Proceedings of the 5th International Conference on Materials Chemistry and Environmental Engineering
DOI: 10.54254/2755-2721/123/2025.19710

Design Principles and Frontiers of Applications for Thermo-Responsive and Self-Healing Hydrogels

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Abstract: Hydrogels are a class of hydrophilic polymer networks formed through chemical or physical cross-linking, which have been widely used in the biomedical field due to their high water absorption and biocompatibility. This article deeply analyzes the principles, classification, synthesis methods, physicochemical properties, and applications of temperature-sensitive hydrogels and self-healing hydrogels in drug release, tissue engineering, and other fields. By revealing their characteristics and mechanisms of action, it provides theoretical support for further research on hydrogels, looks forward to future research directions, and promotes innovation and development in practical applications.

Keywords: hydrogels, temperature sensitivity, self-healing, biomedical applications, drug delivery

1. Introduction

Hydrogels are water-swollen cross-linked polymer structures[1], formed by hydrophilic polymer chains through chemical or physical cross-linking, capable of retaining a large amount of water in their pores[2]. They also have a porous structure, behaving similarly to natural living tissues, making them bioactive materials used in tissue engineering with high biocompatibility with living tissues[3]. The first reported hydrogel can be traced back to 1960 when Wichterle and Lim synthesized poly(2-hydroxyethyl methacrylate) and used it in the contact lens industry[4].

Hydrogels, with their adjustable mechanical properties, controllable biodegradability, and multifunctionality, have been widely used in today's society. Their three-dimensional polymer network structure can efficiently accommodate and lock in more than 90% of their own weight in water while maintaining structural integrity and stability[5]. In addition, the core advantage of hydrogels lies in the natural hydrophilicity of their polymer chains, allowing them to swell and absorb a large amount of water, forming a gel-like substance similar to natural tissues, thereby simulating and optimizing the physical properties of biological interfaces[6]. Therefore, in the medical field, hydrogels are widely used in drug controlled release systems, scaffold construction in tissue engineering, and efficient wound care dressings[7].

A search for "hydrogels" in the PubMed database shows a rapid increase in the number of published articles[8]. However, most articles conduct in-depth research on a specific aspect or horizontally expand on hydrogel reviews, with fewer articles expanding vertically. Therefore, this article provides a new perspective while summarizing the current state of hydrogel research and offers theoretical support for hydrogel research.

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2. Temperature-Sensitive Hydrogels

2.1. Principle of Temperature-Sensitive Hydrogels

The degree of swelling of hydrogels is the result of the interaction between swelling force and elastic retractile force. When water molecules interact with hydrophilic groups in the polymer network, the swelling force drives them to swell, while the elastic retractile force of the polymer chains tries to contract them. Dry hydrophilic polymer networks (i.e., dry gels) begin to absorb water and gradually form swollen hydrogels upon contact with water[9].

In the initial stage of swelling, the swelling force dominates, causing the volume of the hydrogel to increase rapidly. As the swelling process progresses, the elastic retractile force gradually increases. When the elastic retractile force equals the swelling force, the hydrogel enters a state of equilibrium swelling, and its volume remains relatively stable unless environmental conditions change.

For temperature-sensitive hydrogels, changes in temperature will cause changes in both the swelling force and the elastic retractile force, eventually equalizing, and the hydrogel swells to a balanced state. The most significant characteristic of temperature-sensitive hydrogels is that when the environmental temperature reaches a certain critical value (i.e., the lower critical solution temperature, LCST), the hydrogel undergoes a phase change. This temperature-responsive property provides unique advantages for its application in biomedical and other fields.

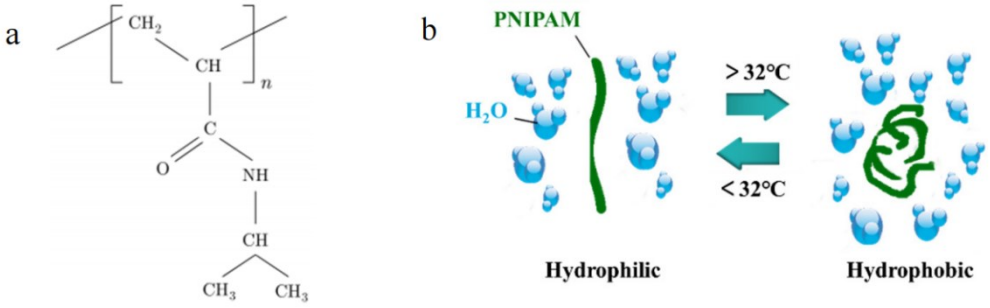


Figure 1: Chemical structure (a) and temperature respond (b) of PNIPAM.

Poly(N-isopropylacrylamide) (PNIPAM) has an LCST of 32°C, close to human body temperature, thus having extensive research value and application potential. Its side chains contain both hydrophilic groups (-$CONH_2$) and hydrophobic groups ($CH(CH_3)_2$). Below the LCST, water molecules form hydrogen bonds with the amide groups of the hydrogel, thus exhibiting good hydrophilicity. Under the combined influence of hydrogen bonds and van der Waals forces, the hydrogel chain segments have good dispersibility in the solution and can dissolve well in aqueous solutions. However, as the temperature rises, the binding force between the hydrogel chain segments and water molecules begins to weaken, and some hydrogen bonds are broken by heat. This leads to the dominance of molecular hydrophobicity, and macroscopically, it gradually turns into a solid state[10].

2.2. Types of Temperature-Sensitive Hydrogels

Based on different temperature responses, temperature-sensitive hydrogels can be mainly divided into two types: positive temperature-sensitive hydrogels, such as PNIPAM, which gradually contract as the temperature rises. The drug release in the biomedical field takes advantage of the property that drugs can be rapidly released when the local temperature rises. The other type is negative temperature-sensitive hydrogels, which maintain their hydrophilicity or even further swell when the

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temperature rises. Their applications are relatively few, but they still have significant value under certain specific environmental conditions.

2.3. Synthesis of Temperature-Sensitive Hydrogels

2.3.1. Free Radical Polymerization Method

Free radical polymerization is a polymerization reaction initiated by free radicals, causing chain growth radicals to continuously grow. It is mainly applied to the addition polymerization of olefins, using olefin monomers containing unsaturated double bonds as raw materials. By opening the double bonds in the monomer molecules, multiple addition reactions are carried out between molecules, thereby linking monomers into large molecules.

Free radical polymerization belongs to chain polymerization reactions, divided into four elementary reactions: chain initiation, chain growth, chain termination, and chain transfer. Chain initiation mainly involves two steps: forming an active center - free radicals, and free radicals initiating monomers. Chain growth is the repeated and rapid addition of active monomers to monomer molecules, forming large molecule free radicals. Chain termination is mainly due to the interaction of two free radicals, forming stable molecules. Chain transfer is the chain free radical from monomer, solvents, initiators, and other small molecules or large molecules that have already formed, causing these molecules that have lost atoms to form new free radicals.

To prepare hydrogels with N-isopropylacrylamide (NIPAAm) as the monomer, free radicals are needed to initiate the polymerization reaction. These free radicals are usually produced by initiators, which are divided into thermal initiators and photo initiators, i.e., molecular bonds are broken by temperature or light to produce free radicals[11]. In addition, cross-linking agents such as N,N'-methylenebisacrylamide, which contain two or more functional groups that can participate in polymerization, can cross-link with monomers during the polymerization process to form a three-dimensional network structure, giving the hydrogel good shape stability.

2.3.2. Copolymerization Method

On the basis of free radical polymerization, other monomers can also be added for copolymerization to obtain hydrogels with better comprehensive performance. For example, temperature-sensitive hydrogels can be prepared by copolymerizing NIPAAm and 2-acrylamido-2-methylpropanesulfonic acid (AMPS). The temperature sensitivity of the hydrogel can also be controlled by adjusting the ratio of monomers. For example, increasing the content of AMPS can increase the LCST of the hydrogel, thereby adjusting its temperature sensitivity.

2.4. Physicochemical Properties of Temperature-Sensitive Hydrogels

2.4.1. Temperature Sensitivity

Temperature-sensitive hydrogels, such as poly(N-isopropylacrylamide), exhibit hydrophilicity below the LCST and transition to hydrophobicity above the LCST. Therefore, as the temperature rises, the hydrogel undergoes a phase change from swelling to contraction. The LCST is usually around 32°C, but it can be adjusted by changing the ratio of copolymer monomers. For example, adding hydrophilic monomers such as acrylic acid (AA) can increase the LCST, while adding hydrophobic monomers such as ethyl acrylate (EA) and butyl acrylate (BA) will lower the LCST. Utilizing temperature sensitivity to perceive external changes and respond, combined with sensors, has a wide range of applications in drug release or environmental detection.

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2.4.2. Swelling Performance

Swelling performance, as an important property of hydrogels, is affected by the type of copolymer monomers, cross-linking degree, temperature, and pH value. The swelling rate shows a trend of first decreasing and then increasing with the increase of buffer solution pH value, that is, the saturation swelling rate is larger in acidic and alkaline buffer solutions, and smaller in neutral and near-neutral buffer solutions. In addition, the swelling rate increases with the increase of alginate (SA) and acrylic acid (AA), and decreases with the increase of cross-linking agents, ethyl acrylate (EA), and butyl acrylate (BA)[12].

2.4.3. Fatigue Resistance

In addition, compared to the easy fatigue of metal materials, some temperature-sensitive hydrogels have high fatigue resistance and can be stretched repeatedly for more than 100,000 times, which makes them widely used in biologically related dynamic environments. The team of MIT's Zhao Xuanheo introduced controllable nano-crystal domains to increase the fatigue threshold of hydrogels to more than 1000 J/m^2[13].

2.5. Applications of Temperature-Sensitive Hydrogels

2.5.1. Cancer Immunotherapy

The advantage of temperature-sensitive hydrogels is that they can spontaneously gelate according to temperature changes, thus precisely delivering antigens or vaccines to the target physiological area and then releasing them. Hydrogels not only enhance immunogenicity but also promote antigen-mediated immune responses, thus serving as effective adjuvants to improve the efficiency of the entire system. In addition, the polymer matrix of hydrogels has the ability to degrade in physiological environments, further enhancing their application potential.

In the field of anti-tumor immunotherapy, vaccine therapy has become a key strategy to effectively inhibit tumor recurrence and metastasis. Activating anti-tumor immunity through vaccination is both efficient and safe, providing patients with a reliable way to avoid disease recurrence. Compared with systemic drug administration, temperature-sensitive hydrogels can release tumor antigens locally, not only improving treatment safety but also potentially stimulating systemic anti-tumor immune responses, thus achieving more accurate and effective treatment[14].

2.5.2. Prevention of Postoperative Adhesions

Postoperative adhesions, as a common postoperative complication, have an incidence rate between 50% and 97%, especially in the abdomen and joints. They are a natural phenomenon in the healing process of surgical sites but can cause serious problems such as infertility, pain, and intestinal obstruction. To address this challenge, Chen and others developed a new material by grafting PNIPAM onto chitosan (CS) and coupling it with hyaluronic acid (HA), successfully synthesizing a temperature-sensitive hydrogel HA-CS-PNIPAM. This hydrogel exists in a free-flowing aqueous solution state at room temperature, which is convenient for injection, and its LCST is about 31°C, with good temperature responsiveness, and has a broad application prospect[15].

Cell culture experiments show that HA-CS-PNIPAM hydrogel exhibits excellent barrier performance in vitro, which can effectively reduce the induction of cell toxicity and the permeation of fibroblasts. In the rat lateral wall defect intestinal wear model, compared with PNIPAM and untreated control groups, HA-CS-PNIPAM hydrogel showed a more significant anti-adhesion effect. After 2 weeks of treatment, scanning electron microscopy observation showed no signs of abdominal

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interruption. At the same time, histological and blood test results indicated that the material did not cause organ damage or acute toxicity reactions, further demonstrating the excellent effect of the hydrogel.

The preparation process of HA-CS-PNIPAM hydrogel is simple, and the effect is better when applied, so it can be used as an ideal anti-adhesion barrier injection material after abdominal surgery, providing an effective solution to reduce the occurrence of postoperative adhesions.

2.5.3. Flexible Strain and Temperature Sensing

Ion-conductive composite hydrogels prepared by adding LiCl and glycerol through free radical polymerization have excellent flexibility and transparency[16]. This hydrogel shows an excellent strain sensitivity of 17.3 in a wide strain range of 1973%. In addition, under the influence of strong ion hydration and strong hydrogen bonding, it shows excellent temperature sensing performance in the wide temperature range of -30 to 80°C. It not only has a high thermensitivity of 5.51%/°C but also accurately captures small temperature changes in the range of 36.5 to 40°C with a high resolution of 0.2°C.

Therefore, hydrogels are expected to become candidates for multifunctional flexible sensors (flexible sensors are sensors with elasticity and bendability that can detect various physical quantities such as pressure, temperature, and humidity, and are widely used in health monitoring, smart wearable devices, and robots), and expand their application in human-computer interfaces and soft robots. For example, the team of Professor Zhu Yutian from Hangzhou Normal University prepared flexible and transparent ionic gels with temperature and strain sensing functions based on temperature-sensitive hydrogels, which are applied to wearable devices and flexible electronic devices[17].

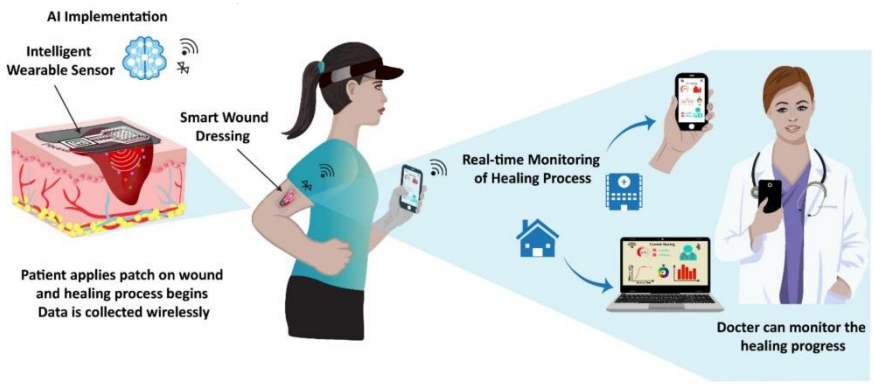


Figure 2: Engineering intelligent composite hydrogels for wearable health monitoring.

2.5.4. Tissue Engineering

Mebiol® Gel is a commercial temperature-sensitive hydrogel that can maintain a sol state at low temperatures and turn into a gel state at body temperature. It has been used in primary tumor cell culture, stem cell culture, organoid culture, organ transplantation, cell transplantation, embryo transfer, in vitro tissue engineering, and tumor research. For example, cells can be cultured in cooled Mebiol® Gel, and cells can be collected by centrifugation after cooling. In the gel state, Mebiol® Gel's high lipophilicity is beneficial for cell proliferation, cell signaling, gas exchange, and the resistance of cells and tissues to external forces.

Chitosan/glycerol phosphate sodium (GP) composites can form in vivo safe temperature-sensitive hydrogels. If compounded with other materials, the mechanical properties, biodegradability, and functionality of the gel system can be improved[18]. For example, by compounding

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dextran-polylactic acid copolymer with chitosan/GP, a composite hydrogel with significantly enhanced mechanical properties and significantly improved in vivo biodegradability can be prepared. In addition, to make the gel have osteoinductivity, osteoconductivity, and in situ mineralization ability, silk and hydroxyapatite can be compounded with chitosan/GP to prepare a new type of injectable gel. These composite gels can be injected and gelled under physiological temperature and pH conditions, and in vitro and in vivo experiments have confirmed their good safety and can effectively repair bone defects.

3. Self-Healing Hydrogels

3.1. Principle of Self-Healing Hydrogels

With the rapid development of biomedical and material science fields, people's requirements for the performance of hydrogel materials are increasingly high. Traditional hydrogel materials often break or tear during use, which is not conducive to long-term use. Therefore, scientists have begun to explore hydrogel materials that can automatically repair damaged parts, that is, self-healing hydrogels, by integrating reversible covalent bonds and non-covalent bonds in the hydrogel structure to enhance their self-healing ability[19]. These relatively weak chemical bonds often act as sacrificial bonds and break preferentially under stress to disperse energy.

Based on different types of chemical bonds, self-healing hydrogels can be further divided into two categories: covalent cross-linked self-healing hydrogels and non-covalent cross-linked self-healing hydrogels. Covalent cross-linked self-healing hydrogels rely on reversible chemical reactions or the reformation of dynamic covalent bonds to achieve self-healing, such as disulfide bonds, imine bonds (also known as Schiff bases), reversible bonds in the products of Diels-Alder addition reactions, and bonds formed by reversible free radical reactions[20].

Non-covalent cross-linked self-healing hydrogels rely on a series of non-covalent interactions, such as the formation and breaking of hydrogen bonds, coordination actions of metal ions with ligands, extensive intermolecular forces, and associations and dissociations caused by hydrophobic interactions. Although these non-covalent bonds are relatively weak, their reversibility and rapid response enable non-covalent cross-linked hydrogels to also have good self-healing ability, which can quickly restore their structural integrity without additional energy.

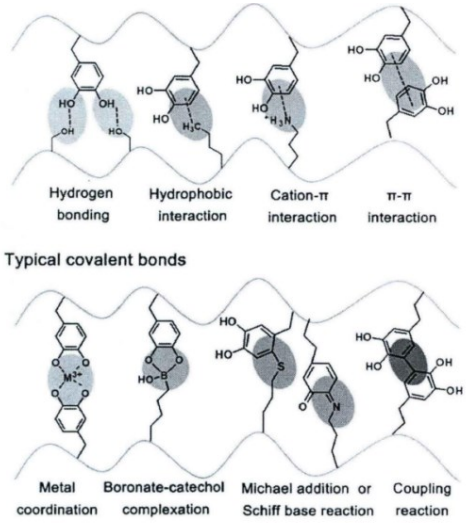


Figure 3: Typical non-covalent and covalent bonds in the cross-linking mechanism of hydrogels.

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3.2. Classification of Self-Healing Hydrogels

Self-healing hydrogels can be divided into two categories based on their cross-linking methods: self-healing hydrogels based on physical cross-linking and self-healing hydrogels based on chemical cross-linking. The former is cross-linked through non-covalent bonds, such as hydrogen bonds, hydrophobic interactions, and host-guest interactions, and can self-heal under external stimuli (such as temperature, pH value, light, etc.). For example, the boron ester bond formed based on polyvinyl alcohol (PVA) and phenylboronic acid (PBA) can self-heal, enhance the solubility of extremely poorly soluble drugs, and increase drug release time[21]. The latter is cross-linked through dynamic covalent bonds, such as acetal bonds, imine bonds, disulfide bonds, etc., which break and reform under specific conditions to achieve self-healing. For example, chitosan is a natural polymer material that can be used as the matrix of an antibacterial self-healing hydrogel, with imine bonds as the main cross-linking structure, producing self-healing behavior under different pH conditions.

3.3. Synthesis of Self-Healing

3.3.1. Schiff Base Reaction Method

The Schiff base reaction method is a chemical reaction-based method, such as forming Schiff base connections through the reaction of aldehydes with amines, thus preparing self-healing hydrogels in an aqueous phase. For example, ε-polylysine (ε-PL) and polyacrylic acid (PAA), polyethyleneimine (BPEI) are synthesized into self-healing hydrogels in an aqueous phase through the Schiff base reaction. It has good biocompatibility and multifunctionality and has a broad application prospect in the biomedical field[22].

3.3.2. Synthesis of Self-Healing Hydrogels Based on Physical Cross-Linking

Physically cross-linked self-healing hydrogels can self-heal after damage by re-forming non-covalent bonds such as hydrogen bonds, hydrophobic interactions, and host-guest interactions. For example, due to the strong hydrogen bonding of PEDOT:PSS, the conductive self-healing hydrogel composed of polyglutamic acid (PGA) and PEDOT:PSS has good self-healing properties[23].

3.4. Physicochemical Properties of Self-Healing Hydrogels

3.4.1. Self-Healing Ability

Self-healing hydrogels rely on reversible physical or chemical interactions to autonomously repair their structure after damage. For example, imine bonds obtained through Schiff base reactions are commonly used dynamic covalent bonds in the preparation of self-healing hydrogels. Gelatin reacts with ethylenediamine to increase amino content, and then periodate carboxymethyl cellulose is used to cross-link amino gelatin, thereby increasing the self-healing ability of self-healing hydrogels[24].

3.4.2. Biocompatibility

The biocompatibility of self-healing hydrogels refers to their ability to come into contact with biological tissues without causing adverse reactions, which is the basis for their application in drug delivery, tissue engineering, and wound healing. Natural polymer materials themselves have good biocompatibility and can be used as the matrix for self-healing hydrogels. For example, chitosan is a natural polymer material made from animal chitin, which has biocompatibility and natural antibacterial properties, and is often used as the matrix for the synthesis of antibacterial self-healing hydrogels.

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3.5. Applications of Self-Healing Hydrogels

3.5.1. Drug Delivery Carriers

When hydrogels are damaged during use, they can quickly repair through chemical reactions or physical interactions, ensuring the integrity of the hydrogel structure and the effectiveness and release performance of the drug. For example, self-healing hydrogels composed of pectin aldehyde and NIPAM are used as anti-tumor drug delivery carriers[25]. Dynamic acetal bonds provide good self-healing properties for hydrogels, making them stable in drug delivery.

3.5.2. Supercapacitors

In the development of flexible supercapacitors, achieving a combination of flexibility and high electrochemical performance is a technical challenge, and self-healing hydrogels provide a breakthrough for this challenge. By introducing poly(3,4-ethylenedioxythiophene) (PEDOT) and poly(4-styrene sulfonic acid) (PSS) into alginate hydrogels, composite hydrogels with high ionic conductivity, excellent flexibility, and self-healing characteristics can be prepared[26]. In situ synthesis of alginate in the presence of dimethyl sulfoxide (DMSO), followed by treatment with sulfuric acid, can further improve the conductivity of the hydrogel. The supercapacitor based on alginate/PEDOT:PSS achieved a specific capacitance of 312 F/g at a scan rate of 80 mV/s. And after two cut hydrogel samples were reconnected for 90 minutes, the hydrogel successfully achieved self-healing.

3.5.3. Oil-Water Separation

The discharge of a large amount of oily wastewater poses a serious threat to human health and the ecosystem. An effective strategy for dealing with these oily wastewaters is membrane separation technology. The adoption of light-induced self-cleaning technology to manufacture oil-water separation membranes has become an effective way to develop materials with self-cleaning characteristics[27]. Silver/silver chloride nanoparticles (Ag/AgCl NPs) are noted for their excellent visible light catalytic activity, simple preparation process, excellent stability, and significant antibacterial properties. A cellulose-based hydrogel (AHCH)[28] enhanced with Ag/AgCl NPs, through immersion coating and in situ gelation processes, can be applied to cotton fabric (AHCH@CF), thus obtaining an efficient "dehydration" membrane material for oil-water separation, as shown in Figure 4.

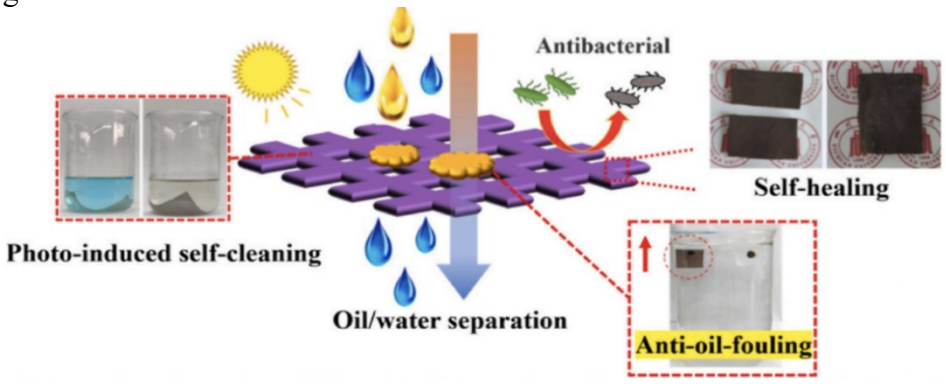


Figure 4: Oil/water separation action of AHCH@CF.

AHCH@CF shows super hydrophilicity and underwater super hydrophobicity, with good anti-oil contamination ability. It shows an excellent separation efficiency of up to 99.5% for various oil-water

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mixtures, and even after 50 cycles of use, the separation efficiency remains above 99.0%. The introduction of Ag/AgCl NPs not only enhances the mechanical properties of the hydrogel but also endows it with photo-induced self-cleaning ability and antibacterial activity, while retaining the original self-healing and anti-swelling characteristics, making AHCH@CF excellent in recyclability and reusability. This self-repair mechanism reduces the need for frequent membrane replacement, reduces membrane maintenance costs, and helps improve the sustainability of the oil-water separation process, providing an economical and environmentally friendly solution for the development of oil-water separation technology.

3.5.4. Food Packaging

Food packaging plays a key role in extending the shelf life of food and ensuring food safety. However, packaging may be damaged due to mechanical stress, temperature changes, or handling during transportation and storage. For example, when ultraviolet light penetrates the packaging film, the packaged food may experience rapid quality loss or spoilage[29], so the self-healing characteristics of packaging materials are crucial.

Carboxymethyl cellulose sodium (CMC)/polyvinyl alcohol (PVA)/polyethyleneimine (PEI)/tannic acid (TA) hydrogel film[30] has a three-dimensional network structure, and the dynamic reversible non-covalent bonds act as sacrificial bonds to dissipate load energy, endowing the hydrogel film with an excellent elongation of 400%, far greater than the elongation of conventional food packaging films (<50%). In addition, PEI molecules can enhance intermolecular interactions through hydrogen bonding, and the incorporation of bioactive TA endows the hydrogel film with good antioxidant and antibacterial properties, further overcoming the limitations of conventional food packaging films with poor stretch strain and relatively simple functions.

4. Conclusion

Temperature-sensitive hydrogels, with their unique temperature response characteristics, show broad application potential in wearable devices and biomedical engineering fields. They can adjust their properties according to temperature changes and respond sensitively to environmental changes. Self-healing hydrogels, with their self-repair ability, have great application prospects in drug delivery and environmental treatment. They can self-repair after being damaged, restoring their original structure and function, extending the service life of the material, and improving economic benefits.

In addition, hydrogels, as a functional biomaterial, have been widely used in contact lenses, sanitary products, and wound dressings, but their potential in drug delivery and tissue engineering has not been fully exploited.

Although many hydrogel drug delivery devices and scaffolds have been designed, researched, and patented, the actual commercialized products are still limited, mainly due to high production costs. Although the early biomedical applications of hydrogels were challenged by the toxicity of cross-linking agents and the limitations of hydrogel formation under physiological conditions, the continuous development of polymer chemistry and in-depth research on biological processes have made the design of multifunctional materials and minimally invasive treatments possible. In the future, with the improvement of hydrogel synthesis processes, it will show a broader development space in the fields of precision drug delivery, tissue engineering, and intelligent medical devices.

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