



A versatile hydrogel platform for oil/water separation, dye adsorption, and wastewater purification

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Abstract The development of a versatile platform that can separate oil/water mixture, remove dye from water, and purify wastewater is extremely desirable, yet still hard to realize. Herein, to address this challenge, a composite hydrogel was produced by freezing–thawing treatment using chitosan, polyvinyl alcohol, and carbon black as the raw materials. The obtained hydrogel displayed both slippery oil-repellency and water-affinity in air, underwater, when submerged in oil, and exploiting this special wettability, the hydrogel coated mesh can be used to separate oil/water mixtures efficiently. After 25 oil–water separation cycles, the hydrogel-coated filter still had a

separation efficiency of over 98%. With its superhydrophilicity and active functional groups, the resulting hydrogel was able to absorb dye molecules dissolved in water effectively. Due to the photothermal effect of carbon black, the local temperature of the hydrogel was increasing quickly under sunlight illumination, which allowed it to be an advanced platform for daily wastewater purification through solar distillation.

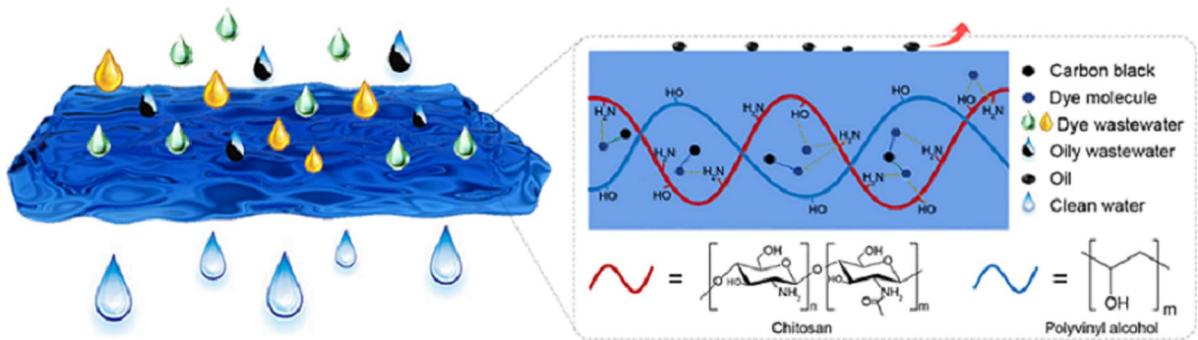
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Graphical abstract A versatile hydrogel platform for oil–water separation, dye adsorption, and domestic wastewater purification was developed.



Keywords Oil/water separation · Wastewater purification · Dye removal · Superhydrophilic · Hydrogel

Introduction

Nowadays, the oily wastewater discharged from daily activities and industrial production processes is increasing, and leakage accidents are frequent, posing a severe threat to drinking water safety, ecological environment, marine life, and human health (Kintisch. 2010; Peterson et al. 2003; Aurell and Gullett 2010; Shannon et al. 2008; Wang et al. 2015). According to statistics, the cost of treating oily wastewater exceeds 38.3 billion dollars per year (Arnold et al. 2004; Liu et al. 2018). Therefore, it is urgent to develop an efficient and economical method to treat oily wastewater. General oily pollution treatment technologies include skimming (Nam et al. 2018), adsorption (Saruchi et al. 2015; Saruchi et al. 2016a, b; Saruchi et al. 2019), flotation (Saththasivam et al. 2016), centrifugation (Turano et al. 2002), chemical flocculation (Mostefa and Tir 2004), and in situ burning (Gelderena et al. 2017), etc. Traditional treatment technologies are inefficient and costly and suffer from secondary pollution. Designing porous membranes with opposite wetting behavior to water and oil has proven to be a good alternative for oil–water separation (Yuan et al. 2008; Xue et al. 2014; Wei et al. 2018). Hydrophobic/oleophilic membranes are typically developed for oil–water separation, but this type of membrane is easily contaminated by oil during the separation process, causing the desired separation

efficiency to be decreased significantly. Moreover, water is always contacting the membrane before oil, due to its bigger density, which will prevent the separation. To overcome these limitations, separation membranes with underwater superoleophobicity have been explored, and they are more resistant to oil fouling when immersed in water. However, the oil will adhere to the membrane surface when the loss of water occurs, leading to decreased separation performance in continuous oil–water separation. Designing membranes with hydrophilicity and oleophobicity in both air and underwater is hoped to address these issues but has been proven to be difficult to realize, as it requires a complex interface with surface energy greater than water. Recently, Kong et al. produced a polymer blend coated mesh, which can exhibit superhydrophilic/superoleophobic properties after surface reconfiguration of the coating (Kong et al. 2021). Our group also developed a poly(vinyl alcohol) composite hydrogel coating that has water-attracting and oil-repelling in air, water, and oil surroundings (Lu et al. 2020). Furthermore, Wang et al. developed a superhydrophilic, underwater superoleophobic cellulose hydrogel-coated mesh (CHCM) produced from deep eutectic solvents. Based on this pre-wetted CHCM, the separation efficiency of various oil–water mixtures is above 98.0%, and the CHCM also has good recyclability and durability (Wang et al. 2021). Yang et al. firstly coated polydopamine (PDA) on the surface of paper fibers (PF@PDA) via $\text{CuSO}_4/\text{H}_2\text{O}_2$ triggering to accelerate the reaction rate. Then, the BaSO_4 nanosheets were deposited on the surface of PF@PDA membrane by alternating soaking process (ASP) to obtain superhydrophilic/underwater

superoleophobic membrane (PF@PDA/BaSO₄) for oil/water separation (Yang et al. 2020).

Apart from the oily wastewater problem, discharge of dye wastewater also leads to serious environmental pollution as well as resource waste. With the continuous increase of social demand, a large number of commodity dyes are used in paper, textile, paint, and other industries, and more than 280 thousand tons of wastewater are discharged into the environment every year, causing the treatment of dyeing wastewater to be a big challenge (Kalsoom et al. 2015). Among all technologies for treating dye wastewater, the adsorption method has attracted much attention due to the higher efficiency, recyclability of sorbent, simplicity (Saruchi et al. 2016a, b; Saruchi et al. 2019; Saruchi et al. 2021). Due to the different physical and chemical properties of soluble dyes and insoluble oils, one approach can only be applicable for one of the two contaminants (Zhu et al. 2017). Therefore, it is urgent to develop a versatile platform that can effectively achieve both oil–water separation and dye removal.

In this study, we have developed a composite hydrogel coating by the freezing–thawing method using polyvinyl alcohol, chitosan, and carbon black as the main raw materials. The obtained hydrogel coating exhibited both water-affinity and oil-repellency, and oil droplets could move freely on the water-wetted hydrogel surface. It also displayed superoleophobicity underwater and had excellent oil fouling resistance when submerged in an oil bath. Exploiting its water-attracting and oil-repellent property, the hydrogel-coated mesh can be used for the oil–water mixture and oil-in-water emulsion separation with high separation efficiency and good reusability. Importantly, the polyvinyl alcohol, chitosan, and carbon black in the hydrogel network were able to interact with the dye molecules through electrostatic effect, hydrogen bond interaction, and π - π interaction, allowing the dyes dissolved in water to be removed by our hydrogel effectively. Considering the simple fabrication process and integration of multiple functions of our hydrogel coating, this study is hoped to offer new insight into the areas of membrane separation industries and water purification.

Experimental section

Materials

Polyvinyl alcohol (PVA, 1799, Mw = 75,000 Da, 99% hydrolyzed) and carbon black (CB) were obtained from Aladdin Chemical Reagent (Shanghai, China). Chitosan (CS, Mw = 130,000 Da, Deacetylation: 80%–95%, Viscosity: 20–300 cps), sodium hydroxide (NaOH), ethanol, and acetic acid (GR grade, $\geq 99.8\%$) were offered by Sinopharm Chemical Reagent Co., Ltd. Poly (diallyldimethylammonium chloride) (PDDA, Mw = 200,000–350,000 Da), perfluorooctanoic acid (signify as PFOA) were all obtained from Sigma-Aldrich. Sodium perfluorooctanoate (signify as PFOA-Na) with a concentration of 1.0 M was fabricated by the reaction of NaOH and PFOA in deionized water. Malachite green (MG), fluorescein sodium salt (FS), rhodamine B (RB), crystal violet (CV), orange II sodium salt (O II), sunset yellow FCF (SY), and methylene blue (MB) were purchased from Shanghai Macklin Biochemical Co., Ltd. Methyl orange (MO) was bought from Shanghai Aladdin Industrial Co., Ltd. All chemical reagents are of analytical-grade and used as received without further purification. Commercially available glass slide and copper mesh (200 mesh) were cleaned with acetone, ethanol, and deionized water successively in an ultrasonic cleaner before use.

Preparation of the PVA-Chitosan-Carbon black (PCC) hydrogel

1.5 g of PVA was magnetically dispersed into 15 mL of deionized water at a temperature of 90 °C to form solution A. 0.8 g of CS and 0.32 g of acetic acid were added into 25 mL of deionized water under stirring at 50 °C to form solution B. Solution A was added into solution B under stirring to form a hybrid solution. Then, 0.8 g CB and 0.4 g PDDA were ultrasonically dispersed in the mixed solution, denoted as solution

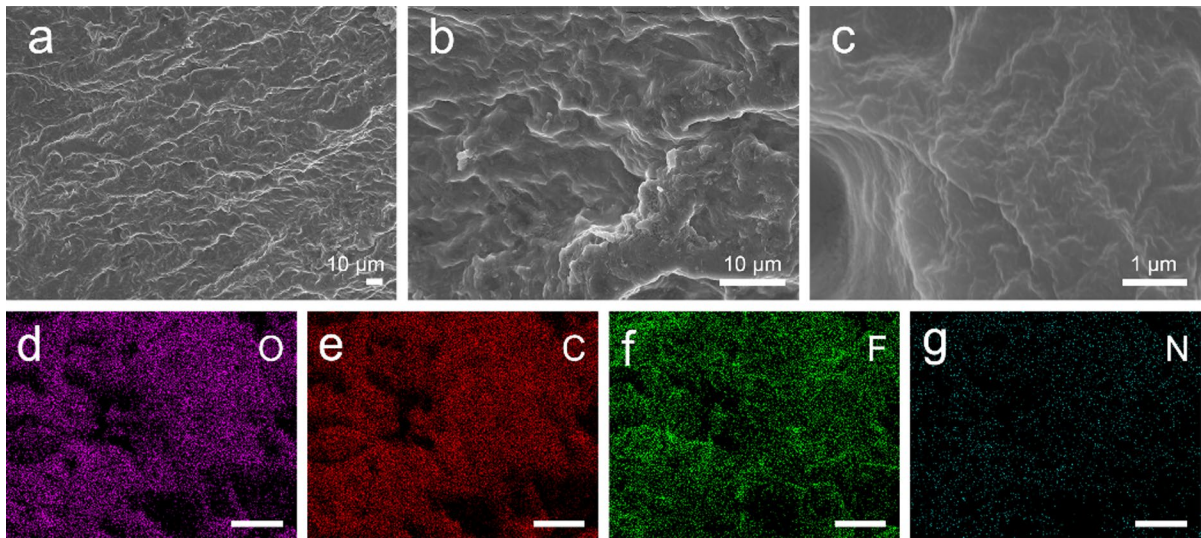


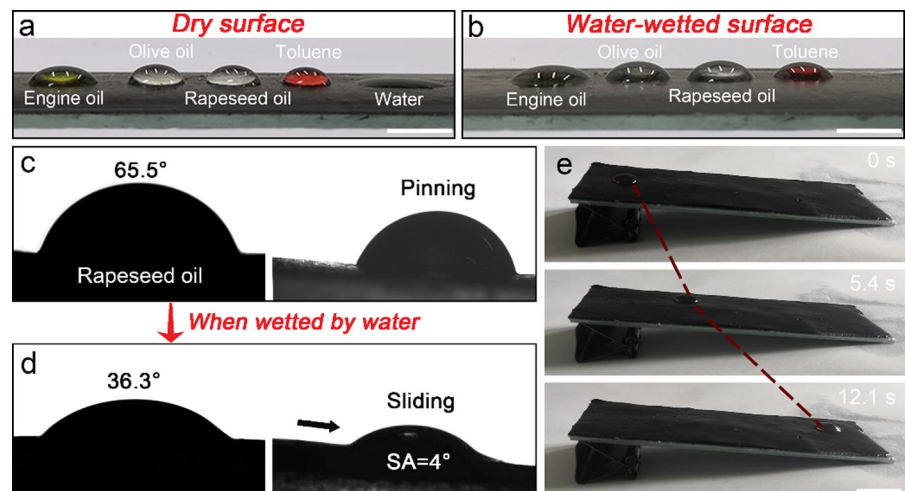
Fig. 1 FESEM images of the obtained PCC hydrogel at low and high magnifications **a–c**; Element distribution maps of the PCC hydrogel (**d–g**); The scale bar in images **d–g** is all 10 μm

C. The solution C was rapidly injected into a plastic dish. The dish containing solution C was frozen at $-25\text{ }^{\circ}\text{C}$ for 6 h and thawed at room temperature for 2 h, and this freezing and thawing treatment was repeated three times. After removing from the plastic dish, the PCC hydrogel was obtained. Finally, the PCC hydrogel was immersed into 1.0 M PFOA-Na solution for 5 min and washed with deionized water to achieve the slippery oil-repellent property.

Characterizations

Contact angle (CA) and sliding angle (SA) values were acquired using a Krüss DSA 100 (Krüss Company, Ltd., Germany) apparatus at room temperature. Scanning electron microscopy measurements were performed using JSM-6701F field-emission scanning electron microscopy (FESEM, JEOL, Japan). Energy-dispersive X-ray (EDX)

Fig. 2 Wetting property in air. Droplets of water (dyed by methylene blue) and different oil droplets on the dry (**a**) and water-wetted (**b**) PCC hydrogel surface; Oil droplet was pinning on the dry PCC hydrogel surface (**c**), while it was moving freely on the water-wetted hydrogel surface (**d** and **e**). The scale bar is 1 cm



spectroscopy analysis was conducted on a NORAN System Six X-ray microanalysis system (THERMO) connected to the FESEM system. We perform oil-in-water emulsion separation through a solvent filtration unit (Tianjin Jinteng Technology Co., Ltd.). The digital image of the distributed droplets in the oil-in-water feed emulsions and filtrates were acquired by an optical microscope (Stemi 508 trino Microscope, ZEISS). UV–visible absorption spectroscopy data were obtained using a HITACHI U-3010 UV–vis spectrophotometer. The PCC Hydrogel absorber was illuminated using a solar simulator (CEL-PE300L-3A). Thermal images were collected, using an IR Thermal Camera (FLIR Ts-650).

Results and discussion

Surface topography and chemistry analysis

The PCC hydrogel was developed through freezing–thawing treatment using CS, PVA, and CB as the raw materials. The CS was chosen due to its rich source, biocompatibility, and its active hydroxyl and amino groups, and PVA was applied to cross-link with CS to form a double network hydrogel that possessed enhanced mechanical property. CB with photothermal effect can convert solar radiation into heat, which was beneficial to power the evaporation of water.

The surface topography and chemistry of the obtained PCC hydrogel are shown in Fig. 1. As

shown in Figs. 1a and 1b, the hydrogel coating surface was un-flat and covered by raised textures with varied sizes, and there were some microporous structures distributed randomly between the raised structure (see Fig. 1c). The elements of oxygen (O), carbon (C), fluorine (F), and nitrogen (N) were well distributed on the hydrogel surface, according to EDX analysis (see Fig. 1d–g). Moreover, the content of the F element was found to be as high as 16.7%, implying that the PCC hydrogel surface was enriched in low-surface-energy fluorocarbon groups.

Surface wettability in the air surrounding

PCC hydrogel surface displayed water-attracting and oil-repelling properties in the air environment. As shown in Fig. 2a, the water droplet was able to wet the PCC hydrogel surface completely and immediately (less than 0.5 s), displaying a water CA of 0° eventually. In comparison, oil droplets such as rapeseed oil exhibited a CA of 65.5° and were pinning on the surface. Moreover, the rapeseed oil droplet did not show any diffusing even after 20 min, as shown in Fig. 2c and Figure S1 (a₁–d₁). Amazingly, when wetted by water, a water layer forms on the surface of the hydrogel, which acts as a lubricant to achieve slippery oil-repellent properties. As shown in Fig. 2b, all tested oil droplets with small CA values floated on the water-wetted hydrogel surface, and they could move freely on it due to the fluidity of the water lubricant. As shown in Fig. 2d and Fig. 2e, a rapeseed oil droplet was moving readily on the water-wetted PCC hydrogel surface tilted by 4° without any oil tracing left on the surface.

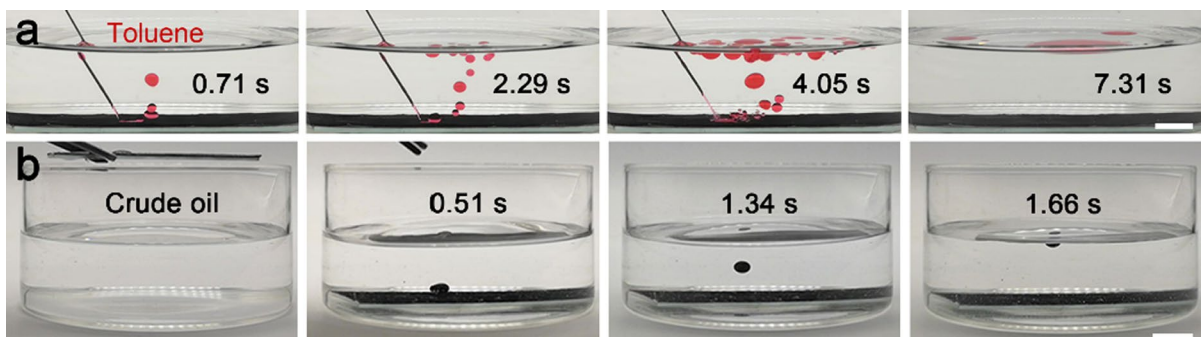
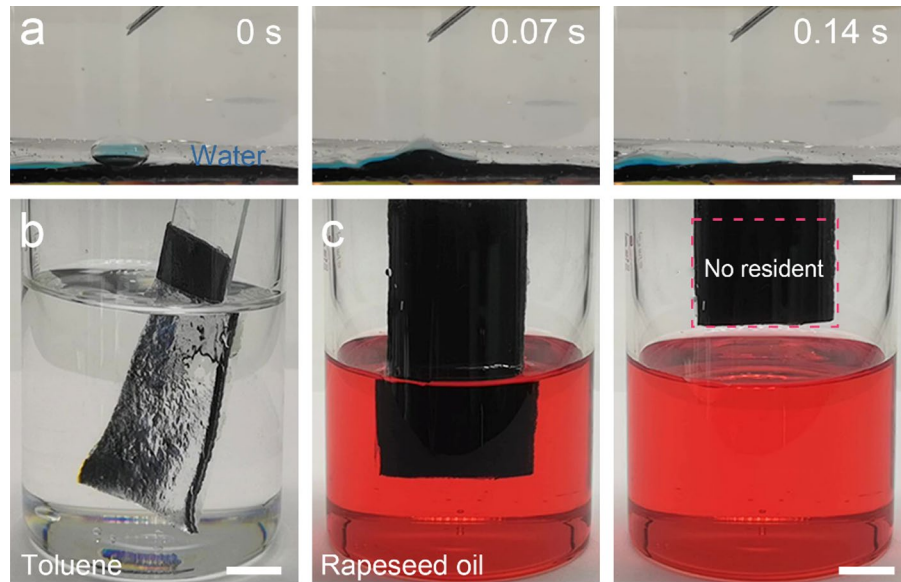


Fig. 3 Underwater wetting behavior analysis. No toluene (colored with Oil red) was pinning on the hydrogel surface after the oil-jetting test (a); Crude oil pinned on the PCC hydrogel was removed completely just by water immersion (b). The scale bar is 1 cm

Fig. 4 Wetting behavior under oil. PCC hydrogel had super-hydrophilicity in toluene (a); A mirror-like phenomenon was observed on the water-wetted PCC hydrogel surface submerged in oil (b); Even after 1 day of oil immersion, no oil was found on the PCC hydrogel surface (c). The scale bar is 1 cm

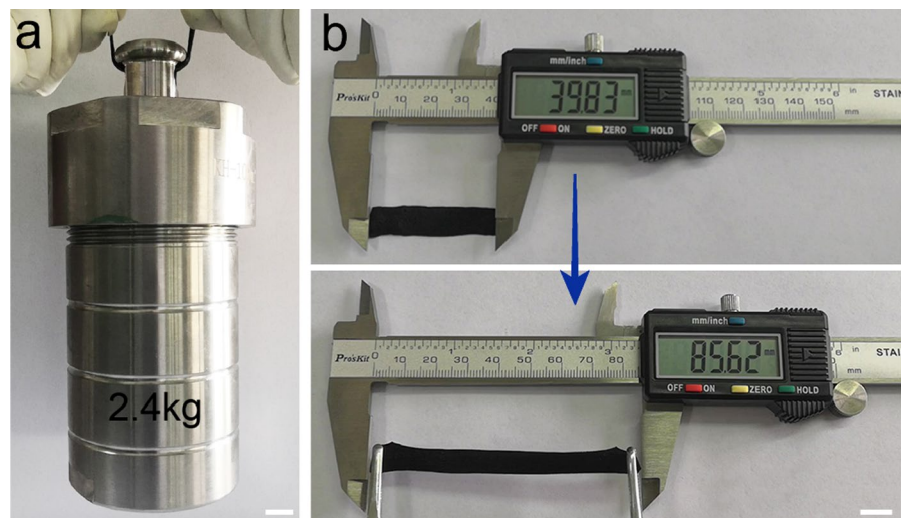


Mechanism of the water-attracting and oil-repelling wettability

The total surface free energy of a solid (γ_s) was considered as the sum of contributions from the dipole hydrogen bonding (γ_s^d) and dispersion force components (γ_s^p) (Fowkes, 1964; Owens and Wendt 1969; Fowkes et al. 1988). The water-attracting and oil-repelling properties can be achieved on one surface if it had an adequately big γ_s^p and an adequately small γ_s^d

simultaneously, based on recent research (Fowkes et al. 1988). As for the PCC hydrogel coating, the fluorinated groups minimize the γ_s^d constituent, resulting in oil repellency, whereas the hydrophilic carboxyl, hydroxyl, and quaternary ammonium groups were able to increase the γ_s^p constituent, leading to a strong affinity to water molecules. When wetted by water, the PCC hydrogel surface was covered by a water layer that can serve as a lubricant to resist oil droplets. With the help of the mobility of the water

Fig. 5 Mechanical robustness of the PCC hydrogel. PCC hydrogel can hold up a steel block with a weight of 2.4 kg (a); PCC hydrogel retains its original shape after the tensile test (b). The scale bar is 1 cm



lubricant, oils immiscible with water were moving easily on the water-wetted PCC hydrogel without any pinning (Fig. 2e). The detailed surface wettability mechanism was described in the Supporting Information section.

Underwater wetting behavior analysis

PCC hydrogel possessed extremely strong water affinity, which allowed it to display superoleophobic and self-cleaning underwater. All tested oil droplets exhibited oil CA greater than 150° and SA less than

5° on the PCC hydrogel surface underwater. Figure 3a shows that a jet of toluene was bouncing off the hydrogel surface readily without any residue leaving underwater. Sticky crude oil was further chosen to check the oil repellency of the hydrogel. The dry PCC hydrogel was fouled by dropping crude oil on it, and then the oil-fouled hydrogel was immersed in water to test its self-cleaning property. The result shows that the crude oil droplets pinned on the hydrogel surface shrank into spheres within a few seconds and then detached from the surface within two seconds, leaving a completely clean surface (see Fig. 3b). Thanks to

Fig. 6 Oil/water separation analysis. The PCC hydrogel coated copper mesh was used as a separation membrane for oil/water separation (a); Variation of separation efficiency for different oil/water mixtures; The T-W, R-W, O-W, E-W, and H-W in image b represented toluene-water, rapeseed oil–water, olive oil–water, engine oil–water, and hexadecane-water, respectively (b); The oil/water separation efficiency of the separation membrane as a function of separation cycles (c)

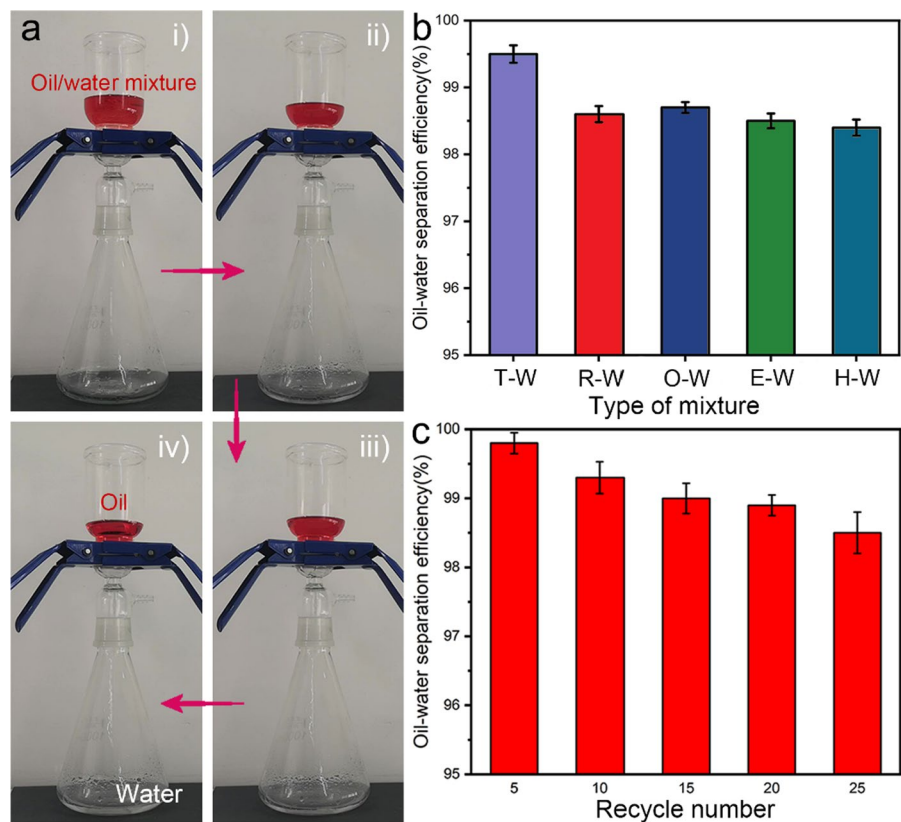
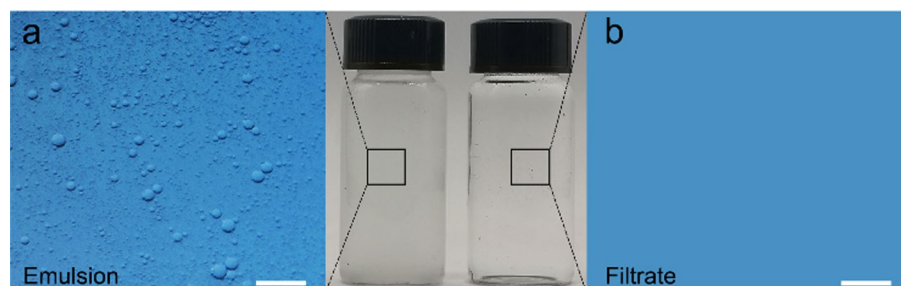


Fig. 7 Oil-in-water emulsion separation. Microscopic images of hexadecane in water emulsion (a) before and (b) after separation. The scale bar is 20 μ m



its strong water affinity, a hydration layer around the hydrogel was forming, which could serve as shielding to reduce the oil-solid interface and decrease the oil adhesion strength (Zhang et al. 2018).

Wetting behavior in oil surrounding

We next study the wetting behavior of the PCC hydrogel under an oil environment, to further analyze the water affinity strength of the hydrogel. As shown in Fig. 4a, a water droplet (dyed with methylene blue) was able to wet the PCC hydrogel surface completely with a time less than 0.14 s under oil conditions, when contacting the PCC hydrogel under oil. This phenomenon indicated that the PCC hydrogel could still bind water strongly even immersion in oil. Interestingly, a mirror-like phenomenon was observed on the water-wetted PCC hydrogel surface summered in an oil bath. This was because a water layer forming on the PCC hydrogel surface can act as a shielding layer to prevent the oil from contacting the surface (see Fig. 4b). Importantly, the water layer remained stable even after immersion in oil for one day, allowing the PCC hydrogel surface to be completely clean when taken out from oil (see Fig. 4c). These results indicated that the PCC hydrogel had a strong water affinity strength even immersion in an oil bath for a long time.

Mechanical property analysis

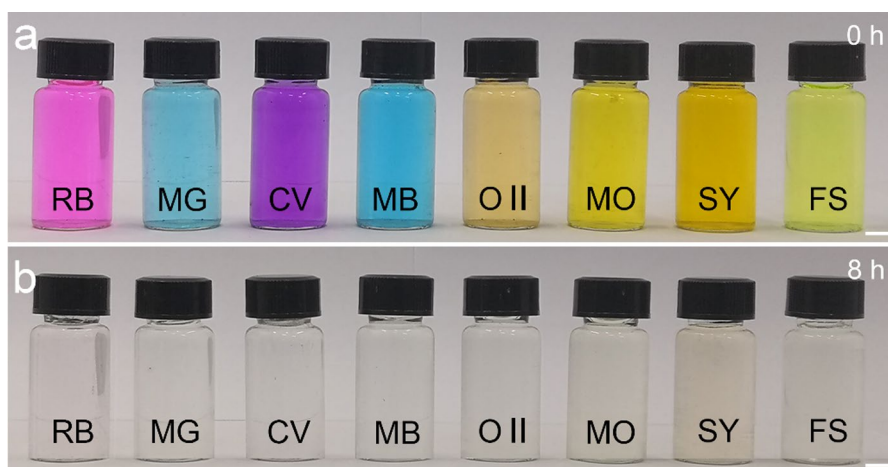
The mechanical properties of the obtained PCC hydrogel were also analyzed, and the result showed

that it possessed enhanced stiffness and elasticity. As shown in Fig. 5a, a PCC hydrogel sample (5.3 cm × 2.2 cm × 0.25 cm) can support a 2.4 kg steel block, implying its high mechanical strength. However, the single network PVA hydrogel sample with the same shape failed when loaded with the same weight (as shown in Figure S2). The obtained PCC hydrogel also had improved elasticity. As shown in Fig. 5b, PCC hydrogel could be stretched to 215% of the original length, and it was restored to its original shape when the load was removed.

Oil/water separation analysis

The PCC hydrogel-coated mesh is hydrophilic and oil-repellent, and it is a suitable membrane for oil/water separation due to these properties. When the oil–water mixture (namely water/toluene mixture) was contacting the separation membrane, water was passed through the membrane and collected underneath, while oil was remaining above it without any permeation (see Fig. 6a). Other types of oil/water mixtures can be separated in the same way with high separation efficiency (see Fig. 6b). Thanks to its slippery oil repellency, residual oil stains on the membrane surface after separation can be easily removed by water rinsing. Moreover, the the membrane still had enhanced separation efficiency to the water/toluene mixtures with varied pH value and temperature, as shown in Figure S5. Importantly, the hydrogel-coated filter still had a separation efficiency of over 98% even after 25 oil–water separation cycles (see Fig. 6c).

Fig. 8 Dye adsorption analysis. The dye-colored aqueous solution before (a) and after (b) adsorption. The scale bar is 1 cm



The PCC hydrogel-coated copper mesh can also be used for oil-in-water emulsion separation. As shown in Fig. 7, the filtrate (hexadecane-in-water) changed from milky white to transparent after one cycle of separation. No hexadecane droplets with the size in the range of 1–7 μm were observed in the filtrate, as evidenced by optical microscope analysis (as shown in Fig. 7b). When the oil-in-water emulsion contacted the PCC hydrogel-coated copper mesh, the continuous water phase quickly penetrated the hydrogel surface to form a water hydration layer and then continuously passed through the coated mesh with the help of gravity and capillary force. Meanwhile, oil droplets dispersed in the water bath were repelling by the hydration layer around the PCC hydrogel, and they were gathering together, growing into oil droplets of bigger size, and floating up the water surface to become a free oil layer eventually. As a result, emulsion separation was realized by the PCC hydrogel-coated copper mesh.

Removing dyes from water

Discharge of dye wastewater leads to serious environmental pollution as well as resource waste. Herein, our created PCC hydrogel can be applied as an absorbent platform for removing dyes from water. The PCC hydrogel sample with the weight of 1 g was placed into the RB dye-colored aqueous solution (18 mg/L, 10 ml) at room temperature. When contacted with the RB aqueous solution, the RB dye molecular was absorbed in the PCC hydrogel network instantaneously. Automatically, the color of the RB aqueous solution was fading gradually with the time going on. The RB dye-colored solution became transparent after PCC hydrogel absorption for 8 h, and no RB was found in the solution, evidenced by UV–vis spectra analysis (see Figure S6). Other types of dyes such as MG and CV dissolved in water can also be absorbed efficiently in the same way, as shown in Fig. 8. Furthermore, our PCC hydrogel can also function as a filter module for dyes

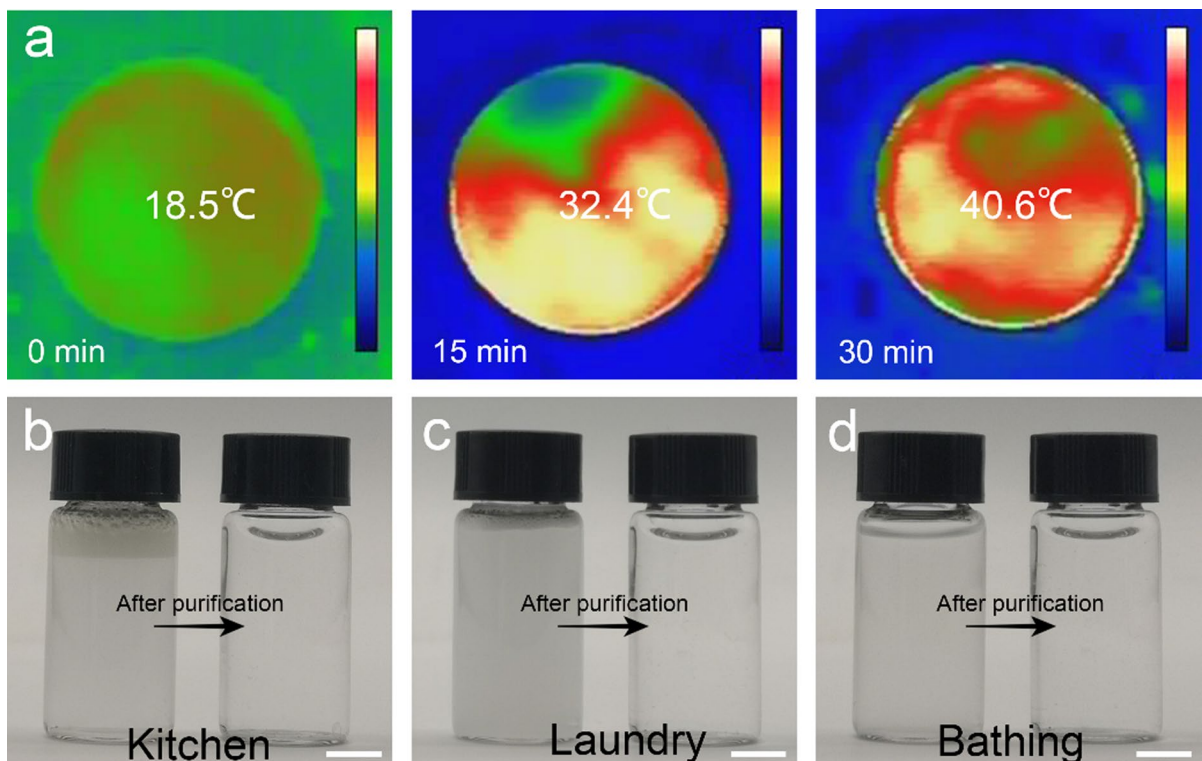


Fig. 9 Wastewater purification was achieved by PCC hydrogel. Infrared thermal images show the temperature of the PCC hydrogel surface under sunlight irradiation (a); Images of the

wastewater gathered from the kitchen, laundry, and bathing before and after purification (b–d). The scale bar is 1 cm

removal, after putting the powdered hydrogel into a syringe. The dyes including MB and CV were retained above the filter (see Figures S8 a and b), and the filtrate became transparent after one cycle of treatment (see Figures S8 c and d). These results showed that the obtained PCC hydrogel was a good candidate for removing dyes from water.

As for the PCC hydrogel, the water-affinity of the PCC hydrogel could provide sufficient contact between it and the dyes molecular in the solution (Bazrafshan et al. 2013; Holkar et al. 2016). Importantly, the unique chemistry of the PCC hydrogel offered the active sites for dyes absorption. The active amino and hydroxyl groups on the CS molecular chain can chelate and adsorb dyes by electrostatic effect and hydrogen bond interaction (Nikolaos et al. 2007; George et al. 2010, 2008; Chandi et al. 2021). With the help of the hydroxyl groups in the PVA, the dye absorption performance of PCC hydrogel was further improved. Also, the well-distributed CB particles in the hydrogel were able to interact with dyes by π - π interaction (Tian et al. 2020; Das et al. 2008; Ramesha et al. 2011; Xiao et al. 2016). The combination of electrostatic effects, hydrogen bonding interactions, and π - π interactions of PCC hydrogels enables the efficient removal of dyes dissolved in water (see Figure S7). Moreover, the absorption capacity of the PCC hydrogel was varied to the dye solutions with different pH values and concentrations, as shown in Figure S9.

Daily wastewater purification

Finally, the PCC hydrogel was demonstrated as an advanced platform for daily wastewater purification through solar distillation. Herein, a homemade device was designed for water purification as shown in Figure S10. The PCC hydrogel sheet was floating on the wastewater surface, the transparent plastic wrap, pebbles, and the beaker located in the wastewater container were applied for water vapor generation, water condensing, purified water movement guiding, and purified water containing, respectively. Due to the photothermal effect of CB, the local temperature of PCC hydrogel rapidly rises from 18.5 °C to 40.6 °C under sunlight illustration (see Fig. 9a), which allowed it to power water evaporation. When touching the plastic wrap, the water vapor condensed on it, and then the condensed water was guided to

flow on the bent plastic wrap and finally collected in the beaker underneath. Figures 9b, c, and d show the wastewater images in our daily life gathered from the kitchen, laundry, and bathing before and after purification. The result showed that all three solutions changed from milky to transparent after purification. Moreover, there was no emulsified droplet in the purified water evidenced by dynamic light scattering analysis, and the pH values for the three water samples after purification were all close to 7 (see Figure S11 and Figure S12). The PCC hydrogel exhibited a high water steam generation rate of $\sim 1.21 \text{ kg m}^{-2}\text{h}^{-1}$ under one sun illumination. These results implied that our PCC hydrogel is hoped to be a promising alternative for daily wastewater purification.

Conclusions

In summary, a PCC hydrogel was fabricated through the freezing–thawing method. The resulting PCC hydrogel displayed both water-attracting and oil-repellency in air, water, and oil surroundings. Water was able to wet the hydrogel surface completely and easily. Wetted hydrogel surfaces appear oil repellent in air, have superoleophobicity underwater, and show resistance to oil fouling when submerged in oil. Due to the crosslinking between CS and PVA, the resulting dual network PCC hydrogel possessed enhanced stiffness and elasticity. Because of its water-affinity and oil-repellency, the hydrogel-coated mesh was able to separate immiscible oil–water mixtures and oil-in-water emulsions with high separation efficiency. The PCC hydrogel was able to interact with the dye molecules through electrostatic effect, hydrogen bond interaction, and π - π interaction, allowing it to serve as an absorbent to remove dyes from water effectively. Finally, the PCC hydrogel was demonstrated as an advanced platform for daily wastewater purification, through solar distillation, exploiting its photothermal effect-induced temperature accelerating. Possible applications of our PCC hydrogel could be found in the areas of membrane separation industries, wastewater purification, and sewage treatment.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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