

Carbon Dot-Integrated Poly(vinyl alcohol)/Chitosan Dual-Network Hydrogel for Fe³⁺ Fluorescence Sensing

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Abstract

Fluorescent carbon dots (CDs) with carboxyl/amino-rich surfaces were synthesized via a hydrothermal method and subsequently incorporated as fluorophores into a poly(vinyl alcohol)/chitosan (PVA/CS) matrix. A PVA/CS@CDs composite hydrogel with stable blue fluorescence emission (λ_{exc} = 380 nm, λ_{em} = 435 nm) was fabricated through physical crosslinking induced by freeze-thaw cycles. Among the prepared hydrogels, PVA/CS@CDs-7 (composed of 4.5 g PVA, 1.5 g CS, and 7 mL of 0.5 mg/mL CDs) exhibited the most outstanding fluorescence–mechanical coupling performance, with a tensile stress of 0.47 MPa and a breaking strain of 681.76%—an increase of 259.31% over pristine PVA/CS—along with favorable mechanical resilience. Fluorescence spectroscopic analysis revealed that the CDs remained well dispersed within the hydrogel matrix. Notably, the fluorescent hydrogel displayed a highly selective fluorescence quenching response toward Fe^{3+} ions, with negligible interference from other common metal ions. Based on the coordination interaction between Fe^{3+} and the carboxyl/amino groups on the CDs, combined with fluorescence lifetime analysis, static quenching was identified as the dominant quenching pathway. The sensor exhibited a good linear relationship over the Fe^{3+} concentration range of 0–40 mM ($R^2 = 0.9939$), and enabled visual detection through a color gradient under 365 nm UV light. This fluorescent hydrogel, as a novel fluorescent material, offers a facile and effective approach for Fe^{3+} sensing, with promising application prospects in environmental monitoring and water quality assessment.

1. Introduction

Fe^{3+} , as an essential trace element for the human body, plays a critical role in various physiological processes, including oxygen transport, electron transfer, and enzymatic catalysis. However, dysregulation of its concentration is closely linked to the pathogenesis of multiple diseases [1–3]. The excessive discharge of Fe^{3+} into industrial wastewater and environmental water samples also poses a serious threat to ecosystems, necessitating the development of efficient and reliable methods for Fe^{3+} detection [4]. Despite their excellent accuracy and sensitivity, conventional detection methods such as atomic absorption spectrometry (AAS) and inductively coupled plasma mass spectrometry (ICP-MS) are hindered by expensive instrumentation and complex pretreatment, making it difficult to fulfill the demands for on-site rapid screening and real-time monitoring applications [5, 6].

Fluorescence sensing technology, owing to its advantages of rapid response, simple operation, and high visual readout capability, has become a research hotspot in the field of ion detection [7]. Carbon dots (CDs), as a novel class of zero-dimensional carbon-based fluorescent nanomaterials, have demonstrated considerable potential in fluorescence sensing, attributable to their excellent photoluminescent properties, good biocompatibility, and tailorable surface functional groups [8, 9]. Through surface functionalization with carboxyl, amino, and other groups, CDs can engage in selective coordination interactions with specific metal ions, thereby enabling specific fluorescence responses [10, 11]. However, traditional CDs-based solution sensors are confronted with prominent issues in practical applications,

including vulnerability to aggregation, fluorescence quenching, and poor recyclability, which severely limit their utilization as fluorescent probes [12, 13].

Immobilizing CDs into functional hydrogel matrices to construct solid-state fluorescent sensors is an effective strategy to address the aforementioned issues [14, 15]. Previous studies have demonstrated that the three-dimensional network of hydrogels can effectively suppress the aggregation-induced quenching (ACQ) of CDs through spatial confinement, thereby maintaining stable fluorescence emission [16, 17]. Among various hydrogel systems, poly(vinyl alcohol) (PVA)/chitosan (CS) composite hydrogels, as an important class of biopolymer materials, have attracted extensive attention in tissue engineering, drug delivery, and sensing due to their favorable biocompatibility, biodegradability, and abundant hydrogen-bonding crosslinking sites [18]. Specifically, PVA, as a synthetic polymer, possesses abundant hydroxyl groups along its molecular chains, which can form microcrystalline domains during the freeze-thaw process, thereby endowing the hydrogel with a physically crosslinked network. CS, as a natural polysaccharide, provides not only additional crosslinking sites via its amino and hydroxyl functionalities but also imparts pH responsiveness and metal coordination capability to the material. Through multiple physical interactions, including hydrogen bonding, electrostatic forces, and hydrophobic effects, these two macromolecules can construct a dual-network architecture with reversible crosslinking characteristics, which maintains a high water content while exhibiting favorable mechanical properties[19].

Freeze-thaw cycling, as a physical crosslinking method that eliminates the need for chemical crosslinking agents, exhibits unique advantages in the preparation of polymer hydrogels. The essence of this approach lies in the fact that the formation of ice crystals at low temperatures forces polymer chains to concentrate and align within the interstitial spaces between crystals; upon thawing, hydrogen-bonding rearrangement between chains establishes a stable three-dimensional network structure. By adjusting parameters such as the number of freeze-thaw cycles, freezing temperature, and polymer concentration, the microcrystalline domain size, crosslinking density, and porosity of the hydrogels can be effectively regulated, thereby enabling the modulation of their mechanical properties and swelling behavior[20]. Compared with chemical crosslinking, freeze-thaw physical crosslinking not only avoids the use of toxic crosslinking agents, but also endows the resulting hydrogels with favorable self-healing properties and shape memory effects, demonstrating promising application prospects in flexible sensing and smart materials[21].

To address the above challenges, this study synthesized fluorescent CDs with carboxyl- and amino-rich surfaces via a hydrothermal method, uniformly dispersed them as fluorophores into a PVA/CS acetic acid mixed solution, and subsequently constructed a PVA/CS@CDs composite hydrogel system with a dense layered structure through physical crosslinking induced by freeze-thaw cycles. This fluorescent hydrogel enables both enhanced mechanical properties and stable blue fluorescence emission, and this paper systematically investigates its morphology, mechanical properties, fluorescence characteristics, and Fe^{3+} sensing behavior. This strategy offers new insights for the development of novel fluorescent materials integrating high mechanical strength, fluorescence stability, and visual sensing capability, and

holds potential application value in the rapid screening of Fe^{3+} in industrial wastewater and environmental water samples.

2. Materials and Methods

2.1. Materials

Poly(vinyl alcohol) (PVA-124, Mw $\approx$ 85,000-124,000, 99% hydrolyzed) was procured from Xilong Chemical Industry Co., Ltd. Chitosan (CS, deacetylation degree $\geq$ 95%), melamine ($\text{C}_3\text{H}_6\text{N}_6$, $\geq$ 99%), ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, $\geq$ 99%), and N,N-dimethylformamide (DMF, anhydrous, $\geq$ 99.8%) were obtained from Shanghai Aladdin Biochemical Technology Co., Ltd. Citric acid monohydrate (CA, $\geq$ 99.5%) was purchased from Tianjin Yongda Chemical Reagent Co., Ltd. All reagents were utilized as received without further purification. Deionized water (DI water, 18.25 M Ω ·cm) prepared in-house was employed throughout all experiments.

2.2. Synthesis of Carbon Dots

Blue-emissive CDs were synthesized via a one-step hydrothermal method. Briefly, 1 mmol of citric acid, 3 mmol of melamine, and 1 mmol of urea were dissolved in 10 mL of N,N-dimethylformamide (DMF) under magnetic stirring. The homogeneous solution was subsequently transferred to a 50 mL Teflon-lined stainless steel autoclave and heated at 140°C for 3 h. After natural cooling to ambient temperature, the reaction product was centrifuged at 9000 rpm for 5 min and filtered to remove insoluble aggregates. The filtrate was then transferred to a dialysis bag (molecular weight cutoff: 1000 Da) and dialyzed against fresh DMF for 24 h to eliminate low-molecular-weight impurities. The as-prepared CDs exhibited intense blue fluorescence under 365 nm UV illumination and were stored at 4°C in the dark for subsequent use.

2.3. Fabrication of PVA/CS@CDs Composite Hydrogels

Composite hydrogels were prepared via a freeze-thaw physical cross-linking protocol. The pH of acetic acid solution was precisely adjusted to 3.0 using a calibrated pH meter. Subsequently, 4.5 g of PVA was added to 50 mL of the acidic solution and magnetically stirred at 90°C until complete dissolution. Upon cooling to 60°C, 1.5 g of CS was introduced, and the mixture was stirred at 60°C for 1 h to ensure homogenization. Aliquots of CDs suspension (0.5 mg/mL, 3, 5, 7, and 9 mL) were then added to the polymer solution under vigorous stirring to achieve uniform dispersion. Following cooling to ambient temperature, the solutions were subjected to three freeze-thaw cycles (freezing at -20°C for 12 h, followed by thawing at 25°C for 2 h). The resultant hydrogels were designated as PVA/CS@CDs-3, PVA/CS@CDs-5, PVA/CS@CDs-7, and PVA/CS@CDs-9, corresponding to the respective CDs incorporation volumes. Pristine PVA/CS hydrogels without CDs were prepared as control specimens following identical procedures.

2.4. Metal Ion Selectivity and Fe^{3+} Detection Assays

Cubic hydrogel specimens (1 cm × 1 cm × 1 cm) were immersed in FeCl₃ solutions of varying concentrations (0–40 mM) and equilibrated at ambient temperature for 5 min. Fluorescence emission spectra were acquired using a fluorescence spectrophotometer (F-4700, Hitachi) with an excitation wavelength of 380 nm. The fluorescence response was quantitatively evaluated using the F/F₀ ratio, where F denotes the fluorescence intensity after analyte exposure and F₀ represents the baseline intensity of the blank control.

2.5. Characterization

The microstructure of CDs was examined using transmission electron microscopy (TEM, JEOL JEM-F200, Japan). Raman spectra of the CDs were recorded using a Raman spectrometer (LabRAM HR Evolution, HORIBA, Japan) with an excitation wavelength of 532 nm and a scanning range of 100–2000 cm⁻¹. Hydrogel samples were lyophilized and sputtered with gold prior to scanning electron microscopy (SEM, ZEISS Sigma 360, Germany) observations. Fourier transform infrared (FT-IR) spectra were recorded to analyze the characteristic functional groups and chemical bonding over the range of 500–4000 cm⁻¹. X-ray photoelectron spectroscopy (XPS) measurements were performed on a Thermo Scientific K-Alpha system, USA using Al K α radiation to analyze the elemental composition, chemical valence states, and surface electronic structure.

To evaluate the optical properties, the excitation, emission, and three-dimensional fluorescence spectra of the CDs and composite hydrogel were measured using a fluorescence spectrophotometer (F-4700, Hitachi, Japan). UV–vis absorption spectra were acquired using a UV-3600i Plus spectrophotometer (Shimadzu, Japan) in the range of 200–800 nm. The fluorescence lifetime of the composite hydrogel was determined using a steady-state/transient fluorescence spectrometer (FLS1000, Edinburgh Instruments, UK) with a 375 nm pulsed laser as the excitation source. Fluorescence decay curves were collected at an emission wavelength of 435 nm and fitted using a biexponential function to obtain the fluorescence lifetime values.

Mechanical properties were evaluated using a universal testing machine (CMT6104, Meters Industrial Systems) according to GB/T 1040.3–2006. Dumbbell-shaped specimens were tested at a crosshead speed of 100 mm/min with an initial load of 0.01 N.

The water content (W) of the PVA/CS@CDs composite hydrogel was determined via the gravimetric method [22]. Freshly prepared PVA/CS@CDs composite hydrogels were cut into regular cuboids with dimensions of 1 cm × 1 cm × 1 cm. After gently wiping off surface moisture with filter paper, the initial mass (W₁) of each sample was accurately weighed. Subsequently, the samples were dried in a vacuum oven at 60°C for 24 h until they reached constant weight; the dried mass (W₂) was weighed immediately upon removal from the oven. Each group of samples was subjected to triplicate measurements, and the average value was calculated for statistical reliability. The calculation formula for water content is presented as Equation:

$$W = \frac{W_1 - W_2}{W_1} * 100\%$$

For the swelling ratio test, the dried samples (after completion of water content determination) were directly used as test specimens without further treatment.

3. Results and Discussion

3.1. Structural and Morphological Characterization

Figure 1 schematically illustrates the fabrication route and structural evolution of the PVA/CS@CDs composite hydrogel in this study. Surface-functionalized CDs were first synthesized via a hydrothermal method using citric acid, melamine, and urea as precursors. The as-prepared CDs were then uniformly dispersed into the PVA/CS acetic acid solution, followed by cyclic freeze-thaw cycles to induce physical crosslinking via ice crystal growth and polymer chain rearrangement, resulting in a dense layered structure. This system is synergistically reinforced through a rigid-flexible dual-network architecture: the rigid framework, formed by hydrogen-bond crosslinking between PVA microcrystalline domains and CS rigid segments, provides structural stability, while the flexible network, constituted by the flexible PVA chains and CDs-polymer interfacial interactions, endows the material with high extensibility and dynamic self-recovery capability. In addition, the carboxyl and amino groups on the CDs form hydrogen bonds and electrostatic interactions with the PVA/CS chains, effectively filling the network interstices [23].

HRTEM analysis revealed that the as-prepared CDs exhibited monodisperse, quasi-spherical morphologies with uniform size distributions and no obvious agglomeration (Fig. 2a). Lattice fringes were clearly resolved, with fast Fourier transform (FFT) and inverse FFT (IFFT) analysis indicating an interplanar spacing of 0.21 nm, consistent with the (100) lattice plane of graphitic carbon [23, 24]. These observations confirm the graphitic crystalline core structure of the CDs; the sp^2 -hybridized carbon framework endows the material with excellent electronic properties, while abundant surface defects facilitate strong interfacial interactions with the polymer matrix. Raman spectroscopy further revealed the carbon skeleton structural features of the CDs (Fig. S1). The CDs exhibited a D band at 1409 cm^{-1} and a G band at 1664 cm^{-1} . After baseline correction, the ID/IG ratio was calculated to be greater than 1, indicating that the as-prepared CDs possess abundant surface defects and disordered structures. These abundant defect sites provide sufficient active centers for the coordination recognition between the CDs and Fe^{3+} [25].

The cross-sectional morphology of the hydrogels was systematically analyzed by SEM. The pristine PVA/CS dual-network hydrogel exhibited a typical three-dimensional sponge-like porous structure (Fig. 2b), whereas the PVA/CS@CDs composite hydrogel showed a significant morphological evolution upon the introduction of CDs (Fig. 2c). In contrast to the rough and porous surface of the pristine PVA/CS hydrogel, the composite hydrogel surface became smoother and denser, with a more uniform network structure. This densification phenomenon is ascribed to the uniform embedding of CDs as zero-dimensional nanofillers into the polymer matrix, where the abundant carboxyl ($-\text{COOH}$) and amino ($-\text{NH}_2$) groups on the CDs surface form interfacial hydrogen bonds and electrostatic interactions with the PVA/CS chains, effectively filling the network voids [26]. Studies have shown that the incorporation of

nanofillers can enhance network density through the “nano-reinforcement effect”, thereby enabling more homogeneous stress distribution and preventing crack propagation induced by stress concentration, which provides a structural basis for the significant improvement in mechanical properties [27, 28].

The PVA/CS@CDs composite hydrogel exhibits excellent mechanical resilience. As shown in Fig. 2d, the sample can rapidly recover to its original shape without noticeable residual deformation after undergoing large deformations including stretching, compression, twisting, and folding. This resilience originates from the reversibility of dynamic physical crosslinks within the hydrogel network: the first network, constructed by PVA microcrystalline domains and CS rigid segments via hydrogen bonding, provides structural stability; the second network, constituted by flexible PVA chains and CDs–polymer interfacial interactions, endows the material with high extensibility [29, 30]. The surface functional groups of CDs form reversible hydrogen bonds with the polymer chains, dissipating energy through a bonding–dissociation mechanism during deformation and facilitating rapid reconstruction of the network structure [31]. This “soft–hard dual-network” synergistic mechanism enables the material to maintain high mechanical strength while exhibiting excellent resilience, laying the foundation for its application in sensing and detection [32].

Swelling kinetics and equilibrium water content (EWC) represent critical parameters for evaluating hydrogel hydrophilicity. As depicted in Figs. 2e and 2f, the swelling ratio increased monotonically with CDs loading, reaching equilibrium after approximately 10 h. This phenomenon is primarily attributed to the abundant hydrophilic functional groups ($-\text{COOH}$, $-\text{NH}_2$, $-\text{OH}$) on CDs surfaces, which significantly enhance network hydrophilicity and promote water molecule penetration via hydrogen bonding [33, 34]. Notably, according to Flory-Rehner theory, moderate swelling ratio enhancement corresponds to optimized cross-linking density. CDs incorporation improves hydrophilicity while simultaneously reinforcing network structural integrity through physical entanglement and hydrogen bonding, enabling the hydrogels to maintain structural stability even in the swollen state [35]. The swelling ratio and EWC of the PVA/CS@CDs composite hydrogel increased simultaneously with increasing CDs content, which is attributed to the contribution of hydrophilic groups on the CDs surface, while the network integrity of the hydrogel was maintained [36].

The stress–strain curves (Fig. 2g, h) show that the PVA/CS hydrogel exhibits typical soft elastic behavior. Upon CDs incorporation, both strength and extensibility were significantly enhanced: the PVA/CS@CDs-7 hydrogel achieved a tensile stress of 0.47 MPa and a breaking strain of 681.76%, representing increases of 262% and 259% over pristine PVA/CS, respectively. The mechanical properties improved with increasing CDs content from 3 to 7 mL, attributed to hydrogen bonding and electrostatic interactions between CDs and PVA/CS chains that increased the physical crosslink density. However, further increasing the CDs content to 9 mL caused a slight decline in both parameters, likely due to local aggregation that reduced stress transfer efficiency. The substantial mechanical enhancement arises from the synergistic effect of the freeze-thaw-induced dense layered network and CDs–polymer interfacial interactions, which also ensure uniform CD dispersion and fluorescence stability within the hydrogel matrix.

The effect of environmental pH on the swelling behavior of the hydrogels was further investigated (Fig. S2). The swelling ratio exhibited a marked pH dependence: under acidic conditions (pH 4), it was substantially higher than that in neutral and weakly alkaline media, with the swelling rate being most rapid within the first 2 h and gradually leveling off thereafter. This behavior is attributed to the protonation of amino groups ($-\text{NH}_2$) on chitosan chains to $-\text{NH}_3^+$ under acidic conditions, which generates strong electrostatic repulsion and promotes network expansion [37, 38]. In contrast, deprotonation under neutral and alkaline conditions weakens the repulsion, leading to network contraction and a lower swelling ratio [39, 40]. Such pH-responsive swelling confirms the critical role of chitosan in the network structure and demonstrates the controllable structural adaptability of the hydrogel, which underpins its potential for sensing applications in complex environments.

FT-IR spectroscopy revealed distinct chemical structural changes upon CDs incorporation (Fig. 3a). The PVA/CS@CDs composite hydrogel exhibited a characteristic absorption band at 1657 cm^{-1} , attributed to C = O stretching vibrations of CDs surface carboxyl groups and/or amide bonds formed between CS amino groups and CDs carboxyl functionalities [28], whereas this signal was less pronounced in pristine PVA/CS. Importantly, the peak intensity increased with CDs loading, confirming successful integration into the hydrogel network. The intense band at 1082 cm^{-1} corresponds to C–O–C and C–OH stretching vibrations in the PVA/CS backbone. This band showed minimal shift upon the introduction of CDs, indicating preservation of the hydrogen-bonded cross-link structure between PVA and CS. Furthermore, the broad band near 3310 cm^{-1} , attributed to O–H and N–H stretching vibrations, became broader and shifted to lower wavenumbers in the composite hydrogels, indicating the formation of new hydrogen bond interactions between CDs surface hydroxyl/carboxyl groups and PVA/CS chains, thereby enhancing network stability.

XPS survey spectra (Fig. 3b, c) further confirmed the successful incorporation of CDs. Since PVA itself contains no nitrogen and the contribution from CS is limited, the N 1s signal intensity is relatively low. Nevertheless, the increased atomic percentage of N (Fig. 3c) and the enhanced N 1s peak intensity (Fig. 3h) both corroborate the successful loading of CDs, which is attributed to the introduction of nitrogen-containing functional groups—such as amino and amide groups—rich on the CDs surface, derived from precursors including urea and melamine used in the synthesis.

High-resolution XPS spectra (Figs. 3d-i) revealed subtle differences in chemical bonding states. In the C 1s spectra (Figs. 3d,g), the peak at 284.8 eV corresponds to C–C/C–H bonds, 286.4 eV to C–O/C–N bonds, and 288.8 eV to C = O/O–C = O bonds. Upon the introduction of CDs, the relative intensity of the C = O peak at 288.8 eV increased, confirming the presence of surface carboxyl groups [13]. The N 1s spectra (Figs. 3e, h) displayed characteristic peaks at 399.6 eV, corresponding to amino ($-\text{NH}_2$) or amide ($-\text{NH}-\text{C}=\text{O}$) nitrogen species; this signal was significantly enhanced in PVA/CS@CDs-7, indicating CDs as an additional nitrogen source. The O 1s spectra (Figs. 3f, i) showed peaks at 532.5 eV attributed to C–O and O–H bonds. Following CDs incorporation, this peak exhibited minimal chemical shift but altered

peak shape, suggesting that CDs surface oxygen-containing groups interact with PVA/CS segments via hydrogen bonding or coordination rather than forming covalent linkages.

3.2. Optical Properties of PVA/CS@CDs Composite Hydrogels

The optical properties of PVA/CS@CDs composite hydrogels were investigated using UV-vis and PL spectroscopy. As shown in Fig. 4a, CDs exhibited a distinct UV absorption maximum at 390 nm, attributed to the $n \rightarrow \pi^*$ electronic transition of surface C = O groups [41], indicating abundant surface oxygen-containing functionalities [41, 42]. To optimize the fluorescent properties, the influence of CDs doping levels on hydrogel luminescence was systematically investigated. As depicted in Fig. 4b, under 390 nm excitation, PVA/CS@CDs hydrogels with varying CDs loadings (3, 5, 7, and 9 mL) all exhibited distinct blue emission peaks centered at 435 nm. Fluorescence intensity initially increased with CDs concentration, subsequently plateauing at higher loadings. This behavior is attributed to the following: at low CDs concentrations, insufficient luminescent centers exist within the hydrogel matrix; conversely, at excessive concentrations, ACQ or self-absorption effects may manifest between adjacent CDs, limiting fluorescence enhancement [43, 44]. Previous studies have demonstrated that hydrogel matrices can effectively suppress the aggregation of CDs, thereby maintaining their homogeneous dispersion and preserving high fluorescence quantum yields [44].

Figure 4c demonstrates that the PVA/CS@CDs-7 hydrogel exhibits bright blue fluorescence with a maximum emission peak at 435 nm under 380 nm excitation, delivering a Stokes shift of approximately 55 nm. Such a large Stokes shift is favorable for reducing self-absorption and improving the signal-to-noise ratio as well as fluorescence quantum yield [45, 46]. Notably, the hydrogel exhibited excitation-independent emission behavior, indicating that CDs maintained uniform dispersion and stable luminescent centers within the hydrogel matrix [8], in contrast to surface-passivated CDs that typically display excitation-dependent emission.

UV-vis spectroscopic analysis (Fig. 4d) reveals that the PVA/CS@CDs-7 hydrogel exhibits a broad absorption band in the 280–600 nm range without distinct characteristic peaks, which is attributed to light scattering effects arising from the dispersion of CDs within the three-dimensional hydrogel network, which is compounded by matrix interference. PL investigations demonstrate that the hydrogel displays excitation-independent emission behavior: the optimal emission peak (435 nm) is observed under 380 nm excitation, with the peak position remaining invariant across the 300–430 nm excitation range while only the fluorescence intensity varies (Fig. 4e). This excitation independence indicates that CDs maintain uniform dispersion and stable emissive centers within the hydrogel matrix, thereby enhancing the reproducibility and reliability of detection [47, 48]. Notably, such excitation-independent emission characteristics are also observed for fully surface-passivated CDs, where this phenomenon is attributed to single-electron transition mechanisms [47].

Under natural light, the PVA/CS@CDs-7 hydrogel appears pale yellow and transparent, while it emits intense blue fluorescence under 365 nm UV irradiation (Fig. 4f), consistent with the optical behavior of pure CDs solution. The distinct color contrast endows the hydrogel with intuitive visual detection potential [4, 43].

3.3. Fluorescence Sensing Performance toward Fe³⁺

To systematically evaluate the recognition specificity of PVA/CS@CDs-7 hydrogels toward Fe³⁺ ions, fluorescence responses were investigated in the presence of various common metal ions (Ca²⁺, Cd²⁺, Sr²⁺, Mn²⁺, Na⁺, K⁺, Cu²⁺, Co²⁺, and Fe³⁺). As illustrated in Fig. 5a, although most metal ions induced varying degrees of fluorescence quenching, Fe³⁺ demonstrated the most pronounced quenching effect, with quenching efficiency substantially exceeding competing ions. This selective response stems from the unique electronic configuration (3d⁵) and high charge density of Fe³⁺, enabling strong coordination interactions with CDs surface functional groups [9].

To evaluate the anti-interference capability of the sensing platform in complex practical matrices, the potential influence of commonly coexisting metal ions on Fe³⁺ detection was further examined. Various interfering ions, including Sr²⁺, Cu²⁺, Mn²⁺, Na⁺, K⁺, and Ca²⁺, were individually introduced at equal concentrations into the PVA/CS@CDs-7 hydrogel system containing Fe³⁺ (Fig. S3). The results revealed that the addition of these interfering ions did not induce appreciable changes in the fluorescence emission spectra of the hydrogel, and the fluorescence intensities remained essentially consistent with those of the system containing Fe³⁺ alone. These findings demonstrate that the PVA/CS@CDs-7 hydrogel sensor possesses satisfactory anti-interference performance for Fe³⁺ detection, providing a practical basis for its application in complex environmental samples.

The influence of Fe³⁺ concentration on hydrogel fluorescence intensity was subsequently investigated. As depicted in Fig. 5b, fluorescence intensity decreased monotonically with increasing Fe³⁺ concentration (1×10⁻³, 1×10⁻², 1×10⁻¹, and 1 M). Notably, under 365 nm UV illumination, the blue fluorescence gradually attenuated with increasing Fe³⁺ concentration, becoming completely quenched at high concentrations (> 0.1 M) (inset, Fig. 5b). This turn-off-type fluorescence response offers intuitive, visually observable detection capabilities suitable for rapid on-site screening [49].

Figure 5c presents the fluorescence response curves of the PVA/CS@CDs-7 hydrogel over the Fe³⁺ concentration range of 0–40 mM. With increasing Fe³⁺ concentration, the fluorescence intensity of the hydrogel decreased progressively, and the quenching process exhibited a satisfactory linear relationship. As shown in Fig. 5d, the fluorescence intensity ratio (F/F₀) followed the Stern–Volmer linear equation: $F/F_0 = -0.0247c + 0.9876$, with a linear correlation coefficient (R²) of 0.9934, indicating that the hydrogel sensor enables reliable quantitative detection over a broad concentration range. Based on the Stern–Volmer linear fitting (R² = 0.9934), the sensor demonstrates dependable quantitative detection capability within the 0–40 mM range. It is worth noting that this detection range is primarily oriented toward the

rapid screening of relatively high-concentration Fe^{3+} contamination scenarios, such as industrial wastewater, thereby complementing trace-level analytical sensors in terms of application scope.

Figure 5e visually illustrates the fluorescence emission changes under UV irradiation across the 0–40 mM Fe^{3+} concentration range. As Fe^{3+} concentration increased from 0 to 40 mM, the blue fluorescence progressively attenuated until complete quenching, exhibiting distinct colorimetric gradients. This visual detection modality requires no complex instrumentation, enabling semi-quantitative analysis through naked-eye observation for rapid on-site detection [8].

To further elucidate the mechanism of Fe^{3+} -induced fluorescence quenching in the hydrogel, the fluorescence decay curves of the PVA/CS@CDs-7 hydrogel before and after the addition of different concentrations of Fe^{3+} were measured using a time-correlated single-photon counting (TCSPC) system (Fig. S4). The amplitude-averaged fluorescence lifetimes were obtained by fitting the decay curves with a biexponential function. The pristine hydrogel exhibited an average lifetime of approximately 2.80 ns, which changed to about 2.69 ns and 2.85 ns upon the addition of 20 mM and 40 mM Fe^{3+} , respectively, corresponding to a variation of less than 6%. This result indicates that the fluorescence quenching of the hydrogel by Fe^{3+} is predominantly governed by a static quenching mechanism [50, 51], in which Fe^{3+} forms ground-state non-fluorescent complexes with the carboxyl and amino groups on the CDs surface via coordination bonds, rather than through dynamic processes such as collisional quenching of the excited-state fluorophores.

To rule out the possibility of non-specific fluorescence quenching induced by ionic strength at high Fe^{3+} concentrations, the equilibrium swelling ratio of the hydrogel was measured at varying Fe^{3+} concentrations (Fig. S5). The results showed that the swelling ratio increased continuously with rising Fe^{3+} concentration, confirming the occurrence of network contraction. However, the distinct concentration dependences between the quenching efficiency and the swelling ratio suggest that the specific coordination of Fe^{3+} , rather than the volumetric collapse-induced ACQ effect, is the dominant factor governing the fluorescence quenching.

4. Conclusion

In this study, a PVA/CS@CDs composite hydrogel featuring a dense layered structure was successfully fabricated via a freeze-thaw physical crosslinking strategy. The resulting material exhibits excellent coupled mechanical and optical properties, achieving a tensile stress of 0.47 MPa and a breaking strain of 681.76%—representing an increase of 259.31% over pristine PVA/CS—along with favorable mechanical resilience. Importantly, the incorporation of CDs not only imparts stable blue fluorescence emission ($\lambda_{\text{exc}} = 380$ nm, $\lambda_{\text{em}} = 435$ nm) to the hydrogel, but also ensures good dispersion of the CDs within the polymer matrix. Fluorescence lifetime analysis revealed that the addition of Fe^{3+} caused no significant change in the fluorescence lifetime of the hydrogel (variation < 6%), confirming that the quenching is dominated by a static mechanism. This quenching arises from the coordination of Fe^{3+}

with the carboxyl and amino groups on the CD surface, leading to the formation of ground-state non-fluorescent complexes. On this basis, the sensor exhibits a highly selective “turn-off” fluorescence response toward Fe^{3+} ($R^2 = 0.9934$), with reliable quantitative detection capability across the concentration range of 0–40 mM, and enables visual detection via a distinct color gradient under 365 nm UV light. Collectively, this work offers a new paradigm for the design of flexible detection materials that integrate high mechanical strength, self-recovery, and visual sensing functionalities, holding promise for the rapid screening of Fe^{3+} in industrial wastewater and environmental water samples.

Declarations

Conflict of Interest

The authors declare no competing interests.

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Author Contribution

Dandan Zhang: Conceptualization, Investigation, Data curation, Writing – original draft. Jiayi Chen: Investigation, Data acquisition. Xiaoli Wu: Investigation, Data acquisition. Jiajia Du: Visualization, Formal analysis. Xi Yang: Conceptualization, Funding acquisition, Writing – review & editing, Supervision. Xiaonan Liu: Conceptualization, Funding acquisition, Writing – review & editing, Supervision. Daohai Zhang: Conceptualization, Funding acquisition, Writing – review & editing, Project administration, Supervision. All authors reviewed and approved the final manuscript.

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Data Availability

All data supporting the findings of this study are available within the article and its supplementary materials.

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Figures

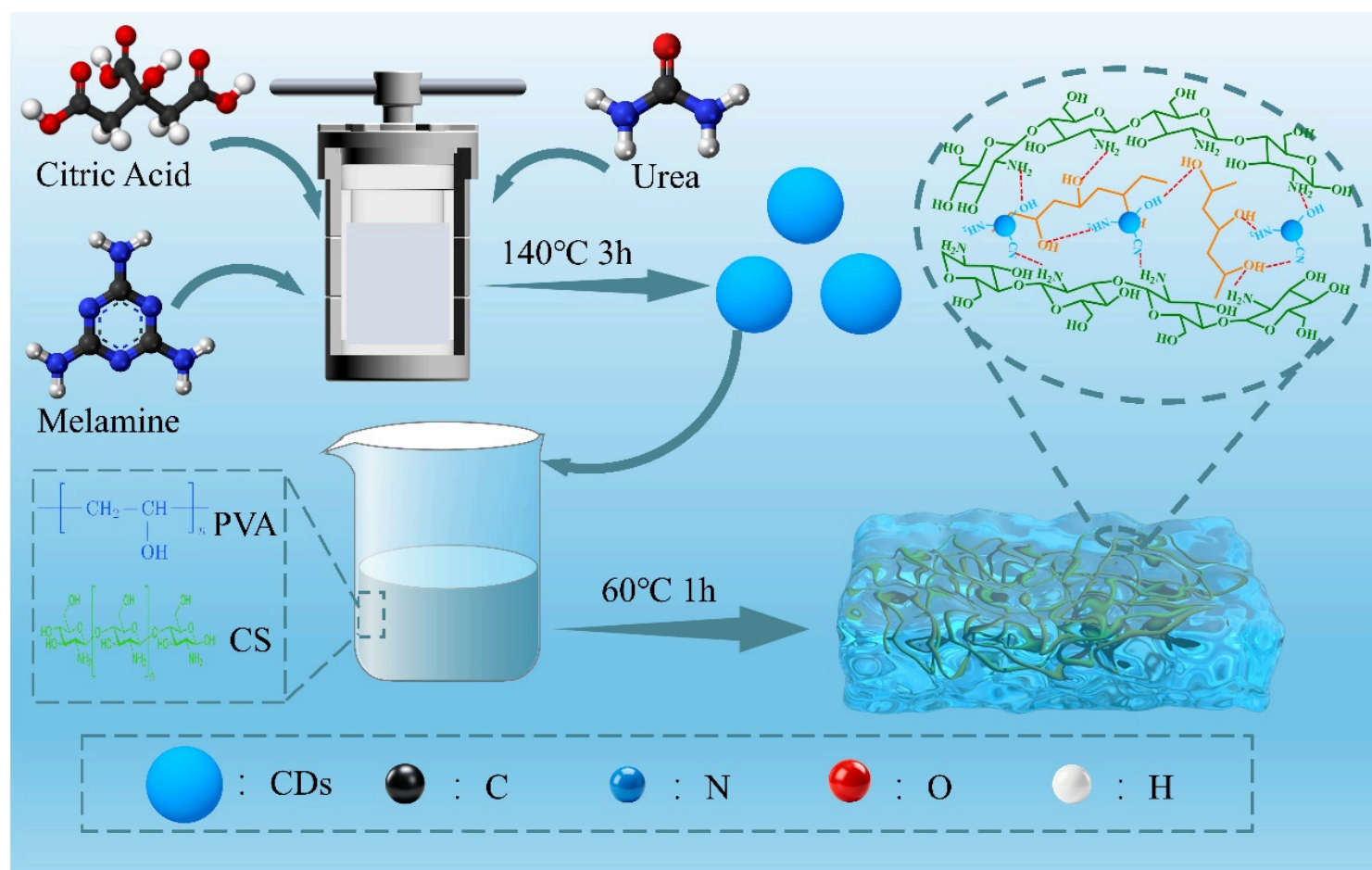


Figure 1

Schematic illustration of the fabrication route for PVA/CS@CDs composite hydrogel.

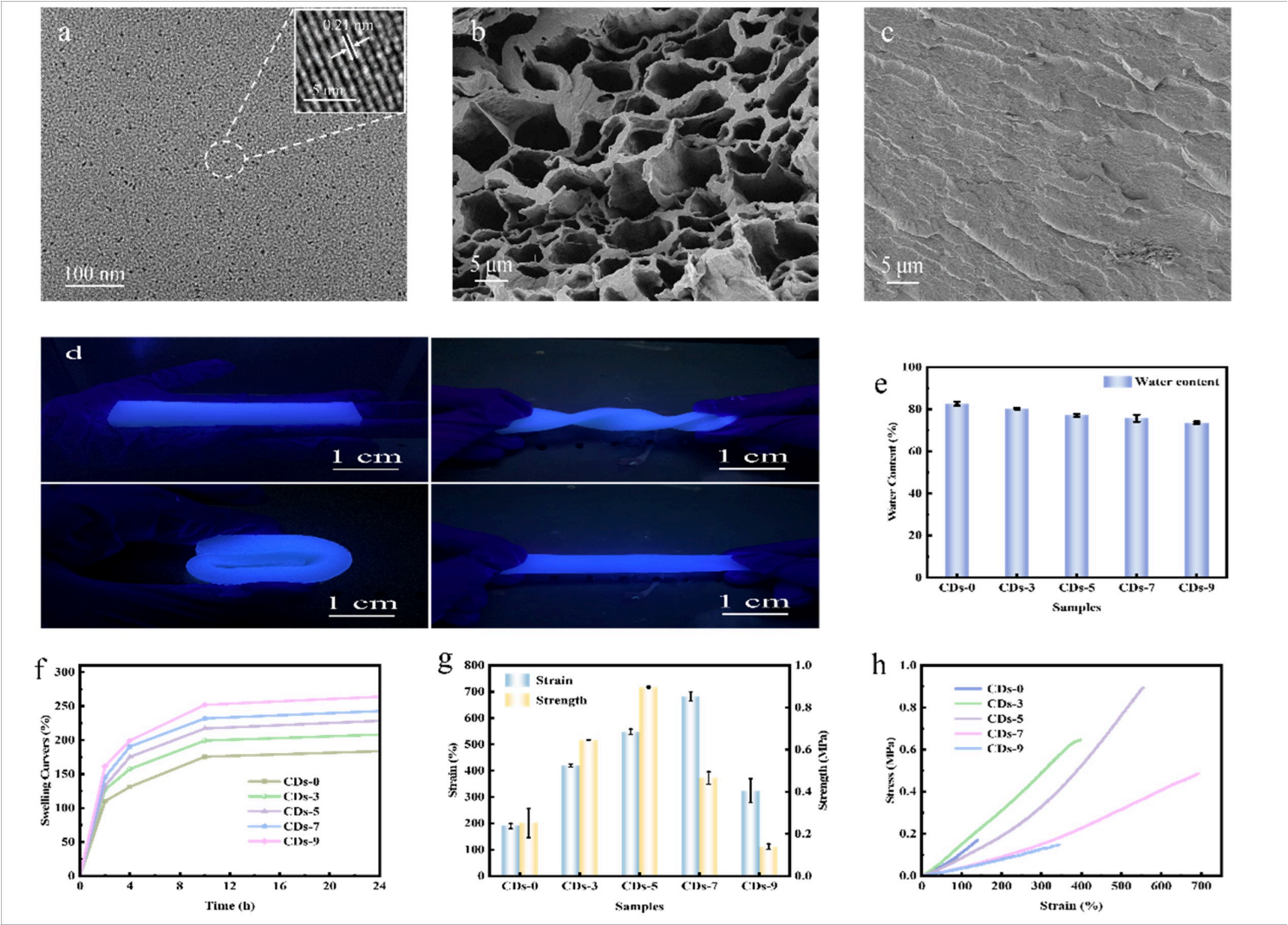


Figure 2

(a) TEM image of CDs; (b) SEM image of pristine PVA/CS hydrogels; (c) SEM image of PVA/CS@CDs composite hydrogels; (d) Photograph of the flexibility of PVA/CS@CDs hydrogel; (e) water content; (f) swelling kinetics; (g) breaking strain and tensile strength; and (h) stress-strain curves of hydrogels with varying CDs contents.

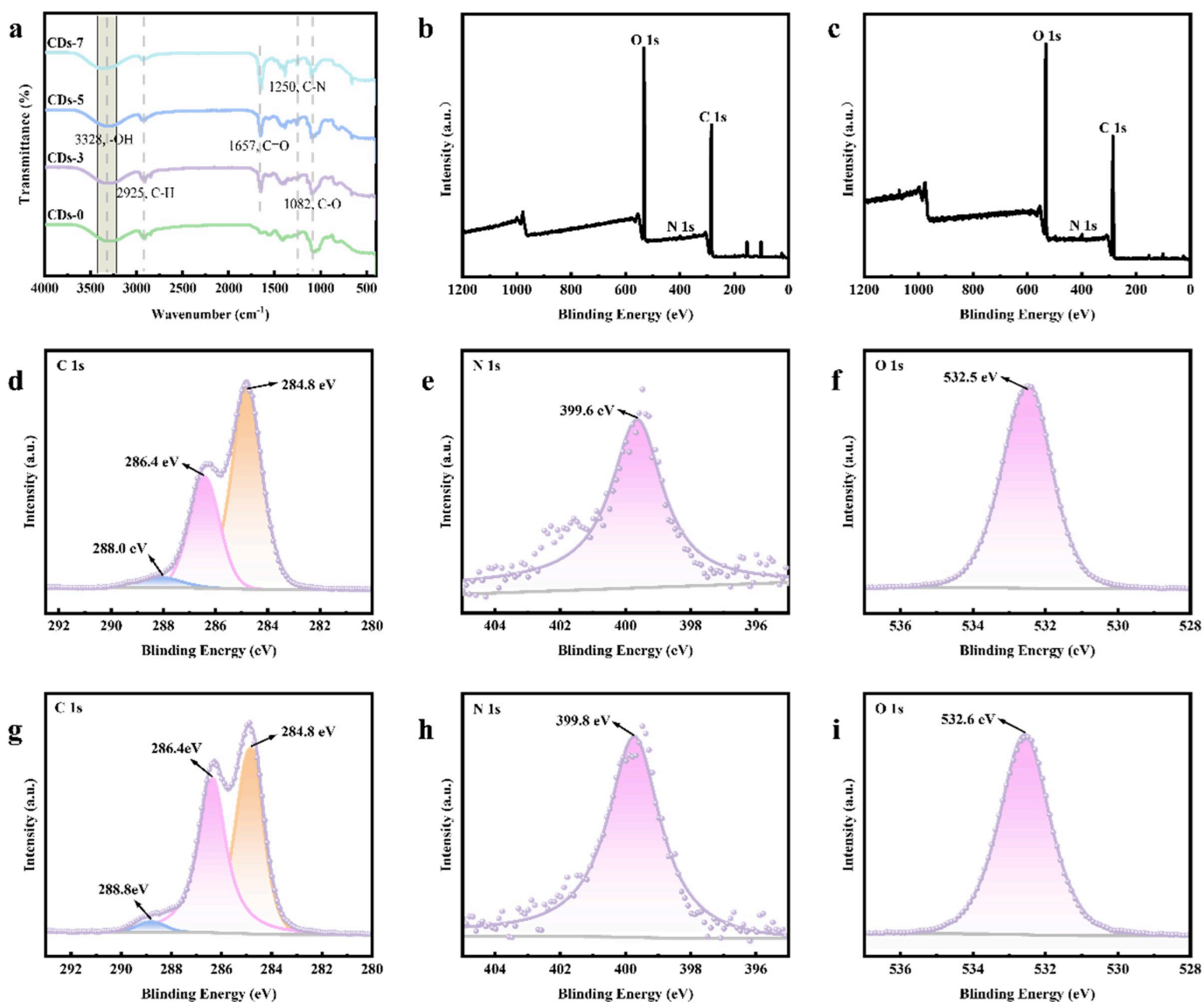


Figure 3

(a) FTIR spectra of PVA/CS and PVA/CS@CDs composite hydrogels; (b, c) survey XPS spectra of pristine PVA/CS and PVA/CS@CDs-7 hydrogels; (d-f) high-resolution C 1s, N 1s, and O 1s XPS spectra with peak fitting for PVA/CS; and (g-i) high-resolution C 1s, N 1s, and O 1s XPS spectra with peak fitting for PVA/CS@CDs-7.

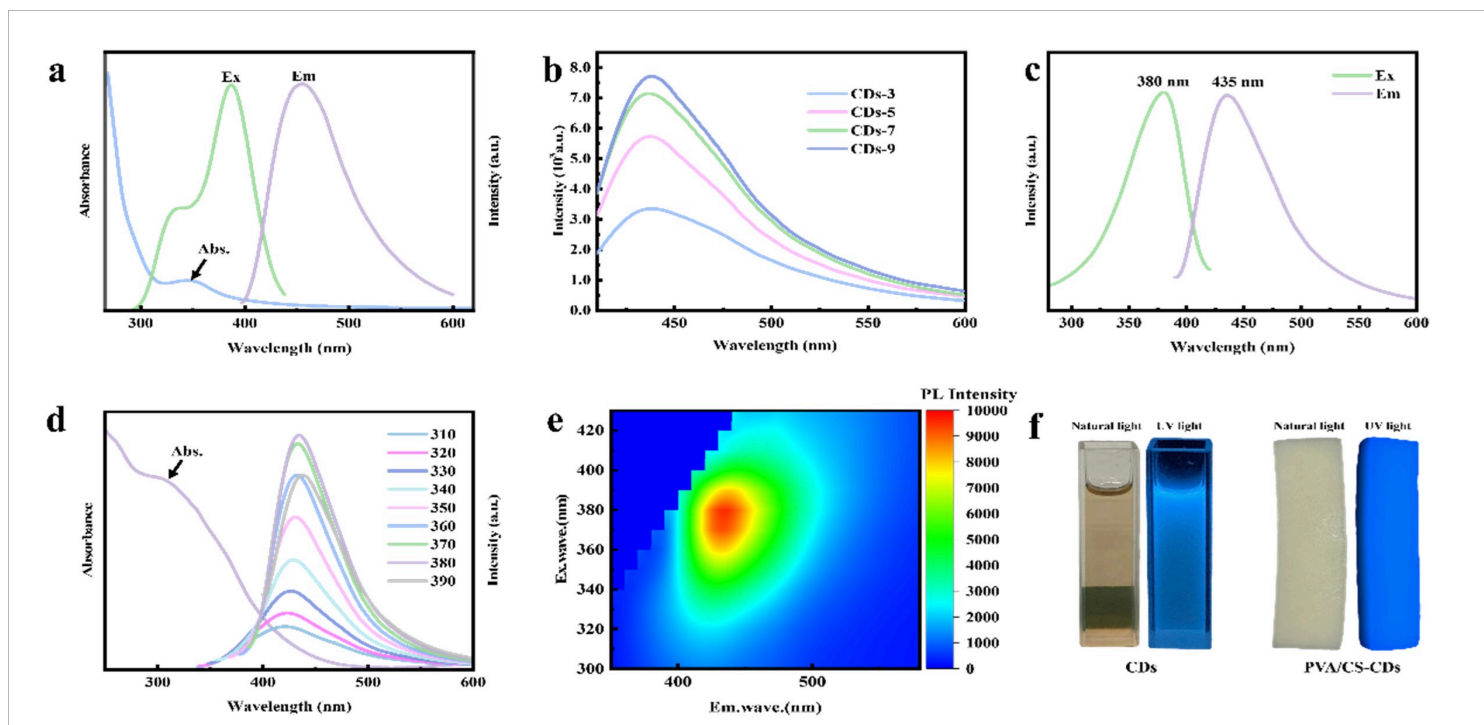


Figure 4

a) UV-vis absorption, fluorescence excitation, and emission spectra of the CDs; (b) photoluminescence spectra of PVA/CS@CDs hydrogels with varying CDs contents; (c) optimal excitation and emission wavelengths of PVA/CS@CDs-7; (d) UV-vis absorption and fluorescence emission spectra of PVA/CS@CDs-7; (e) three-dimensional fluorescence spectrum of PVA/CS@CDs-7; and (f) optical photographs of CDs (left) and PVA/CS@CDs-7 (right) under natural light (top) and 365 nm UV irradiation (bottom).

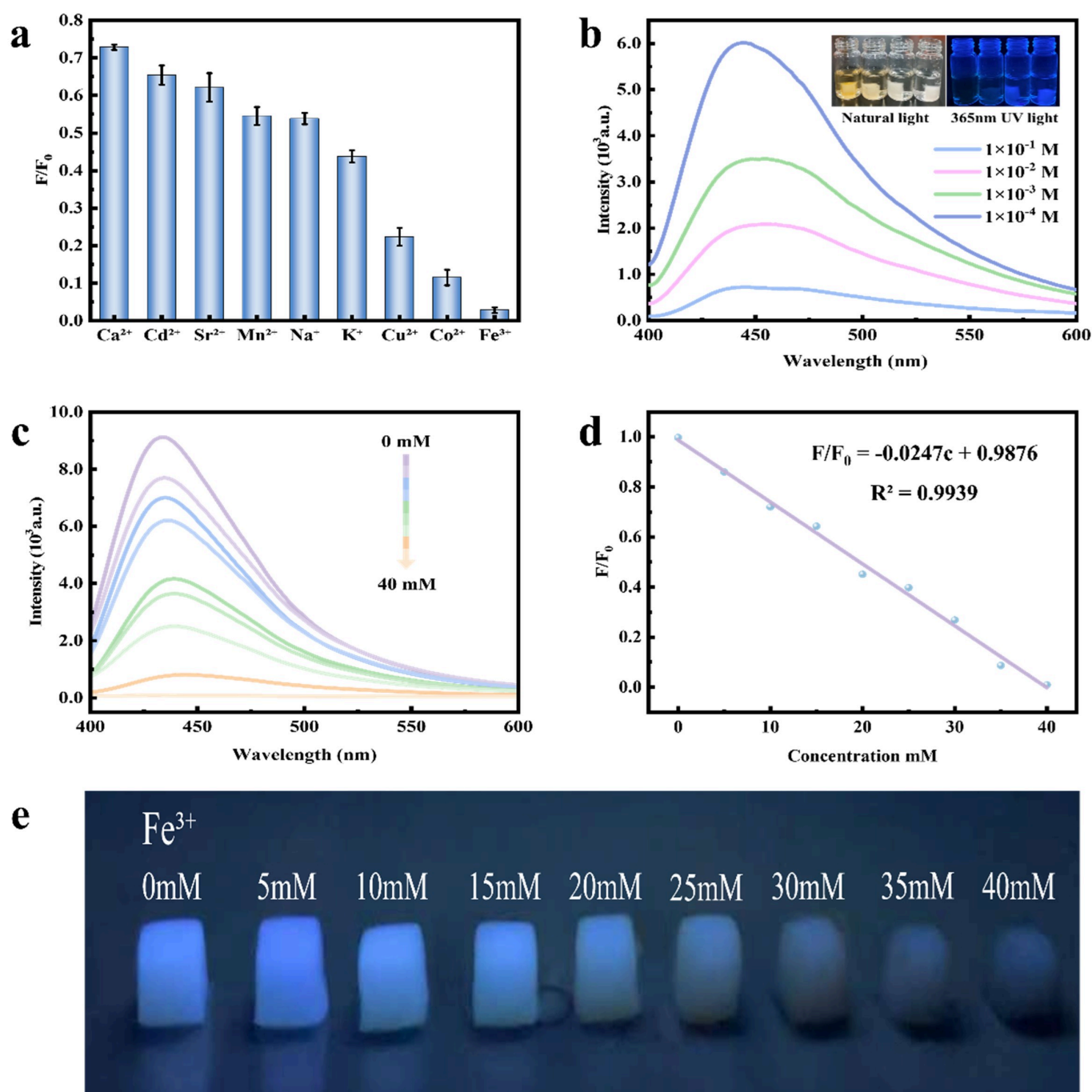


Figure 5

(a) Selectivity of PVA/CS@CDs hydrogels toward various metal ions (F/F_0); (b) fluorescence emission spectra of PVA/CS@CDs hydrogels in Fe^{3+} solutions with different concentrations ($\lambda_{\text{exc}}=380 \text{ nm}$); (c) fluorescence spectra of the hydrogels as a function of Fe^{3+} concentration (0-40 mM); (d) linear relationship between F/F_0 and Fe^{3+} concentration; and (e) photographs of PVA/CS@CDs hydrogels incubated with different concentrations of Fe^{3+} under 365 nm UV irradiation.

Supplementary Files

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