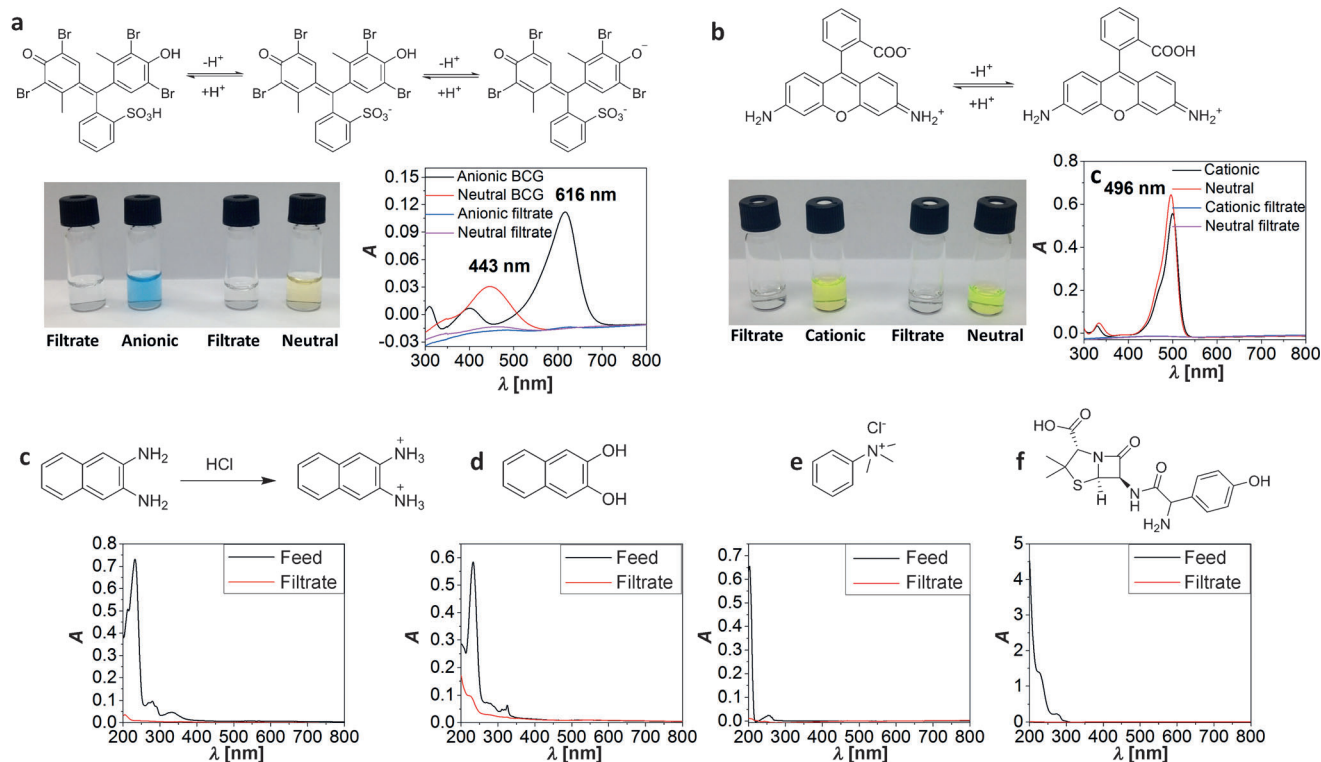


Figure 4. Molecules filtration capabilities. a) Bromocresol green structure, solutions before and after filtration and UV/Vis spectrum. b) Rhodamine 110 structure, solutions before and after filtration and UV/Vis spectrum. c) 2,3-diaminonaphthalene structure and its UV/Vis spectrum before and after filtration (10^{-4} M). d) 2,3-dihydroxynaphthalene structure and its UV/Vis spectrum before and after filtration (10^{-4} M). e) Trimethylphenyl ammonium chloride structure and its UV/Vis spectrum before and after filtration (5×10^{-4} M). f) Amoxicillin structure and its UV/Vis spectrum before and after filtration (10^{-3} M, dissolved with 5 drops of NaOH 1 M).

two fractions: the organic layer containing PP2b, and the aqueous one containing Nafion, as well as the dye adsorbed by it (the solution is partially colored by the dye, and Nafion can be detected by F NMR spectroscopy, Supporting information Figure S12).

The organic phase can be purified, reassembled, and re-used in membrane fabrication. Nafion layer could not be recycled, and we are currently working on a procedure that will allow full recycling. Both BCG and Rhodamine 110 filtration using the recycled PP2b layer on which Nafion was deposited was quantitative (Figure S13). Control dye-filtration experiments were performed using PES filters, on which Nafion was directly deposited (it is only partially retained on the support). We observed that the dyes are not retained (Figure S14).

To further evaluate membrane performance regarding retention of pharmaceuticals (an important issue in water purification of urban sewage)^[23–25] we performed filtration of Amoxicillin, a commonly used antibiotic. UV/Vis spectroscopy indicated quantitative retention (Figure 4f), which is similar or superior to currently employed polymeric membranes.^[27,28]

Targeting filtration of small water-soluble organic molecules, we compared two naphthalene derivatives with similar size but different charge: 2,3-diaminonaphthalene which is positively charged (10^{-4} M, dissolved with few drops of 1 M HCl) and neutral 2,3-dihydroxynaphthalene. Both molecules are retained, with 2,3-diaminonaphthalene showing better retention according to UV/Vis spectroscopy (Figure 4c,d). An even smaller molecule, such as trimethylphenyl ammonium chloride (Figure 4e), was also retained, while phenol and benzoic acid were not retained. These results suggest that both charge and molecular size contribute to the retention. Thus, the Nafion layer acts as a membrane having ion-exchange properties, while the densification of PP2b appears to play a role in the size-selective rejection.

To further probe the role of the densified supramolecular layer in the filtration, we performed filtration of a mixture of gold nanoparticles (1–13 nm in size, average 5.1 ± 2.1 nm) covered with neutral NEG (nona-ethylene glycol-based) capping layer. EDS measurements show that the nanoparticles pass the Nafion layer and then are retained by the PP2b layer, indicating that a cut-off size of the PP2b layer is less than 1 nm (nondensified PP2b exhibits 5-nm cut-off),^[9] confirming the size-selective rejection role of the densified supramolecular layer (Figures S15–S17). Thus, less-polar solutes or the ones having lower affinity to the Nafion sulfonates can pass the Nafion layer but be retained by the PP2b layer according to their size, reflecting the synergetic function of the membrane materials.

We have also found that decreasing the deposited Nafion quantity from 50 mg to 20 mg maintains the good retention efficiency for both heavy metals (Pb and Cd retention > 99.5 %, Table S6) and organic molecules (Amoxicillin, Figure S18). In this case we obtain a thinner layer of Nafion and a somewhat thicker layer of PP2b (Figure S19), without significant change in the rejection properties.

A challenging issue regarding the currently employed polymeric membranes is irreversible fouling^[29] that leads to

low flow rates. Cleaning the conventional covalent membranes is usually a difficult and expensive process, which is infeasible in some cases.^[8,30,31] In the case of our self-assembled membrane it can be deposited from solution on the standard filtration module (e.g. having large pore PES as a support membrane), disassembled upon fouling, cleaned, and reassembled again on the same module, emphasizing the advantage of the self-assembled nature of the Nafion/PP2b membrane.

We have also performed high-pressure experiments since commercial processes involving membranes use pressure as the driving force for permeation. The permeance of our system ($3 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$) measured at low pressure is typical for nanofiltration applications, which are typically driven by 3 to 20 bar operating pressures.^[32,33] Our system's performance in partial desalination is actually rivaling reverse osmosis membranes, which use higher trans-membrane pressures (between 10 and 100 bar).^[33,34] We note that operating pressure can influence membrane performance.^[35,36] Hence, the performance of hybrid membranes containing 20 mg or 50 mg Nafion deposited on top of PP2b layer was explored under elevated pressures. With 20 mg Nafion, we observe a permeance of approximately $29 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$ (2.8 bar). Under these conditions, Pb retention is over 98.3 % (from 600 ppb to less than 10 ppb). Permeance further increases under higher pressure of 8.7 bar ($46 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$). Increasing the pressure above 9 bar leads to a pressure drop as evidenced by flux/permeation-pressure plots (Figure 5a,b), probably because of membrane damage. However, when 50 mg of Nafion is deposited, permeance starts at around $2.7 \text{ L h}^{-1} \text{ m}^{-2} \text{ bar}^{-1}$ at the lowest pressures, and then decreases stepwise upon pressure increase, indicating a stepwise membrane compression (Figure 5c,d). This experiment was conducted over three consecutive days, providing us with the

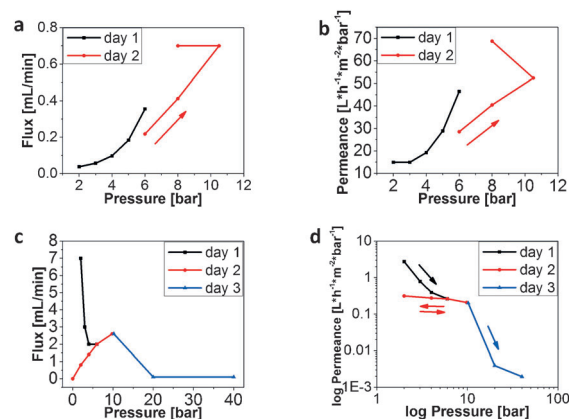


Figure 5. Hybrid membrane flux and permeance under high pressure over several days. The membranes were operated for the entire day, increasing the pressure every hour, then pressure was reduced to zero overnight and gradually ramped up again the next day. a) 20 mg Nafion membrane, flux. b) 20 mg Nafion membrane, permeance. c) 50 mg Nafion membrane, flux. d) 50 mg Nafion membrane, permeance on three consecutive days: After the first operation up to 10 bar, the permeance remained at this plateau over three days of variable pressures, before the pressure was increased up to 40 bar, inducing membrane collapse without recovery.

insight that the membrane is highly robust and stable at 10 bar for at least 18 h. Operating at 10 bar, a filtration of 2,3-dihydroxynaphthalene resulted in similar decrease in UV/Vis absorbance (Figure S20) as for the low-pressure operation (Figure 4d). The membrane is thus fully functional at this pressure. Stable operation in the range between 2 bar and 10 bar is observed and remains the same also after pressureless recovery overnight. This performance is reproduced in three repeated experiments with three independently prepared membranes. The permeance drops significantly at pressures of 20 bar and above, but the composite membrane was found to withstand high pressures up to 40 bar, undergoing significant compression (eventually the permeance values are limited as no single drop of filtrate emerged over several hours). Remarkably, in the experiments where 40 bar pressure was reached, the steel frit used to support the membrane was bent, while no damage to the membrane was observed. The PP2b amphiphile is stable at high pressures: no chemical change in PP2b is observed after applying pressures of 40 bar, as evidenced by UV/Vis spectroscopy and mass spectrometry (Figure S21). Cryo-SEM imaging of the membranes cross-section after pressurizing at 40 bar reveals a layered structure with Nafion on top, but also penetrating to some extent into the PP2b and PES support (Figure S22), which probably causes the permeance drop. We believe that strong hydrophobic interactions and densification of the PP2b layer are responsible for the remarkable stability of the membrane.

Further studies addressing mechanisms underlying the observed robustness are in progress.

In conclusion, the reported material exhibits performance and robustness that neither of the individual building blocks is capable of, demonstrating outstanding filtration capabilities with regard to toxic heavy metals and small molecules under low and high pressures. The stability, adaptive properties, and performance of the self-assembled membrane confirm that aqua materials can possess robustness and functionality suitable for demanding industrial applications.

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Conflict of interest

The authors declare no conflict of interest.

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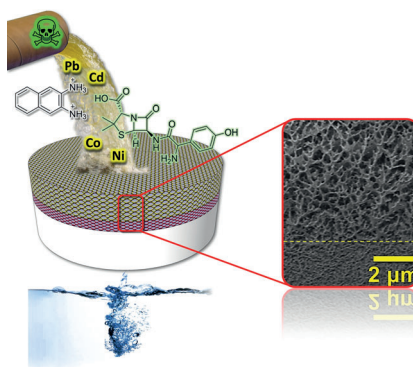
Communications



Membranes

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A tough aqua material for a tough application: The hybrid membrane's components self-assemble to synergistically enhance each other's structure and properties to result in a tough membrane that efficiently purifies water of heavy metals and organic molecules.