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# Hydrophobic Initiator Activates the Interface Reaction to Trigger Polymerization Graft Polytetrafluoroethylene Catheter

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## ABSTRACT

Decreasing the surface friction coefficient of medical catheters is important especially in interventional treatment, but the current solution involves high cost or cumbersome manufacturing processes. In order to effectively solve the problem of high friction coefficient of the inner and outer layers in the use of medical catheters and simplify the manufacturing process, we use polytetrafluoroethylene (PTFE) as a catheter material to graft directly with PAAm hydrogel. In order to overcome the high chemical inertness and low surface energy of PTFE, nitrogen ion injection is used to modify the surface of PTFE. The resulting porous PTFE surface is loaded with initiators and the hydrogel monomers polymerize in situ to form a lubricating layer. The hydrogel coating is strongly bonding on PTFE substrate, the maximum 180° peel strength between PAAm hydrogel and PTFE substrate reaches at 222 N/m. The hydrogel lubricating layer provide a friction coefficient of 0.04. In addition, in comparison to silicone rubber, the surface biofilm adhesion is diminished by 350%, demonstrating exceptional anti-fouling characteristics. This study provides a new idea and method for the surface low friction design of implantable medical devices.

## KEYWORDS

Friction coefficient; Medical catheters; Polytetrafluoroethylene; PAAm hydrogel

## 1. INTRODUCTION

Medical catheters are critical instruments in minimally invasive interventional medicine, facilitating precise access to lesion sites through the use of a guide wire. They act as conduits for the delivery of medical devices and pharmaceuticals. During minimally invasive procedures, catheters experience two primary types of frictional movement [1]. The first occurs during catheter insertion, when friction can develop between the outer surface of the catheter and the epithelial cells lining the human body cavity. Excessive friction during this phase can cause damage to the lumen, potentially leading to complications such as bleeding and inflammation [2~3]. The second form of friction is caused by the interaction between the medical device being introduced and the inner surface of the catheter. Increased friction in this scenario can make it difficult to navigate these devices through complex and tortuous anatomical pathways [4].

Polytetrafluoroethylene (PTFE) is acknowledged for possessing one of the lowest friction coefficients among solid materials, which is advantageous in medical catheter applications as it effectively reduces friction between the catheter's inner wall and the transport device. However, its inherent rigidity presents challenges in maneuvering through curved anatomical structures, such as blood vessels, potentially resulting in increased patient discomfort during medical procedures. As a result, many commercially available medical catheters use PTFE as the inner layer and materials with flexible properties such as polyurethane (PU), polyvinyl chloride (PVC) and silicone rubber as the

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outer layer. This design makes the medical catheter softer, but the outer material has a higher coefficient of friction, making it potentially dangerous during intubation. Hydrogel coatings are characterized by their notable softness and biocompatibility, which protect cells and tissues within the human aqueous environment. As a result, hydrogel composite catheter materials have become the preferred choice for most implantable medical devices, as they significantly diminish friction between the catheter and the human body during intubation [5]. Numerous studies have reported methods of grafting a water lubrication layer to the outer layer of medical catheters. For instance, researchers have successfully applied a poly (AAm-MPC-ZMA) hydrogel to silicone rubber catheters, utilizing a polyvinyl acetate (PVAc) primer layer. This innovative approach reduced the contact angle from 107.01° to 23.22°, resulting in a highly hydrophilic silicone rubber catheter [6]. Xu et al, selected vinyl pyrrolidone (NVP) and quaternary ammonium dimethylaminoethyl methyl ester (QDMAEMA) as functionalized monomers, and grafted on the surface of hydrophobic medical PVC catheters. The surface water contact angle of the material can be reduced to about 20°, and the sterilization rate of PVC material in static and circulating flow states can reach up to 99%, thus constructing a medical PVC catheter material with successful expression of hydrophilic/sterilization dual properties [7]; Yan et al. used the principle of interface penetration to graft gel coating on the surface of SEBS material to achieve surface resistance reduction lubrication, and the friction coefficient as low as 0.12±0.04, and enhanced the mechanical properties and anti-protein adhesion properties of SEBS materials, and realized the surface modification of SEBS material [8]. The above study solved the problem of friction on the surface of medical catheters, but the design of an inner PTFE coating with a material such as silicone in the middle and a modified hydrogel lubricating layer on the outside made the manufacturing process too cumbersome.

In order to effectively solve the problem of high coefficient of friction caused by the relative sliding of the inner and outer layers encountered during the use of medical catheters, and to simplify the manufacturing process, the aim of this work is to apply a hydrogel coating directly on the surface of PTFE tube. Herein, in this work, nitrogen ion implantation is used to modify PTFE. T The resulting porous PTFE surface is loaded with initiators and the hydrogel monomers polymerize in situ to form a lubricating layer. The hydrogel coating is strongly bonding on PTFE substrate, while ensuring the coating lubrication effect, the adhesion performance of the composite material is also ensured. In comparison to silicone rubber, the surface biofilm adhesion is significantly reduced, demonstrating exceptional anti-fouling characteristics.

## 2. PREPARATION OF HYDROGEL AND PTFE COMPOSITE MATERIALS

### 2.1. Materials

Acrylamide (AAm), N, N'-methylenebisacrylamide (MBAA), sodium dodecyl sulfate (SDS), α-ketoglutaric acid (α-KA), and azobisisobutyronitrile (AIBN) were sourced from Shanghai Titan Technology Co., Ltd., based in Shanghai, China. Isopropanol, ethanol, and dibromomethane were obtained from China National Pharmaceutical Group Chemical Reagents Co., Ltd., also situated in Shanghai, China. All reagents utilized in this study are of analytical grade purity, ensuring their suitability for high-quality research applications.

### 2.2. Preparation of Polyacrylamide Hydrogel

To prepare a uniform hydrogel prepolymer solution, combine 2.5 mol/L acrylamide (AAm), 0.5 mol% N, N'-methylenebisacrylamide (MBAA), 0.006 mol/L sodium dodecyl sulfate (SDS), and 0.1 mol% α-ketoglutaric acid (α-KA), which serve as the monomer, crosslinking agent, surfactant, and photoinitiator, respectively. Utilize deionized water as the solvent and employ an ultrasonic cleaner to achieve complete dissolution and ensure uniform mixing of the components.

### 2.3. Preparation of PTFE Composite Materials

The hydrogel prepolymer is subjected to a continuous nitrogen purge for a duration of 15 minutes to effectively remove dissolved oxygen, all while being protected from light exposure. Subsequently, the prepolymer is placed into a glass plate reaction mold with a spacing of 2 mm, which contains AIBN@PTFE-N. The hydrogel undergoes curing under UV light at a wavelength of 365 nm, resulting in a hydrophilic functionalized PTFE composite material. The preparation process is illustrated in Figure 1.

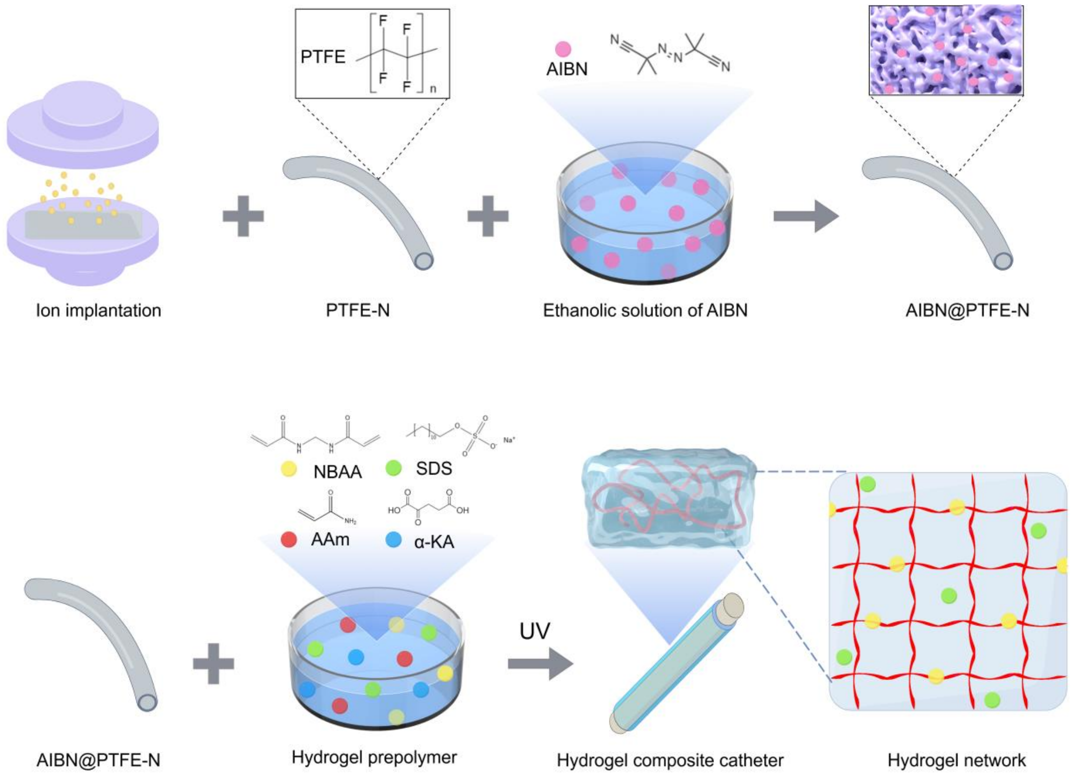

**Figure 1.** Schematic of coating hydrogel lubrication layer on PTFE tube

The modification of the PTFE surface through ion injection induces chain scission, thereby enhancing surface activity and increasing surface free energy [9]. When polymer materials undergo plasma exposure, free radicals are generated within the polymer chains. These free radicals initiate polymerization reactions upon interaction with monomers in the liquid phase, resulting in the formation of graft copolymers on the surface [10]. Subsequently, a hydrophobic initiator, such as AIBN, is adsorbed onto the substrate surface via solvent-assisted diffusion, serving as a critical primer for covalent bonding with the subsequently formed hydrogel. Utilizing an aqueous solution, acrylamide (AAm), a crosslinking agent (MBAA), and a hydrophilic initiator (α-KA) permeate the micelle layer. Polymerization is triggered by ultraviolet light, and the embedded hydrophobic initiator facilitates interfacial reactions that secure the hydrogel to the substrate [11], resulting in a hydrophilic surface. Uncrosslinked materials can be readily removed with deionized water, yielding a hydrogel coating. Macroscopic observations confirm that the PAAm hydrogel coating is effectively integrated onto the PTFE surface.

## 3. CHARACTERIZATION AND PERFORMANCE EVALUATION

### 3.1. Scanning Electron Microscope (SEM) Testing

The experiment employed the VEGA 3SBU scanning electron microscope (SEM) manufactured by TESCAN, situated in the Czech Republic, to investigate the micromorphology of the prepared samples. The PTFE materials, both pre- and post-modification, were securely mounted on the sample stage using conductive adhesive and underwent a gold sputtering treatment in an ion sputtering apparatus. The SEM operated at a working voltage of 15 kV, facilitating the observation of the sample surface and the acquisition of micromorphology images at magnifications of 1,000 and 5,000. The hydrogel samples were processed via a freeze-drying method, and their cross-sections also underwent a fixed gold sputtering treatment. Observations and micromorphology image captures were performed at magnifications of 200 and 500.

### 3.2. Surface Tension Test

The evaluation of the surface free energy of PTFE samples was conducted utilizing the C602 optical contact angle meter from Cono, USA. The surface tension of the prepolymer was measured using the YS-ZL1020 tension measuring instrument. After calibration with pure water, the concentrations of monomers and surfactants in the prepolymer were meticulously adjusted to establish a concentration-surface tension curve.

### 3.3. Water Contact Angle (WCA) Test

Utilizing the C602 contact angle meter, a series of tests were performed to quantify the contact angle of the gel pre-solution. The contact angles of the gel solution on both modified and unmodified polytetrafluoroethylene (PTFE) membranes were evaluated at ambient temperature. The stop-drop method was implemented, commencing with an initial pause. A micropipette was employed to vertically dispense 2.0 μL of gel solution onto the PTFE surface for each trial, with photographs taken after allowing the water droplet on the membrane surface to reach equilibrium. The data were analyzed through the circle fitting method to ascertain the contact angle values. The average contact angle was determined by conducting tests at three distinct positions for each sample.

### 3.4. Peel Strength Test

Utilize an electronic universal testing machine to precisely cut the hydrogel/PTFE composite material into rectangles with dimensions of 25 mm in width and 70 mm in length. Prior to conducting the tests, apply a layer of polyethylene film to the reverse side of the gel to act as a constraint for the hydrogel layer, thereby mitigating the risk of deformation during the peeling test, which could potentially compromise the integrity of the results. Evaluate the peeling strength of the gel coating from the PTFE material by performing a 180° peeling test at a controlled speed of $30\ \mathrm{mm \cdot min^{-1}}$. Each sample should be subjected to three tests to facilitate the calculation of an average value.

### 3.5. Friction Coefficient Testing

The friction coefficient of the hydrogel was assessed utilizing a rotational rheometer. Prior to the evaluation, the swollen equilibrium gel samples were meticulously cut to conform to the rotor's specifications, ensuring a consistent thickness of 1 mm and a radius of 25 mm. The sample stage temperature of the rheometer was regulated at 37℃ to simulate the physiological conditions of the human body, with deionized water utilized as a lubricant. To evaluate the frictional performance of PAAm hydrogel, friction tests were conducted at a sliding speed of 5 mm/s under a load of 1 N.

### 3.6. Anti-pollution Performance Testing

The outlined methodology entails the evaluation of antifouling performance of hydrogels utilizing crystal violet staining. Initially, the hydrogels are swollen, cut, and fixed. A peristaltic pump is employed to facilitate the continuous circulation of a Pseudomonas aeruginosa culture over the hydrogel surface for a period of three days. Subsequently, the hydrogel surface is subjected to staining with a 5 g/L gentian violet solution for 30 minutes. The samples are then immersed in deionized water for one hour. Finally, 2 mL of the solution is extracted for absorbance measurement at 590 nm using a UH4150 spectrophotometer.

## 4. RESULTS AND DISCUSSION

Due to PTFE's low surface energy (~17 mN/m, as detailed in Table 1) and high chemical inertness, both chemical modification and surface grafting pose considerable challenges. Thus, a three-step strategy including N-ion implantation surface modification and surface tension adjustment was used to establish a hydrogel lubricating layer on PTFE, as depicted in Figure 1 [12]. Firstly, PTFE was surface modified through nitrogen ion implantation (denoted as PTFE-N) [13]. After that, the smooth (Figure 2a) surface of PTFE was bombarded by energetic ions, creating numerous micron-sized pores (Figure 2b). This increased the specific surface area and raised the surface energy to 46.35 mN/m (Table 1) [14]. Then, the hydrophobic initiator AIBN was introduced to PTFE-N by immersion in an ethanol solution, which allowed effective wetting of the porous surface. After drying, the surface energy decreases to 38.77 mN/m (as indicated in Table 1), but it remains significantly higher than prior to modification. ATR/FTIR characterization of the PTFE material before and after the loading of AIBN reveals that the infrared spectrum of AIBN@PTFE-N exhibits characteristic peaks corresponding to AIBN functional groups at approximately 3000 cm-1 (C-H stretching vibration), 2240 cm-1 (C≡N), and 1500 cm-1 (N=N stretching vibration), thereby confirming the successful loading of the hydrophobic initiator onto the surface. At last, the pre-gel solution was poured onto the PTFE surface, and forming a hydrogel coating via an in-situ free radical polymerization [15]. The key to this step is to reduce the surface tension of the pre-gel solution by incorporating the surfactant SDS. When the molar concentration of the monomer exceeds 1.5 mol/L, increasing the SDS content from 0 mmol/L to 6 mmol/L results in a significant reduction in the surface tension of the pre-gel solution from 48.1 mN/m to 34.08 mN/m (Figure 2d), which is lower than the surface free energy of AIBN@PTFE-N (38.77 mN/m). The experimental results show that the surface tension of the pre-gel decreases sharply with increasing SDS concentration from 0 to 6 mmol/L. The SDS-containing pre-gel solution reaches its critical micelle concentration at 6 mmol/L of CSDS, thus the surface tension shows a slightly increasing tendency [16].

As the decreasing of surface tension of the pre-gel solution, it is conducive to its spreading on PTFE substrate, and realizing the in-situ polymerization and bonding on the PTFE surface. To improve this point, further investigations were carried out on the relation on the contact angle of pre-gel solution with varying concentrations of sodium dodecyl sulfate (SDS) and acrylamide (AAm) on the surface of AIBN@PTFE-N (Figure 2e, f). The observed trend in contact angle variations corresponds to the changes in the surface tension (Figure 2d) of the pre-gel solution. Data presented in Table 2 indicates that the contact angle of the pre-gel solution containing 6 mmol/L SDS on AIBN@PTFE-N is significantly lower than that of 0, which decreases from 107° to 67° [15]. Compared with the original PTFE, AIBN@PTFE-N has the significantly lower contact angle to the pre-gel solution. In addition, the contact angle diminishes as the AAm content increases; for example, when the AAm concentration escalates from 1.5 mol/L to 3 mol/L, the contact angle decreases from 77° to 61°, indicating an improvement spreading of the pre-gel solution on AIBN@PTFE-N substrate. These results indicate that the synergistic effects of SDS and AAm considerably enhance the spreading and wettability of the pre-gel solution on the AIBN@PTFE-N surface [17].

Bonding of the PAAm hydrogel to PTFE was achieved by optimizing the formulation of the pre-gel solution with CAAm of 2.5 mol/L and CSDS of 6 mmol/L. The optical microscope image (Figure 2g) visually shows a uniform and seamless bonding interfaces between the PAAm hydrogel and the PTFE substrate. Scanning electron microscopy (SEM) observations of the vacuum freeze-dried hydrogel/PTFE bonding interface show that the hydrogel, characterized by a fluffy network structure, adheres uniformly adhered to the PTFE surface, with no delamination at the bonding interface (Figure 2h). It is also demonstrated on a macroscopic scale that the hydrogel achieved a strong bond on both flat (Figure 2i-I) and tubular (Figure 2i-II) PTFE surfaces (the hydrogel on the PTFE tubes is colored green for better visualisation) [18~19].

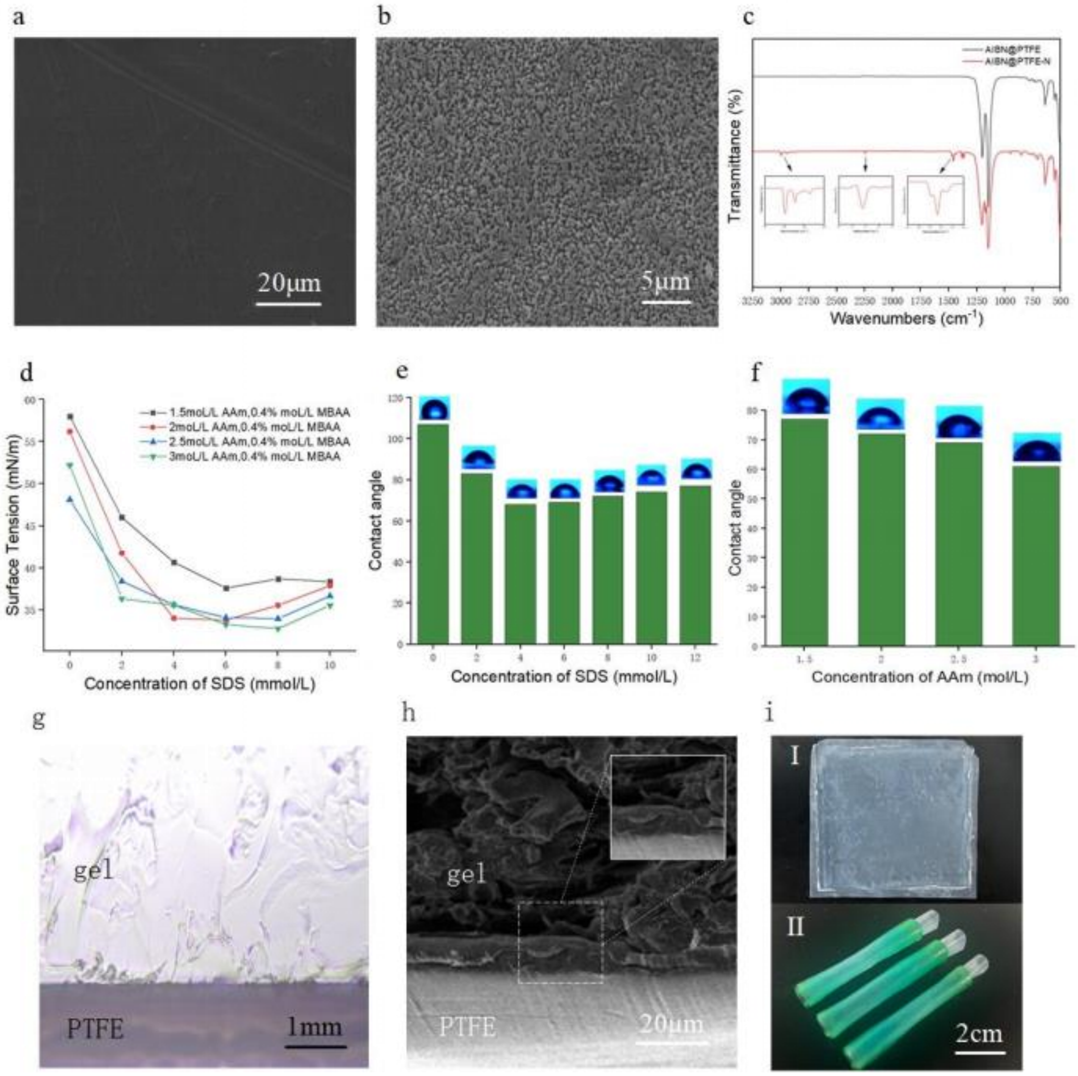

**Figure 2.** a) Surface morphology SEM image of original PTFE; b) SEM image of the modified sample after N-ion implantation (abbreviated as PTFE-N); c) infrared spectrum of PTFE-N loaded with AIBN (AIBN@PTFE-N) before and after modification; d) surface tension of pre-gel solution; e) contact angle of pre-gel solution with different SDS concentrations on AIBN@PTFE-N surface; f) contact angle of pre-gel solution with different AAm concentrations on AIBN@PTFE-N surface; g) microscopic image of gel-PTFE bonding interface; h) SEM image of gel-PTFE bonding interface; i) physical appearance of gel-PTFEl composite (I- hydrogel coating on planar PTFE; II- green colored hydrogel coating on PTFE tube).

**Table 1.** Surface free energy of PTFE before and after N-ion implantation

| | PTFE | PTFE-N | AIBN@PTFE-N |
|---|---|---|---|
| Surface free energy (mN/m) | 17.31±1.8 | 46.35±35 | 38.77±2 |

**Table 2.** Contact angles of pre-gel solutions on different PTFE surfaces

| SDS concentration (mmol/L) | PTFE | AIBN@PTFE | PTFE-N | AIBN@PTFE-N |
|---|---|---|---|---|
| 0 | 109°± 4.5° | 101°± 4° | 99°± 3.5° | 107°± 4° |
| 6 | 90°± 3° | 87°± 3° | 67°± 2.5° | 69°± 3° |

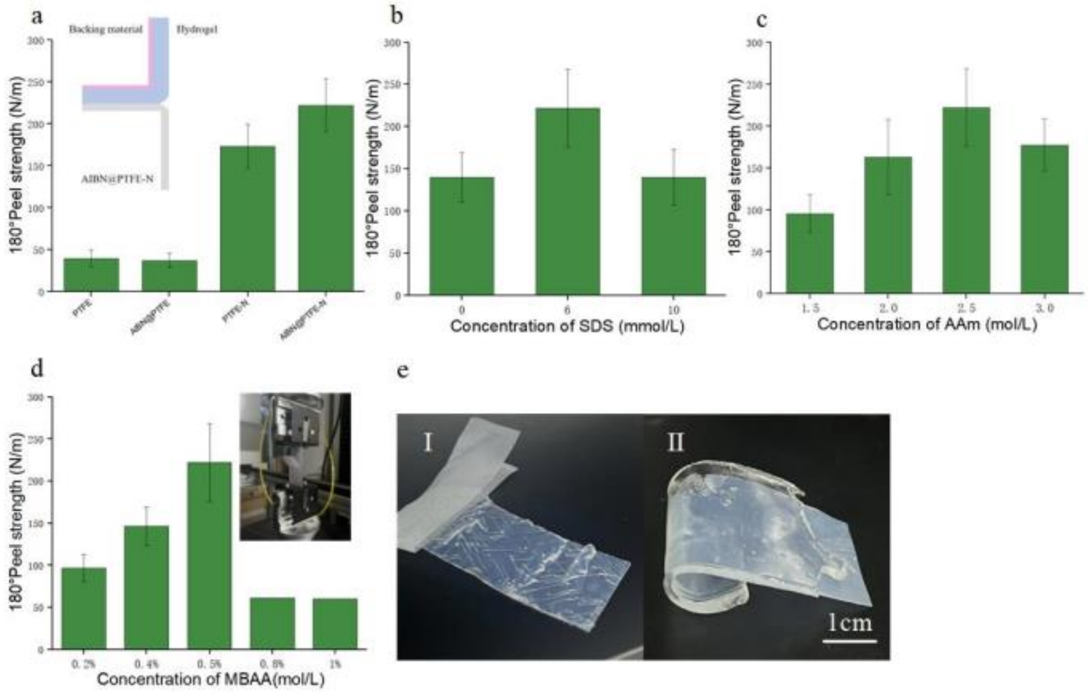

**Figure 3.** Optimization of bonding between PAAm hydrogel and PTFE. a) 180°peeling strength of PAAm hydrogel to different substrate materials including original PTFE, AIBN@PTFE, PTFE-N and AIBN@PTFE-N; b) 180°peeling strength of hydrogel prepared with varying SDS concentrations to AIBN@PTFE-N; c) 180°peeling strength of hydrogel prepared with varying AAm concentrations; d) 180°peeling strength of hydrogel prepared with varying MBAA concentrations; e) photographs of specimens after peeling tests (I - hydrogel residues on PTFE; II - brittle fracture of hydrogel on PTFE).

The 180° peel test was utilized to evaluate the adhesion performance between hydrogel and PTFE substrate. To mitigate excessive elongation of the hydrogel samples in the peeling direction during the process, a soft yet less extensible backing layer (PE) was employed to study the peak peel force (as shown in the embedded schematic in Figure 3a). The results from the peel strength tests for PAAm hydrogel on various substrates, including PTFE, AIBN@PTFE, PTFE-N, and AIBN@PTFE-N, are presented in Figure 3a. The peel strength of the hydrogel on the unmodified PTFE surface was the lowest, recorded at 39±10 N/m and 37±10 N/m for the original PTFE surface and the AIBN@PTFE, respectively. Following ion implantation modification of PTFE, the peel strength significantly increased to 173 N/m. When both ion implantation modification and hydrophobic initiator loading were applied, the peel strength further increases to 222 N/m. This enhancement is attributed to the hydrophobic initiator's capacity to induce AAm polymerization at the PTFE interface [20~21], while ion implantation modification elevates the surface energy of PTFE. Both techniques facilitate the accumulation of PAAm polymer chains from the hydrogel on the PTFE surface, thereby strengthening the physical interactions between the hydrogel and PTFE [22], which in turn increases the peel strength. The peel strengths of PAAm hydrogel with different concentrations of SDS, and AAm on AIBN@PTFE-N are shown in Figures 3b, c. The results showed that the trend of peel strength was inversely correlated with the surface tension and contact angle of the pre-gel solution (Figures 2d, e, f). A lower surface tension and contact angle on the AIBN@PTFE-N substrate facilitates the spreading behavior of the pre-gel solution on the substrate and consequently strengthens the bonding [23].

In addition, the crosslinking concentration (CMBAA) of PAAm hydrogel on 180° peeling strength has also been studied (Figure 3d). The peel strength increases with CMBAA gradually, and reaching a maximum value of 222 N/m at CMBAA of 0.5 mol%. But then decreases with the continues increasing of CMBAA. When CMBAA< 0.5 mol%, the intrinsic strength of the hydrogel is weaker than the bonding strength of the adhesive interface [23], the 180° peel strength depends on the strength of the hydrogel. This observation is confirmed by the image in Figure e-I, which shows that the low CMBAA hydrogel has a residue on the PTFE surface after the peel test [24]. When CMBAA exceeds 0.5 mol%, the high crosslink density results in a hydrogel network that is susceptible to brittle fracture during deformation, resulting in reduced peel strength. As shown in Figure e-II, the hydrogel coating breaks and leading to the failure of peeling test. Thus, the CMBAA should be kept below 0.5 mol% in the preparation of hydrogel coatings.

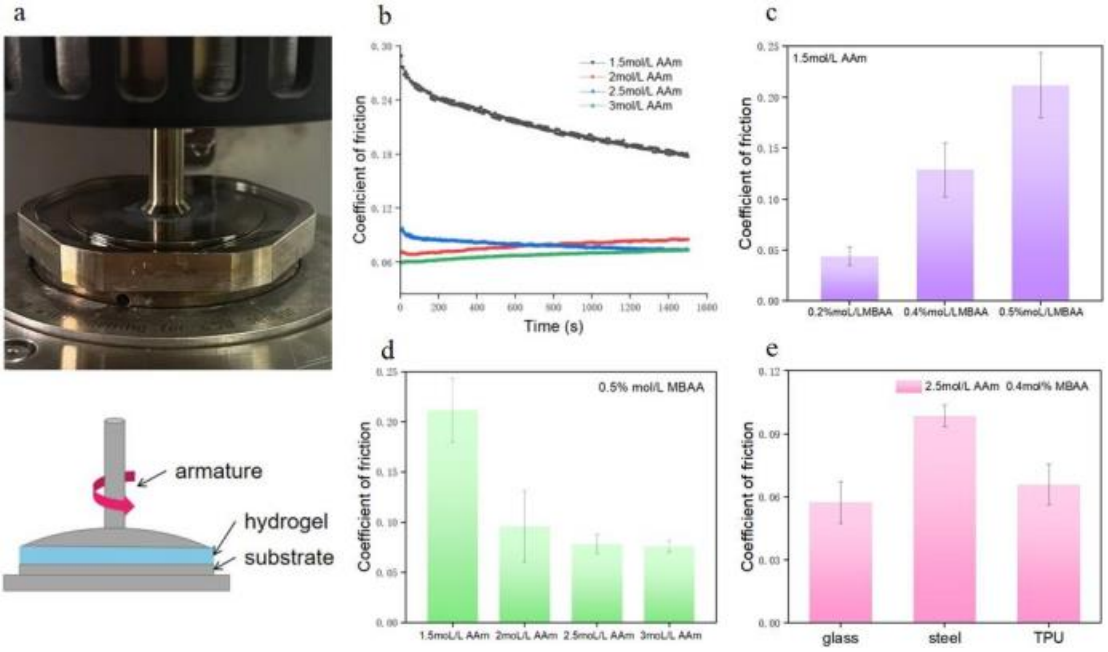

**Figure 4.** Surface lubrication of hydrogel coating. a) Schematic diagram of friction test; b) Line graph of friction coefficient varying with AAm concentration; c) Trend graph of friction coefficient varying with MBAA concentration; d) Trend graph of friction coefficient varying with AAm concentration; e) Friction coefficients between hydrogel and glass, steel, TPU.

The friction performance of a hydrogel lubrication layer grafted onto a PTFE surface was assessed using a rotational rheometer under a normal force of 1 N, the friction coefficient test is shown in Figure 4a. This investigation specifically examined the impact of CMBAA and CAAm on the coefficient of friction (CoF). The results showed in Figure 4c indicate that the increasing in CMBAA leading to the increasing of CoF between the hydrogel and glass, and the minimum CoF is ~0.04 when CMBAA is 0.2 mol%. With the increasing of CAAm, the CoF decreasing significantly from 0.21 to 0.08 (Figure 4d). The influence of CMBAA and CAAm on the lubricating performance of the hydrogel can be explained as follows: both the decrease in CMBAA and the increase in CAAm will lead to an increase in the water content of the hydrogel [25], which will favor the formation of a water lubricating layer between the hydrogel and the glass, thus reducing the CoF. In addition, the friction performance of the hydrogel lubrication layer against stainless steel and thermoplastic polyurethane (TPU) has also been studied. As shown in Figure 4e, the lowest friction coefficient between the glass and the glass is 0.056, and the friction coefficient between hydrogel and stainless steel and TPU is 0.097 and 0.065 respectively. The coating shows good lubrication and resistance reduction effect on the surface of all three materials.

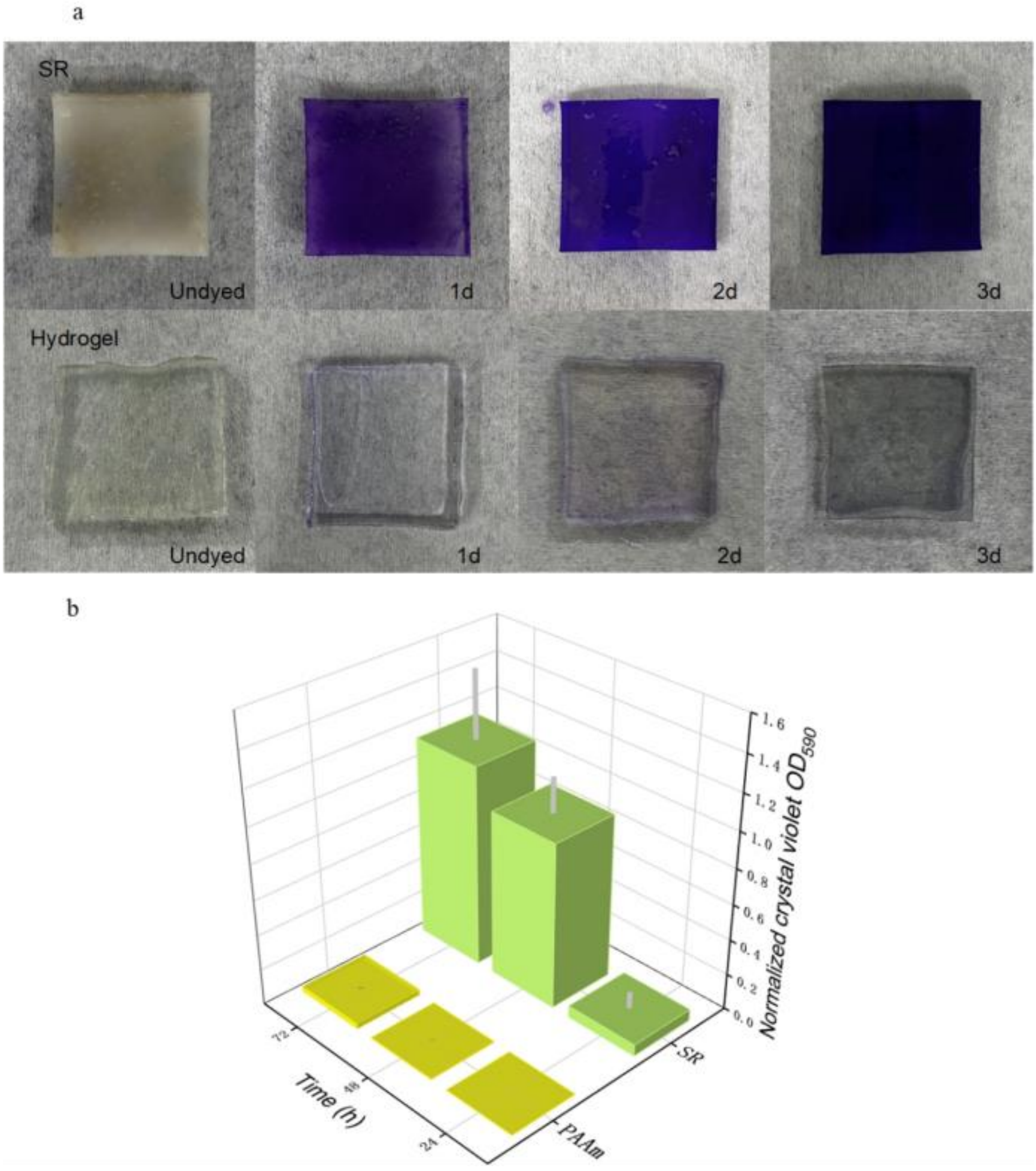

**Figure 5.** Anti-fouling tests. a) Bacterial culture staining images for 1d, 2d, 3d of SR and PAAm hydrogel; b) absorbance for PAAm hydrogel and SR during bacterial culture.

The biocompatibility of medical catheter surfaces plays a pivotal role in mitigating infection risks during surgical interventions. The crystal violet staining technique was utilized to evaluate the biocompatibility of hydrogels. In a sterile laboratory setting, a continuous flow of Pseudomonas aeruginosa bacterial solution was circulated over the hydrogel surface via a peristaltic pump at a rate of 10 mL/min to assess the biocompatibility of the hydrogel coating [26]. Silicone rubber (SR), a commonly employed outer layer material for medical catheters, served as a comparative standard.As shown in Figure 5a,f0-ollowing three days of circulation testing, both SR and PAAm hydrogel surfaces displayed a light-yellow coloration. Concurrent experiments were conducted to culture bacterial samples from both the hydrogel and control groups over 1, 2, and 3 days, facilitating the observation of biofilm attachment at various intervals [27]. The samples underwent staining with gentian violet solution, and absorbance was quantified at 590nm. The results revealed a direct correlation between absorbance and biomass attachment. Significantly,the results showed in Figure 5b, the average biofilm attachment on the PAAm hydrogel surface is reduced by 350% in comparison to SR, indicating that the PAAm hydrogel surface effectively inhibits the adhesion of Pseudomonas aeruginosa and minimizes biofilm formation. This effectiveness is attributed to the hydrophilic nature of the PAAm hydrogel, which promotes superior water absorption, thereby creating a stable hydration layer that enhances its antifouling characteristics [28].

## 5. CONCLUSION

This research investigates a surface adsorption hydrophobic initiator integrated with a polymer substrate and a hydrophilic initiator within a hydrogel pre-gel solution. An interpenetrating strategy is utilized to develop a low-friction, high-adhesion, and flexible antifouling hydrogel composite catheter material. The results indicate that the friction coefficient of the gel coating is 0.04, with a peak peel strength of 322.8 N/m and exceptional antifouling biological performance. Unlike other designs that employ PU, PVC, or silicone rubber as the inner layer of medical catheters with a composite hydrogel coating on the outer layer, this study selects PTFE, known for its remarkably low friction coefficient, as the inner layer. This selection significantly reduces the relative friction between the catheter and medical devices. Furthermore, an in-situ free radical polymerization reaction is applied to graft the hydrogel coating onto the outer layer of the modified polytetrafluoroethylene catheter. This method diminishes the rigidity of PTFE, enhancing navigation through the intricate pathways of the body and reducing the risk of damage during catheter insertion.

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