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The Smart Hydrogels for the Controllable Adsorption and Desorption of PFAS

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Abstract
In order to address the regeneration issue of adsorbents for the elimination of per- and polyfluoroalkyl substances (PFAS), this work synthesized a novel smart hydrogel based on a temperature-responsive polymer, poly(N-isopropylacrylamide) (PNIPAm). These hydrogels exhibit hydrophobicity and hydrophilicity above and below the the lower critical solution temperature (LCST) of PNIPAm, respectively. Concurrently, the monomer 2-(methacryloyloxy)ethyl)trimethylammonium chloride (MTAC), distinguished by its quaternary ammonium groups, was co-polymerized with NIPAM to augment its electrostatic effect. Utilizing perfluorooctanoic acid (PFOA) as a case study, the smart hydrogel attained an 80% desorption efficiency through the mere adjustment of temperature, obviating the necessity for salt solution or organic solvent. This work is expected to overcome the challenges associated with the regeneration of adsorbents and contribute to the sustainable management of PFAS.
Keywords: PFAS, smart hydrogels, adsorption, regeneration

1. Introduction

PFAS have been utilized across a wide range of industrial sectors for several decades, and detected on a global scale exhibiting. . PFAS have the potential to accumulate within specific ecosystems and organisms due to the high degree of persistence, Causing public concern. The adsorption method has become a prevalent approach in practical applications, primarily due to its established technological foundation and uncomplicated operational characteristics[1]. Nevertheless, the issue that restricts the widespread utilization of these materials is the regeneration of the adsorbents that have been exhausted[2]. Conventional methods exhibit a range of drawbacks: thermal treatment consumes energy and causes loss of the adsorbent, in-situ contaminant degradation damages the adsorbent structure, and the solvents for elution are toxic, dangerous and expensive. In order to address the issue of regeneration, a new type of smart hydrogel based on a temperature-responsive polymer PNIPAm was proposed. The material's temperature-responsive function is characterized by its hydrophilic properties at temperatures below the LCST, and hydrophobic properties at temperatures above the LCST[3]. The hydrophobic effect is one of the most significant driving forces for PFAS adsorption. It is hypothesized that by modulating the hydrophilicity and hydrophobicity of the smart hydrogel through temperature control, it will be possible to achieve controllable adsorption and desorption of PFAS. This will facilitate convenient regeneration without reliance on organic solvents and salt solutions.

2. Materials and Methods

2.1 Synthesis of smart hydrogels

In the typical synthesis procedure, 200 mg NIPAM (N), a certain amount of MTAC (N^+) and 20 mg N,N′-methylenbisacrylamid (MBA) were added to 1 ml of water, mixed evenly, and then 50 µl of 40% sodium persulfate (PS) solution was added into the solution to start the polymerization reaction, and the solution was mixed. The mixture was left to stand for 24 h to finally obtain a hydrogel. Further synthesis procedures were conducted under changing the dosage of N^+ to adjust the adsorption and desorption properties of the hydrogel. According to the different dosages of N^+, hydrogel materials named pN, pN/N^+-1, 2, 3, 4 and 5 were obtained. pN^+ was prepared by the same process without the addition of N.

2.2 Experimental Procedure

Regarding to the adsorption experiments, a certain mass of hydrogel was added into a glass bottle with PFOA solution in deionized water. The bottle was put to a shaker with temperature control at 45°C (unless specifically stated) for adsorption. After 24 h, the hydrogels were taken out and put into another glass bottle with water (or salt solution) and shaking at 25°C for desorption. Samples were taken at some time points during the ad-/desorption process. The desorption efficiency is calculated by the equation below:

$$Desorption\ (\%) = \frac{m_{PFOA}(25°C\ desorbed)}{m_{PFOA}(45°C\ adsorbed)} \times 100\%$$

3. Results

The structure of the smart hydrogel is shown in Fig. 1a. It has the temperature response part N, the positive charge

part N^+ and the cross-linker MBA. Seven smart hydrogels with different ratio of N and N^+ show different performance for the adsorption of PFOA at different temperature (25 and 55℃). The pN hydrogel shows low adsorption capacity due to the lack of charge-balancing adsorption sites for the negatively charged PFOA, while pN^+ shows relatively good adsorption due to the abundant cationic sites at two temperatures. Further, hydrogels 1-5 obtained by the copolymerization of the two monomers N and N^+ exhibit different temperature response strengths. Among them, the largest difference (nearly 60%) in adsorption performance at the two temperatures was found in pN/N^+-2 (Fig. 1b). Therefore, hydrogel pN/N^+-2 was selected to further investigate its adsorption mechanism.

The adsorption isotherms of pN/N^+-2 and pN^+ at different temperatures are shown in Fig. 1c and d. Obviously, the q_e values (loading of PFOA on the hydrogel) of pN/N^+-2 at the same c_e (aqueous phase PFOA concentration) is greatly affected by temperature, while that of pN^+ is less affected. This further proves the temperature response function of pN/N^+-2. Besides, according to the rising trend of q_e values in the adsorption isotherm, the response temperature of pN/N^+-2 is around 35℃, thus 45 and 25 ℃ are selected as the temperatures for the adsorption and desorption experiments, respectively.

Furthermore, after adsorption of PFOA at 45℃, the desorption performance of pN/N^+-2 was also investigated with deionized water (blank) as well as in presence of different concentrations of different inorganic ions in the solution (including Cl^-, NO_3^- and SO_4^{2-}) at 25℃. 80% adsorbed PFOA was released with only deionized water. This indicates that the temperature response effect has the potential for application in controlled desorption to achieve regeneration. In addition, by adding a small amount of inorganic anions to the water, the desorption rate can be further increased to 100%. In future, the smart hydrogels need further tests via fixed-bed column experiments under continuous flow conditions to explore its application potential.

4. Conclusion

In this work, temperature-responsive monomers and cation-containing monomers were polymerized to synthesize smart hydrogels by a simple copolymerization method. The composition ratio was precisely tuned to optimize the best hydrogel with both temperature response and adsorption capacity. Temperature-dependent adsorption isotherms demonstrated that the adsorption capacity varied with temperatureand the switch temperature. Further, 80% PFOA could be desorbed by using deionized water at room temperature and further addition of a small amount of inorganic salts achieved a complete desorption, proving the potential of the smart hydrogel for switchable ad-/desorption. This smart hydrogel provides a new design idea for renewable adsorbents for PFAS removal.

5. Acknowledgment

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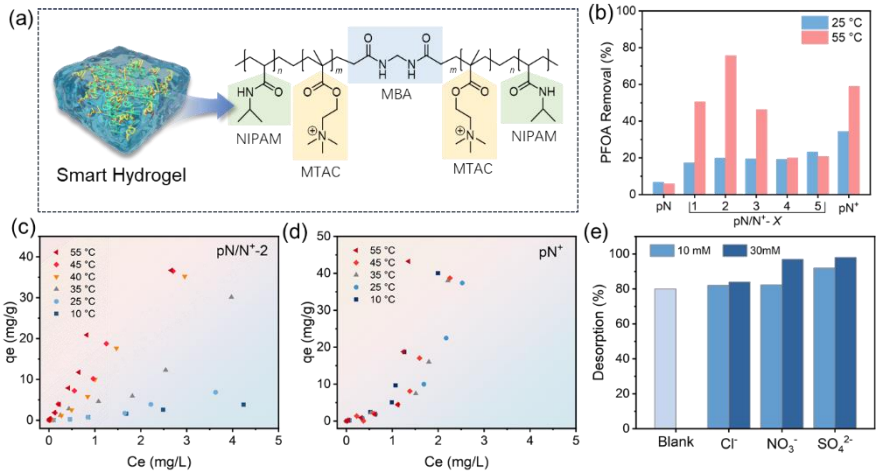

Figure 1. (a) the structure of the smart hydrogel; (b) the removal of PFOA by different smart hydrogels via adsorption ([PFOA]= 2 mg/L, [adsorbent]= 0.2 g/L, pH= 5.6); the isotherm of (c) pN/N^+ and (d) pN^+ ([PFOA]= 0.01-10 mg/L, [adsorbent]= 0.2 g/L, pH= 5.6); (e) the desorption of PFOA by pN/N^+ under different conditions ([adsorbent]= 0.2 g/L, [ions]= 10 or 30 mM)

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