



## Article

# Synthesis and Evaluation of Peptide–Manganese Dioxide Nanocomposites as Adsorbents for the Removal of Strontium Ions

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**Abstract:** In this study, a novel organic–inorganic hybrid material IIGK@MnO<sub>2</sub> (2-naphthalenemethyl-isoleucine-isoleucine-glycine-lysine@manganese dioxide) was designed as a novel adsorbent for the removal of strontium ions (Sr<sup>2+</sup>). The morphology and structure of IIGK@MnO<sub>2</sub> were characterized using TEM, AFM, XRD, and XPS. The results indicate that the large specific surface area and abundant negative surface charges of IIGK@MnO<sub>2</sub> make its surface rich in active adsorption sites for Sr<sup>2+</sup> adsorption. As expected, IIGK@MnO<sub>2</sub> exhibited excellent adsorbing performance for Sr<sup>2+</sup>. According to the adsorption results, the interaction between Sr<sup>2+</sup> and IIGK@MnO<sub>2</sub> can be fitted with the Langmuir isotherm and pseudo-second-order equation. Moreover, leaching and desorption experiments were conducted to assess the recycling capacity, demonstrating significant reusability of IIGK@MnO<sub>2</sub>.



**Citation:** Lu, X.; Liu, Z.; Wang, W.; Wang, X.; Ma, H.; Cao, M. Synthesis and Evaluation of Peptide–Manganese Dioxide Nanocomposites as Adsorbents for the Removal of Strontium Ions. *Nanomaterials* **2024**, *14*, 52. <https://doi.org/10.3390/nano14010052>

Academic Editor:  
Diego Cazorla-Amorós

Received: 1 December 2023

Revised: 19 December 2023

Accepted: 21 December 2023

Published: 23 December 2023



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**Keywords:** peptide assembly; MnO<sub>2</sub>; Sr<sup>2+</sup> removal

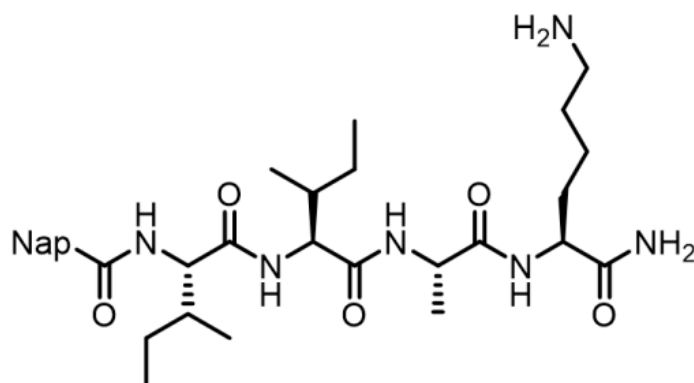
## 1. Introduction

Nuclear energy is widely used due to its ability to reduce greenhouse effects, high energy density, and ease of storing nuclear fuel [1]. However, in nuclear disasters such as Chernobyl and Fukushima, the release of large amounts of radioactive isotopes and waste has had a catastrophic impact on ecosystems, causing significant damage. In terms of radioactive safety, strontium (<sup>90</sup>Sr), cobalt (<sup>60</sup>Co), and cesium (<sup>137</sup>Cs) are considered the main radioactive isotopes due to their relatively long half-lives, high solubility, and easy transfer [2]. Among them, <sup>90</sup>Sr is a carcinogen and hazardous pollutant that has chemical properties similar to calcium and is easily absorbed by the human body [3]. Therefore, the development of new materials for removing strontium ions (Sr<sup>2+</sup>) from aqueous solutions has received great attention. At present, various methods have been developed to remove radioactive toxic ions from aqueous solutions, including chemical precipitation, membrane, solvent extraction, ion exchange, and adsorption [4–8]. Among them, the adsorption method is the most used and highly efficient technology. Up to now, a large number of nanomaterials, such as hydroxyapatite nanoparticles, metal sulfide, metallic oxide, graphene oxide (GO), and MOFs, have been employed as adsorbents for removing Sr<sup>2+</sup> [9–13]. Attributed to their larger specific surface area and more active sites, MnO<sub>2</sub> nanoparticles have been widely studied and applied as effective adsorbents for Sr<sup>2+</sup> [14]. Moreover, MnO<sub>2</sub> particles also exhibit selectivity for Sr<sup>2+</sup> in the presence of competing ions, including Na<sup>+</sup>, K<sup>+</sup>, Ca<sup>2+</sup>, and Mg<sup>2+</sup> [15]. However, MnO<sub>2</sub> nanoparticles have drawbacks such as susceptibility to van der Waals forces, which may lead to aggregation and a decrease in their adsorption performance [16]. Therefore, it is necessary to fix MnO<sub>2</sub> nanoparticles

on the carrier to improve their dispersion stability as well as adsorption performance and reusability for  $\text{Sr}^{2+}$  removal.

Due to the presence of abundant functional groups such as hydroxyl groups, thiol groups, and carboxyl groups as adsorption sites, along with diverse secondary structures, a series of peptides has been studied for their binding to various metallic materials, including metal oxides, metal sulfides, and metals [17,18]. Moreover, some specifically designed peptides demonstrate a selective affinity for particular metal ions, which can serve as effective metal-binding moieties in aqueous solutions and promote selective metal ion adsorption [19]. For example, Mondal et al. confirmed the affinity of a tripeptide gel for metal ions, including lead and cadmium [20]. Besides direct metal ion adsorption, the peptides can be used to composite with other materials to achieve synergistic metal ion adsorption. At present, various peptide sequences have been used for biomimetic synthesis of ceramics, metal sulfides, metal oxides, and metal nanoparticles, and composite materials with low dispersion, good crystallinity, diverse morphology, and excellent biological functions have been constructed in mild water environments [21,22]. In addition, peptides have excellent assembly performance and can form different functional structures through covalent and noncovalent interactions (such as hydrophilicity and hydrophobicity,  $\pi$ - $\pi$  stacking, hydrogen bonding, and electrostatic interactions). These peptide assembly structures can regulate the structure of peptide-inorganic nanoparticles by inducing the nucleation and growth process of inorganic nanoparticles and selectively binding to certain crystal planes. This makes peptides an excellent template for in situ loading and construction of inorganic nanomaterials.

As mentioned in the above text, though  $\text{MnO}_2$  nanoparticles are good candidates for  $\text{Sr}^{2+}$  absorption and removal, they easily experience severe agglomeration and poor dispersibility in aqueous solution, which influences the adsorption performance. To address this issue, we chose the self-assembled nanofibrils of short peptide 2-naphthalenemethyl-isoleucine-isoleucine-glycine-lysine (IIGK) (Figure 1) as templates to mediate the mineralization of  $\text{MnO}_2$  on their surface, which can greatly enhance the dispersibility of  $\text{MnO}_2$  nanoparticles. Compared with other methods for synthesizing  $\text{MnO}_2$ , such as electrodeposition, demulsification, and hydrothermal methods, this method is environmentally friendly, mild, and easy to operate. The physicochemical properties of the as-prepared IIGK@ $\text{MnO}_2$  were systematically characterized first by using various techniques. Then, experiments were performed to investigate the adsorption performance of the IIGK@ $\text{MnO}_2$  nanocomposites towards  $\text{Sr}^{2+}$ . The results show that the IIGK@ $\text{MnO}_2$  nanocomposites have a strong affinity and adsorption capacity for  $\text{Sr}^{2+}$ , and a synergistic effect on the adsorption of  $\text{Sr}^{2+}$  with IIGK@ $\text{MnO}_2$  can be concluded. This study demonstrated the merits of IIGK@ $\text{MnO}_2$  as a promising adsorbent to remove  $\text{Sr}^{2+}$  from nuclear wastewater.



**Figure 1.** Molecular structure of the short peptide IIGK.

## 2. Materials and Methods

### 2.1. Materials

Manganese chloride tetrahydrate ( $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ ), potassium permanganate ( $\text{KMnO}_4$ ), sodium nitrate ( $\text{NaNO}_3$ ), potassium nitrate ( $\text{KNO}_3$ ), strontium nitrate ( $\text{Sr}(\text{NO}_3)_2$ ), sodium hydroxide ( $\text{NaOH}$ ), and potassium hydroxide ( $\text{KOH}$ ) were acquired from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). All working solutions were prepared using distilled water. The pH was adjusted using  $\text{NaOH}$  and  $\text{HCl}$  as needed and monitored with a digital pH meter. All adsorption experiments were conducted at ambient temperature.

### 2.2. Preparation of IIGK@ $\text{MnO}_2$ Nanocomposite

First, 300 mg IIGK powder was dissolved in 250 mL ultrapure water, which was then subjected to ultrasonic dispersion for approximately 10 min. Then, the mixture was left to stand overnight, followed by adding another 225.6 mL ultrapure water. Subsequently, 9.6 mL  $\text{KMnO}_4$  (30 mM) aqueous solution and 9.6 mL  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (42 mM) aqueous solution were added to the mixture, which was then stirred for 24 h. Finally, the samples were centrifuged, washed three times with ultrapure water, and subsequently freeze-dried to obtain IIGK@ $\text{MnO}_2$  powder for further use.

### 2.3. Characterization

The morphology of samples was characterized using a scanning electron microscope (SEM, Hitachi S-4800, Tokyo, Japan) and transmission electron microscope (TEM, JOEL JEM1400 Plus, Tokyo, Japan). The SEM equipped with an Oxford Instruments energy-dispersive X-ray spectroscopy (EDS) system was used for analyzing elemental composition. The thickness of materials was measured using atomic force microscopy (AFM, Santa Barbara Veeco Nanoscope Iva, Santa Barbara, CA, USA). The size and zeta potential of samples were determined on a Zetasizer Nano (DLS, Malvern NANO ZS, Marvern City, UK). The element analysis of the samples before and after  $\text{Sr}^{2+}$  adsorption was carried out using X-ray photoelectron spectroscopy (XPS, Thermo Scientific Esclab 250Xi, Waltham, MA, USA) with a monochromatic Al  $K\alpha$  X-ray source (15 KV). Fourier-transform infrared spectroscopy (FT-IR, Thermo Scientific Nicolet iS5, Waltham, MA, USA) was performed to investigate the interaction between  $\text{MnO}_2$  and IIGK. X-ray diffraction (XRD, PANalitical X'Pert PRO MPD, Almelo, The Netherlands) was performed to study the crystal structure of the samples using  $\text{Cu-K}\alpha$  radiation in the  $2\theta$  range of  $10^\circ$ – $80^\circ$  at room temperature.  $\text{N}_2$  adsorption–desorption isotherm was conducted on the gas sorption analyzer at 373.15 K (Malvern PANalitical Autosorb-6B, Marvern City, UK), and the specific area and the average pore diameters were stimulated using the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) method. Inductively coupled plasma optical emission spectroscopy (ICP-OES, HORIBA JY 2000-2, Loos, Naples, FL, USA) was employed to determine the concentration of residual metal ions in the solution.

### 2.4. Adsorption Performance of IIGK@ $\text{MnO}_2$

All adsorption experiments were carried out in 15 mL polyethylene centrifuge tubes. After mixing 1 mg IIGK@ $\text{MnO}_2$  with 10 mL  $\text{Sr}^{2+}$  aqueous solution, the pH of the mixture was adjusted to a suitable value. The mixture was left in a constant-temperature shaker for a certain time, followed by centrifugation at 12,000 rpm for 20 min to extract the supernatant. Finally, the residue  $\text{Sr}^{2+}$  concentration in the adsorbed solution was measured using ICP-OES. The equilibrium adsorption amount ( $q_e$ , mg/g) and removal rate ( $R$ , %) of IIGK@ $\text{MnO}_2$  towards  $\text{Sr}^{2+}$  can be calculated by the following equations:

$$q_e = (c_0 - c_e) \times \frac{V}{M} \quad (1)$$

$$R = \frac{c_0 - c_e}{c_0} \times 100\% \quad (2)$$

where  $c_0$  (mg/L) and  $c_e$  (mg/L) are the initial and equilibrium concentrations of  $\text{Sr}^{2+}$  in the solution, respectively.  $V$  (mL) represents the volume of the adsorbed solution, and  $M$  (g) is the quantity of adsorbent.

### 2.5. Desorption of Strontium Ions from IIGK@MnO<sub>2</sub>

The desorption behavior of the IIGK@MnO<sub>2</sub> was investigated by treating IIGK-MnO<sub>2</sub>-Sr composites with 1 M HCl aqueous solution. The amount of  $\text{Sr}^{2+}$  desorbed from the IIGK@MnO<sub>2</sub>,  $Q_{D\_Sr}$ , was determined according to Equation (3), while the desorption efficiency ( $De$ , %) was obtained from Equation (4).

$$Q_{D\_Sr} = \frac{C_D \times V_D}{M_D} \quad (3)$$

$$De = \frac{Q_{D\_Sr}}{q_e} \times 100 \quad (4)$$

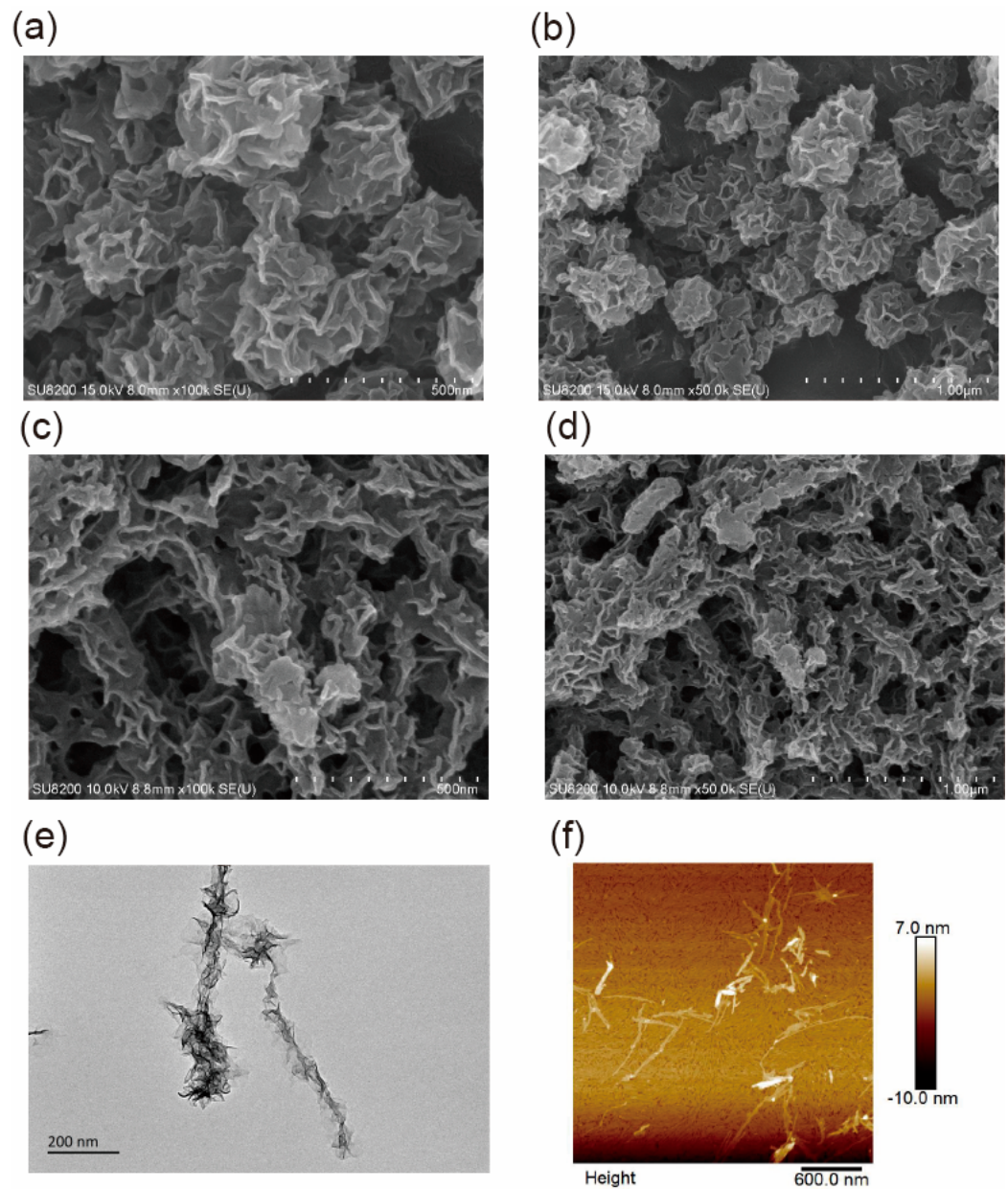
where  $C_D$  (mg/L) is the equilibrium desorption concentrations of  $\text{Sr}^{2+}$ ,  $V_D$  (mL) represents the volume of the eluent, and  $M_D$  (g) is the weight of adsorbent after  $\text{Sr}^{2+}$  adsorption.

## 3. Results and Discussion

### 3.1. Synthesis and Characterization of IIGK@MnO<sub>2</sub> Nanocomposite

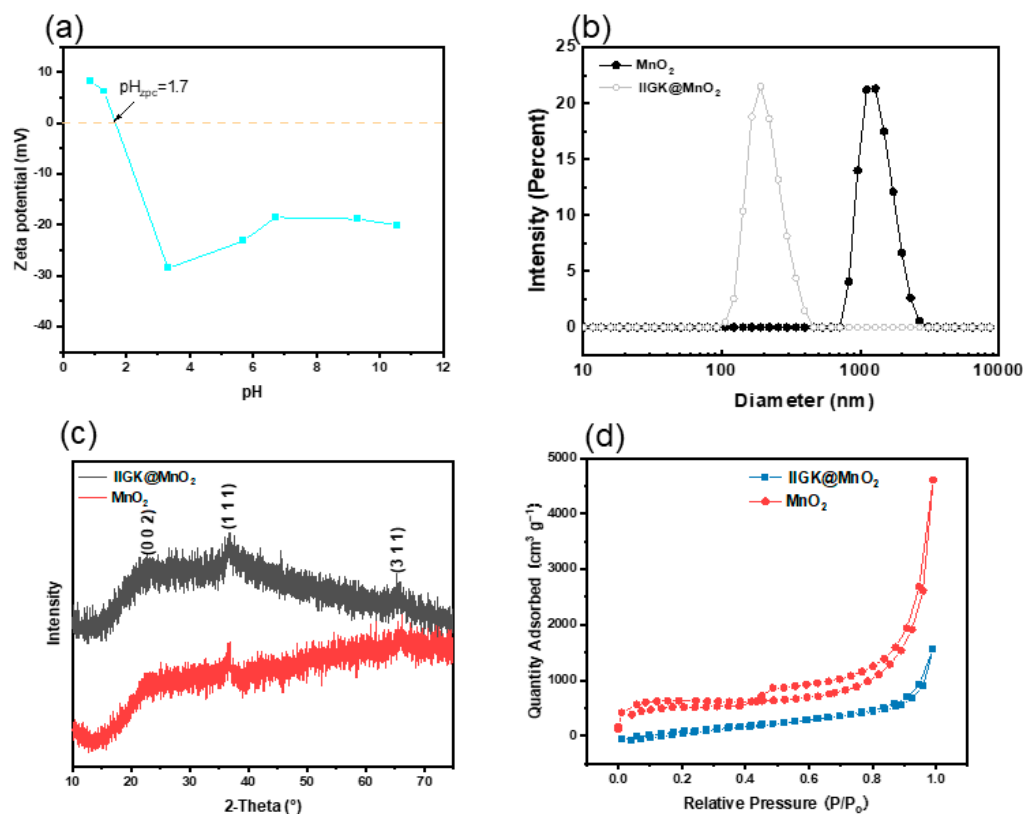
In this work, the IIGK@MnO<sub>2</sub> nanocomposite was synthesized by depositing MnO<sub>2</sub> nanoparticles along peptide fibers using a green, simple, and easy-to-operate biomimetic mineralization method. For comparison, pure manganese dioxide nanoparticles without peptide involvement were also synthesized. The morphology of MnO<sub>2</sub> nanoparticles and IIGK@MnO<sub>2</sub> nanocomposites was characterized using SEM, TEM, and AFM. As shown in Figure 2a,b, the MnO<sub>2</sub> nanoparticles generated without peptide show a disordered flower shape with a diameter of 400–500 nm, while the IIGK@MnO<sub>2</sub> nanocomposites took on elongated structures with many MnO<sub>2</sub> nanoparticles aligned in an axial direction (Figure 2c,d). The TEM result (Figure 2e) shows more clearly the one-dimensional nanowire/nanosheet composited structure. The results indicate that MnO<sub>2</sub> is generated through biomimetic mineralization using IIGK short peptides as templates. The inner core should be the IIGK fibrils, while the outer layer sheets should be MnO<sub>2</sub> nanostructures. The results demonstrate clearly the template role of the peptide fibrils in mediating MnO<sub>2</sub> mineralization. The formation of fibrous IIGK@MnO<sub>2</sub> nanocomposite can be confirmed with an AFM image (Figure 2f).

The surface zeta potential of the one-dimensional fiber-like IIGK@MnO<sub>2</sub> nanocomposites at different pH was then measured, as shown in Figure 3a. It can be observed that the isoelectric point of IIGK@MnO<sub>2</sub> appears at pH 1.7. While the pH value increases from 0.9 to 10.5, the zeta potential of IIGK@MnO<sub>2</sub> decreases gradually from 10 mV to −40 mV. Notably, when the pH value is greater than 1.7, IIGK@MnO<sub>2</sub> undergoes charge reversal, changing from a positively charged surface to a negatively charged surface. Moreover, the excess negative charges on the IIGK@MnO<sub>2</sub> surface are beneficial for the subsequent adsorption of  $\text{Sr}^{2+}$ . The particle size distribution of MnO<sub>2</sub> and IIGK@MnO<sub>2</sub> was measured using the dynamic light scattering measurement. As shown in Figure 3b, the size of IIGK-induced IIGK@MnO<sub>2</sub> nanocomposites is about 100–400 nm, while that of MnO<sub>2</sub> produced in the absence of peptide is above 1000 nm. This result indicates that the IIGK@MnO<sub>2</sub> nanocomposites induced with IIGK have a higher dispersion stability in the solution and a larger specific surface area, which is beneficial for the adsorption of  $\text{Sr}^{2+}$ .



**Figure 2.** (a,b) SEM images of MnO<sub>2</sub>, (c,d) SEM images of IIGK/MnO<sub>2</sub>, (e) TEM, and (f) AFM images of IIGK/MnO<sub>2</sub> nanocomposite.

In order to investigate the crystal structure, X-ray powder diffraction (XRD) was employed to characterize IIGK/MnO<sub>2</sub> and MnO<sub>2</sub>. The XRD patterns of the IIGK/MnO<sub>2</sub> nanocomposites and MnO<sub>2</sub> itself are shown in Figure 3c. For MnO<sub>2</sub>, the peaks are found at  $2\theta = 22.8^\circ$ ,  $36.5^\circ$ , and  $65.7^\circ$ . These crystalline microregions correspond to  $\delta$ -standard diffraction patterns (002), (111), and (311) of MnO<sub>2</sub> (JCPDS 80-1098), respectively. The same peaks were found in the XRD patterns of IIGK/MnO<sub>2</sub>, indicating that MnO<sub>2</sub> was successfully loaded on the IIGK fibril's surface. Moreover, as indicated by the weak XRD peaks, the IIGK/MnO<sub>2</sub> nanocomposites show a low crystallinity level. The reason for the poor crystallinity may be due to the fast reaction rate during the interaction between KMnO<sub>4</sub> and MnCl<sub>2</sub>, which leads to the rapid formation and precipitation of MnO<sub>2</sub> [23]. This rapid process may have hindered the formation of an orderly atomic arrangement.

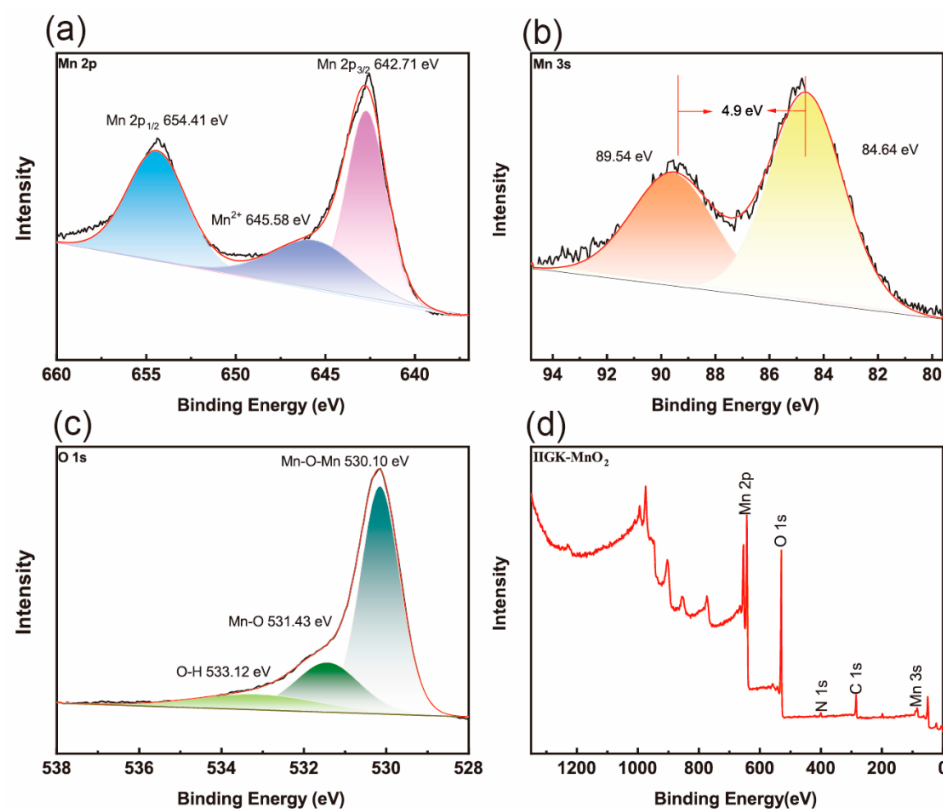


**Figure 3.** (a) Zeta potential of IIGK@MnO<sub>2</sub>, (b) diameter distribution of MnO<sub>2</sub> and IIGK@MnO<sub>2</sub>, (c) XRD spectra of IIGK@MnO<sub>2</sub> and MnO<sub>2</sub>, and (d) N<sub>2</sub> adsorption–desorption isotherms of IIGK@MnO<sub>2</sub> and MnO<sub>2</sub>.

Subsequently, the surface area, pore size, as well as pore volume of the IIGK@MnO<sub>2</sub> and MnO<sub>2</sub> were determined using BET analysis and the BJH method. According to the adsorption–desorption isotherms shown in Figure 3d, the surface area of IIGK@MnO<sub>2</sub> is approximately 46.19 m<sup>2</sup>·g<sup>−1</sup>. In addition, the pore volume and average pore size of IIGK@MnO<sub>2</sub> are calculated to be 2.40 cm<sup>3</sup>·g<sup>−1</sup> and 14.02 nm, respectively. The whole results also indicate that the IIGK@MnO<sub>2</sub> nanocomposite has suitable physical properties as an adsorbent and is suitable for the adsorption of Sr<sup>2+</sup> [24].

Furthermore, Fourier transform infrared spectroscopy (FTIR) was used to characterize IIGK@MnO<sub>2</sub> at a molecular level (Figure S1). The broad band at about 3421.6 cm<sup>−1</sup> belongs to the stretching vibration of -OH, which may be attributed to the adsorption of a small amount of water by IIGK@MnO<sub>2</sub> and the presence of hydroxyl groups on the Mn surface [25]. In addition, the characteristic absorption peaks at 1628.2 cm<sup>−1</sup>, 1383.7 cm<sup>−1</sup>, and 1022.8 cm<sup>−1</sup> can be attributed to the bending vibration of the hydroxyl group (Mn-OH) directly connected to the Mn atom. The peak appearing at 500 cm<sup>−1</sup> indicates the presence of [MnO<sub>6</sub>] octahedra, which is caused by the stretching of Mn-O and Mn-O-Mn bonds in the octahedral structure [26]. The elements' valence states of IIGK@MnO<sub>2</sub> were further characterized using XPS spectroscopy. As shown in Figure 4a, the XPS spectrum of Mn 2p exhibits two peaks at 642.7 eV and 654.4 eV, corresponding to Mn 2p 3/2 and Mn 2p 1/2, respectively. The spin energy range of 11.7 eV indicates that the IIGK short peptide fibers are mainly used as templates for the deposition of MnO<sub>2</sub> [27]. In addition, the weak peak appearing at 645.6 eV indicates the presence of a small amount of unreacted Mn<sup>2+</sup> in the system. According to Chigane et al., the XPS spectrum of Mn 3s can split into two weaker peaks, which are located at 84.6 eV and 89.5 eV, corresponding to Mn<sup>4+</sup> and Mn<sup>3+</sup>, respectively [28]. The difference in binding energy between these two peaks increases with the decrease in the valence state of Mn. As shown in Figure 4b, a 4.9 eV difference can be observed between the two peaks, indicating that Mn in IIGK@MnO<sub>2</sub> is mainly composed of

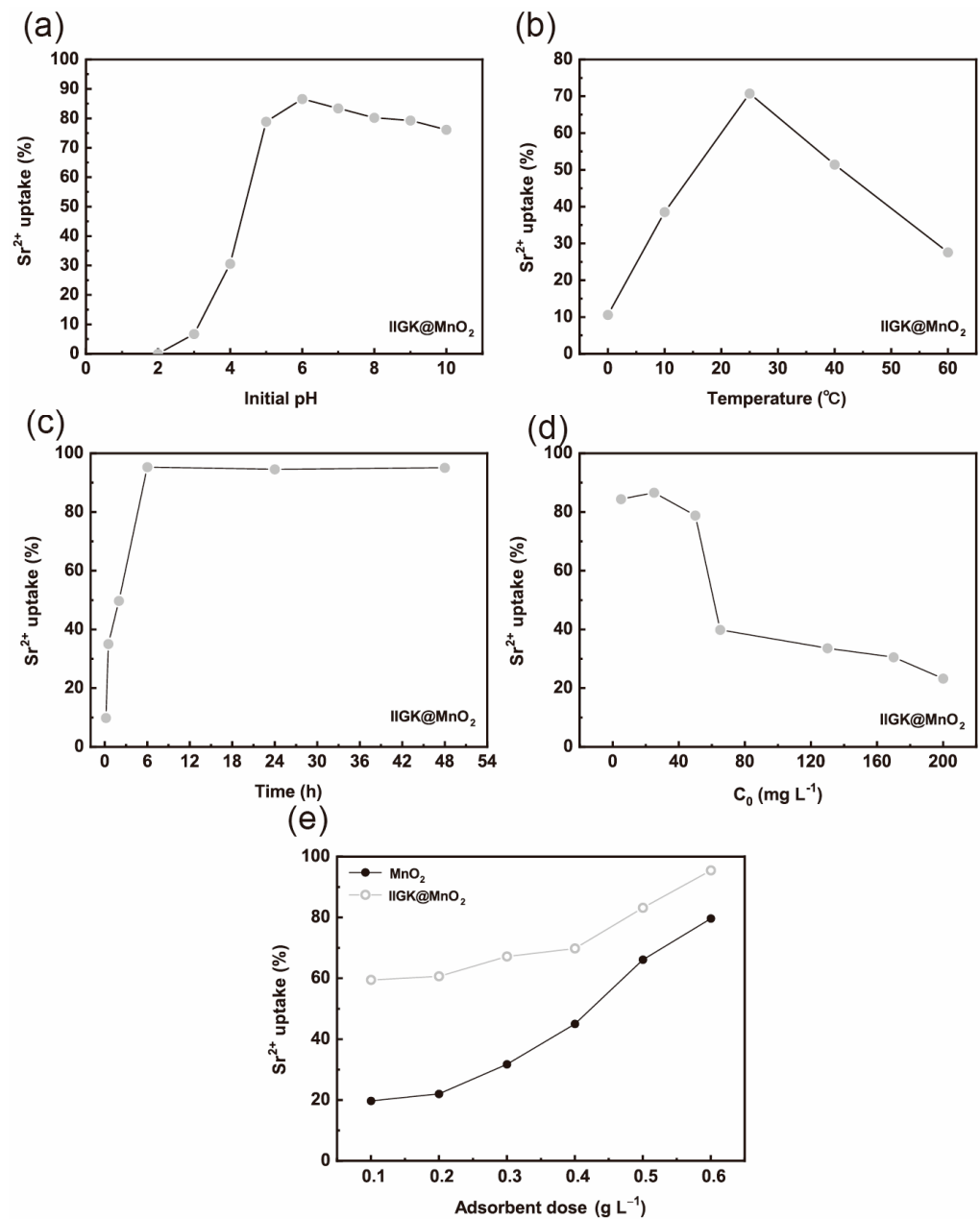
$\text{Mn}^{4+}$ , with a small amount of  $\text{Mn}^{3+}$  present. The O 1s peak exhibits three resolved peaks, with binding energies concentrated at 530.1 eV, 531.4 eV, and 533.12 eV (Figure 4c). These peaks are attributed to Mn-O-Mn, Mn-O, and O-H, respectively [29]. Figure 4d shows the complete XPS spectrum of IIGK@ $\text{MnO}_2$ , including Mn 3s, Mn 2p, C 1s, O 1s, and N 1s signals, indicating the successful mineralization of  $\text{MnO}_2$ .



**Figure 4.** XPS spectra of (a) Mn 2p, (b) Mn 3s, and (c) O 1s of IIGK@ $\text{MnO}_2$ . (d) XPS full survey spectra of IIGK@ $\text{MnO}_2$ . The fitting data of spectra (a–c) are shown by different colors.

### 3.2. $\text{Sr}^{2+}$ Adsorption Studies

Previous reports have indicated that  $\text{MnO}_2$  possesses excellent adsorbing performance for  $\text{Sr}^{2+}$ . Then, the performance of IIGK@ $\text{MnO}_2$  in adsorbing  $\text{Sr}^{2+}$  was studied. The ability of IIGK@ $\text{MnO}_2$  to adsorb  $\text{Sr}^{2+}$  at different pH values was first evaluated. As shown in Figure 5a, the adsorption efficiency of  $\text{Sr}^{2+}$  increases with the increase in pH value and then tends to equilibrium. The low adsorption efficiency of IIGK@ $\text{MnO}_2$  for  $\text{Sr}^{2+}$  in acidic pH environments may be caused by the competitive interaction between  $\text{H}^+$  and  $\text{Sr}^{2+}$ . At higher pH conditions, the concentration of  $\text{H}^+$  is lower; thus, there are more active sites left on IIGK@ $\text{MnO}_2$  for the adsorption of  $\text{Sr}^{2+}$ . In addition, under high pH conditions, the surface of IIGK@ $\text{MnO}_2$  carries more negative charges, which has a stronger electrostatic force on positively charged  $\text{Sr}^{2+}$ , thereby improving the adsorption efficiency of  $\text{Sr}^{2+}$  [30]. Except for pH, temperature also affects the adsorption efficiency of IIGK@ $\text{MnO}_2$  for  $\text{Sr}^{2+}$ . As shown in Figure 5b, IIGK@ $\text{MnO}_2$  has the highest adsorption efficiency for  $\text{Sr}^{2+}$  at room temperature. When the temperature rises above 40 °C, the adsorption efficiency sharply decreases. This phenomenon may be due to the weakening of the interaction between IIGK@ $\text{MnO}_2$  and  $\text{Sr}^{2+}$  at higher temperatures, resulting in the desorption of  $\text{Sr}^{2+}$  [31].



**Figure 5.** Sr<sup>2+</sup> removal efficiency of the IIGK@MnO<sub>2</sub> nanocomposite at different (a) pH, (b) temperature, (c) contact time, (d) initial Sr<sup>2+</sup> concentration, and (e) different adsorbent dosage.

The contact time between IIGK@MnO<sub>2</sub> and Sr<sup>2+</sup> would also affect the adsorption efficiency. As shown in Figure 5c, the adsorption of Sr<sup>2+</sup> by IIGK@MnO<sub>2</sub> rapidly reaches equilibrium at 6 h. The initial rapid adsorption may be attributed to the large number of available adsorption sites exposed on the surface of IIGK@MnO<sub>2</sub>. Then, the adsorption process reached equilibrium after 6 h, indicating that most of the available adsorption sites were occupied. The whole adsorption process was assessed using both the pseudo-first-order and pseudo-second-order kinetic models. The equations are as follows [32]:

$$\lg(q_e - q_t) = \lg q_e - \frac{k_1}{2.303} t \quad (5)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \quad (6)$$

where  $q_e$  and  $q_t$  represent the  $\text{Sr}^{2+}$  adsorption capacity (mg/g) at equilibrium or at time  $t$  (adsorption time), respectively;  $k_1$  ( $\text{min}^{-1}$ ) and  $k_2$  ( $\text{g}/(\text{mg}\cdot\text{min})$ ) represent the rate constants for the pseudo-first-order and pseudo-second-order models. The fitted parameters are summarized in Table 1. Comparing the  $R^2$  value of these models, the pseudo-second-order model fits better than the pseudo-first-order model [33].

**Table 1.** Fitted parameters of pseudo-first-order and pseudo-second-order kinetic models.

| Model               | Parameters                                      | Value   |
|---------------------|-------------------------------------------------|---------|
| Pseudo-First-Order  | $q_e$ (mg/g)                                    | 238.94  |
|                     | $k_1$ ( $\text{min}^{-1}$ )                     | 0.489   |
|                     | $R^2$                                           | 0.939   |
| Pseudo-Second-Order | $q_e$ (mg/g)                                    | 252.542 |
|                     | $k_2$ ( $\text{g}/(\text{mg}\cdot\text{min})$ ) | 0.003   |
|                     | $R^2$                                           | 0.936   |

For adsorbents, the initial concentration of the adsorbed substance often affects the adsorption rate and efficiency. Therefore, the effect of initial  $\text{Sr}^{2+}$  concentration on the adsorption efficiency of IIGK@MnO<sub>2</sub> was further studied. According to Figure 5d, the adsorption efficiency of IIGK@MnO<sub>2</sub> for  $\text{Sr}^{2+}$  shows a trend of first increasing and then decreasing with the increase in initial  $\text{Sr}^{2+}$  concentration. The maximum adsorption efficiency occurs when the  $\text{Sr}^{2+}$  concentration is  $25 \text{ mg}\cdot\text{L}^{-1}$ . When the initial  $\text{Sr}^{2+}$  concentration reaches  $40 \text{ mg}\cdot\text{L}^{-1}$ , the adsorption efficiency sharply decreases. When the  $\text{Sr}^{2+}$  concentration increases to  $60 \text{ mg}\cdot\text{L}^{-1}$ , the adsorption efficiency tends to stabilize. The adsorption isotherms of  $\text{Sr}^{2+}$  on IIGK@MnO<sub>2</sub> were studied using Freundlich and Langmuir isotherm models. The model is shown as follows [34]:

$$q_e = \frac{q_m b C_e}{1 + b C_e} \quad (7)$$

$$q_e = K_f C_e^{\frac{1}{n}} \quad (8)$$

where  $q_m$  (mg/g) represents the maximum adsorption capacity at the isotherm temperature,  $b$  (L/mg) is the Langmuir constant associated with the free energy or net enthalpy of adsorption,  $C_e$  (mg/L) and  $q_e$  (mg/g) denote the concentration of the adsorbate in the equilibrated solution and the amount adsorbed on the adsorbent, respectively,  $K_f$  and  $n$  are equilibrium constants that provide information about the adsorption capacity and intensity. Shown in Table 2 is the fitting effect of equilibrium data, and the Langmuir isotherm model ( $R^2 = 0.991$ ) is better than that of the Freundlich model ( $R^2 = 0.982$ ). Furthermore, the  $\text{Sr}^{2+}$  adsorption performance of the IIGK@MnO<sub>2</sub> nanocomposite was compared with that of other reported materials (Table 3). Compared to other adsorbents, IIGK@MnO<sub>2</sub> showed better performance in the aspects of higher adsorption capacity (748.2 mg/g), which is beneficial for the treatment of nuclear wastewater. Therefore, the adsorbent surface is considered homogeneous and monolayer in terms of adsorption energy [35]. Compared with pure MnO<sub>2</sub>, IIGK@MnO<sub>2</sub> composites exhibit significantly stronger adsorption efficiency for  $\text{Sr}^{2+}$  (Figure 5e). As previously speculated, after combining IIGK peptide with MnO<sub>2</sub>, the obtained IIGK@MnO<sub>2</sub> composites contain more adsorption active sites due to their high dispersion, large specific surface area, and high negative charge, which is beneficial for the adsorption of  $\text{Sr}^{2+}$ .

**Table 2.** The parameters calculated from the Langmuir and Freundlich for  $\text{Sr}^{2+}$  adsorption by the IIGK@MnO<sub>2</sub> nanocomposite.

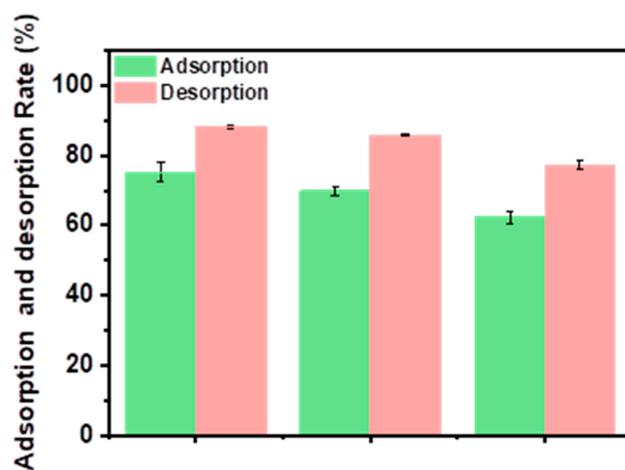
| Models              |              | Parameters |       |
|---------------------|--------------|------------|-------|
| Langmuir isotherm   | $q_m$ (mg/g) | $b$ (L/mg) | $R^2$ |
|                     | 748.193      | 0.015      | 0.991 |
| Freundlich isotherm | $k$          | $1/n$      | $R^2$ |
|                     | 33.542       | 0.55       | 0.982 |

**Table 3.** Comparison of the adsorption performance of IIGK@MnO<sub>2</sub> nanocomposite and other adsorbents for removal of  $\text{Sr}^{2+}$ .

| Adsorbent                        | Maximum Adsorption Capacity (mg/g) | Optimal pH | Equilibrium Time (min) | Reference  |
|----------------------------------|------------------------------------|------------|------------------------|------------|
| IIGK@MnO <sub>2</sub>            | 748.193                            | 6          | 360                    | This study |
| bentonite                        | 63.01                              | -          | -                      | [36]       |
| zeolite 4A                       | 252.5                              | 4–9        | 5                      | [37]       |
| K <sub>2</sub> SbPO <sub>6</sub> | 175.9                              | 3          | 1440                   | [38]       |
| SBA-15                           | 17.67                              | 10         | 100                    | [39]       |
| A-ZrP                            | 300                                | 3–11       | 240                    | [40]       |
| NaTS                             | 80.0                               | 5          | 0.083                  | [10]       |
| SZ-6                             | 61.4                               | 10         | 50                     | [41]       |
| MnO <sub>2</sub>                 | 53.5                               | 10         | 0.25                   | [24]       |

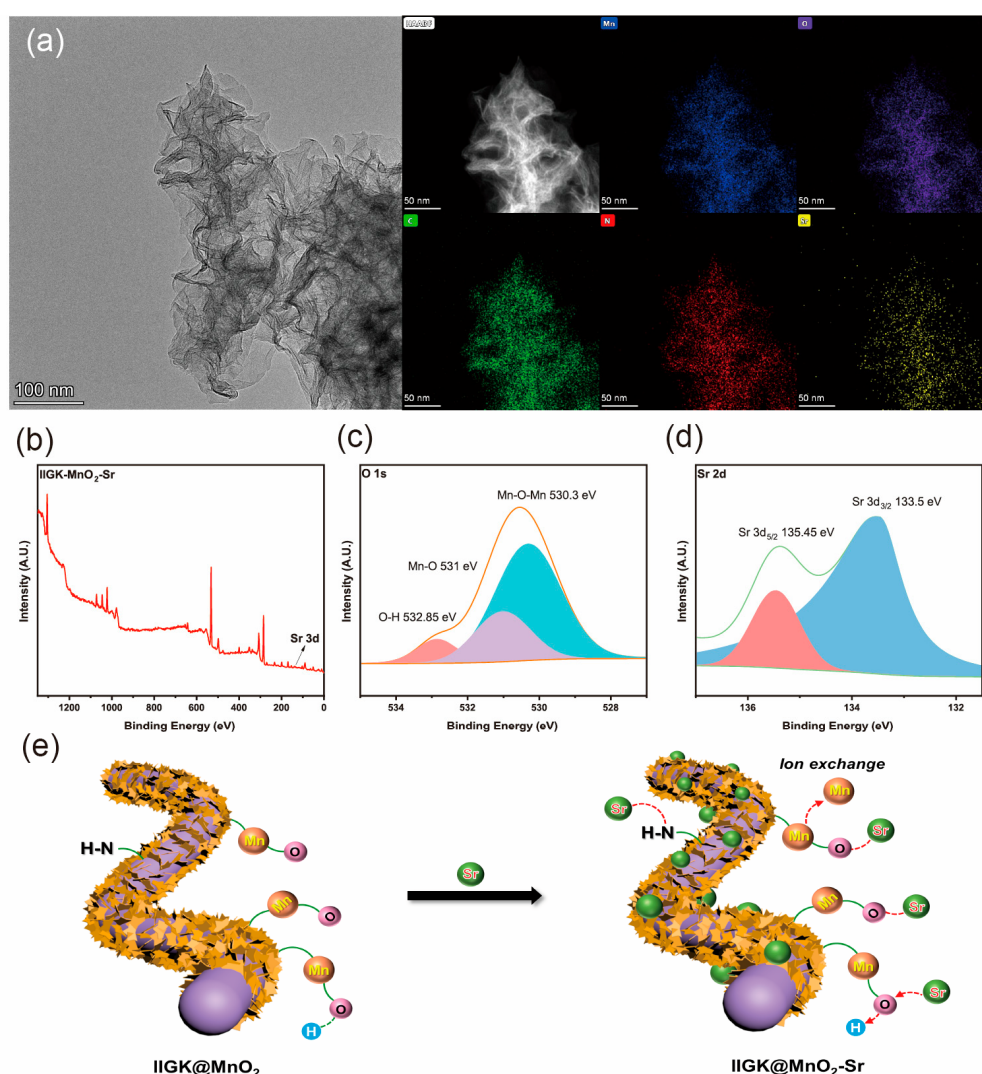
### 3.3. Desorption and Reusability Studies

When evaluating the economy and applicability of an adsorbent, the reusability is an important indicator that plays a crucial role in the practical application of adsorbents. Herein, the adsorption/desorption process was repeated to assess the reusability of IIGK@MnO<sub>2</sub>. Firstly, IIGK@MnO<sub>2</sub> was used to adsorb  $\text{Sr}^{2+}$ , and then the IIGK@MnO<sub>2</sub>- $\text{Sr}^{2+}$  composite was treated with 1M HCl solution for  $\text{Sr}^{2+}$  desorption. The whole adsorption/desorption process was repeated three times. As shown in Figure 6, when the regeneration cycle is repeated three times, the adsorption efficiency of IIGK@MnO<sub>2</sub> for  $\text{Sr}^{2+}$  is still higher than 64%, indicating that 1M HCl solution can effectively desorb  $\text{Sr}^{2+}$  from IIGK@MnO<sub>2</sub>. Moreover, the adsorption capacity of IIGK@MnO<sub>2</sub> for  $\text{Sr}^{2+}$  decreased with the increase in cycling number, which may be due to  $\text{H}^+$  occupying some adsorption sites. However, it can also be noted that the adsorption efficiency of IIGK@MnO<sub>2</sub> for  $\text{Sr}^{2+}$  can still reach over 60% at the end of three cycles, indicating that IIGK@MnO<sub>2</sub> has good reusability.

**Figure 6.** Reusability of the IIGK@MnO<sub>2</sub> nanocomposite during three cycles of  $\text{Sr}^{2+}$  adsorption and desorption.

### 3.4. Adsorption Mechanism of $\text{Sr}^{2+}$ by the IIGK@ $\text{MnO}_2$ Nanocomposite

The adsorption mechanism of IIGK@ $\text{MnO}_2$  for  $\text{Sr}^{2+}$  was studied. Figure 7a shows the morphology and distribution of various elements of IIGK@ $\text{MnO}_2$  after the adsorption of  $\text{Sr}^{2+}$ . After adsorbing  $\text{Sr}^{2+}$ , there was no significant change in the morphology of IIGK@ $\text{MnO}_2$ . The mapping of IIGK@ $\text{MnO}_2$ -Sr elements shows that the adsorbed  $\text{Sr}^{2+}$  is evenly distributed on the surface of IIGK@ $\text{MnO}_2$ , indicating that the adsorption sites on IIGK@ $\text{MnO}_2$  are relatively uniform. XPS data confirmed the presence of strontium on IIGK@ $\text{MnO}_2$  after adsorption (Figure 7b). The deconvolution peaks located at 133.5 eV and 135.4 eV belong to Sr 3d 5/2 and Sr 3d 3/2, respectively (Figure 7c) [10]. There is a shift in binding energy in the high-resolution spectrum of O1s in Figure 7d. The binding energy of the O1s-Mn-OH peak decreased from 531.4 eV to 531.0 eV, and the binding energy of the O 1s O-H peak decreased from 533.1 eV to 532.8 eV. This result indicates that oxygen atoms interact with  $\text{Sr}^{2+}$  and play a dominant role in the  $\text{Sr}^{2+}$  adsorption process.



**Figure 7.** (a) TEM image and EDS element-mapping of IIGK@ $\text{MnO}_2$ -Sr composite. (b) XPS full survey spectra of the IIGK@ $\text{MnO}_2$ -Sr nanocomposite. (c,d) XPS fine spectra of O 1s and Sr 3d of the IIGK@ $\text{MnO}_2$ -Sr nanocomposite. (e) Schematic diagram of the adsorption mechanism of  $\text{Sr}^{2+}$  by IIGK@ $\text{MnO}_2$ .

From the above results, the mechanism of  $\text{Sr}^{2+}$  adsorption by IIGK@ $\text{MnO}_2$  can be proposed, as schemed in Figure 7e. On the one hand, the peptide IIGK can bind with  $\text{Sr}^{2+}$  through coordination or electrostatic interactions. On the other hand, the  $\text{MnO}_2$

nanoparticles on the IIGK fibrils' surface have many absorption sites, which can adsorb  $\text{Sr}^{2+}$  by either coordination interaction or ion exchange reaction. Therefore, IIGK and  $\text{MnO}_2$  work synergistically to enhance the removal efficiency of the IIGK@ $\text{MnO}_2$  nanocomposites.

#### 4. Conclusions

In this work, the IIGK@ $\text{MnO}_2$  nanocomposites were synthesized using environmentally friendly and mild methods. Using the short peptide IIGK as a template leads to the formation of a one-dimensional fibrous structure of IIGK@ $\text{MnO}_2$  nanocomposite, which not only gives the mineralized IIGK@ $\text{MnO}_2$  complex a large specific surface area but also makes the overall IIGK@ $\text{MnO}_2$  negatively charged. These unique properties are conducive to the adsorption and removal of  $\text{Sr}^{2+}$  from wastewater. According to the adsorption performance of IIGK@ $\text{MnO}_2$  towards  $\text{Sr}^{2+}$ , it can be observed that the IIGK@ $\text{MnO}_2$  hybrid adsorbents possess excellent  $\text{Sr}^{2+}$  adsorption effects in a large range of temperature and pH values and show much higher  $\text{Sr}^{2+}$  adsorption efficiency than pure  $\text{MnO}_2$  nanoparticles. In addition, IIGK@ $\text{MnO}_2$  also exhibits good reusability, achieving a  $\text{Sr}^{2+}$  adsorption efficiency of over 60% after three cycles of adsorption–desorption processes. By analyzing the element distribution and valence states of IIGK@ $\text{MnO}_2$ -Sr after the adsorbing process, the mechanism of  $\text{Sr}^{2+}$  adsorption was proposed. The interaction between N-H groups on IIGK and oxygen atoms on  $\text{MnO}_2$  with  $\text{Sr}^{2+}$  may play an important role in the adsorption of strontium ions. In conclusion, the designed IIGK@ $\text{MnO}_2$  nanocomposite in this work exhibits excellent adsorption performance towards  $\text{Sr}^{2+}$  and will shed light on the construction of organic–inorganic hybrid adsorbents with multiple active adsorption sites and high adsorption efficiency for adsorbing radioactive ions in wastewater.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/nano14010052/s1>, Figure S1. FTIR spectra of IIGK@ $\text{MnO}_2$  and  $\text{MnO}_2$ .

**Author Contributions:** Conceptualization, M.C. and W.W.; software, X.L., Z.L., X.W. and H.M.; resources, M.C. and W.W.; writing—original draft preparation, X.L., Z.L., X.W. and H.M.; writing—review and editing, H.M. and M.C.; supervision, M.C.; project administration, M.C.; funding acquisition, W.W. and M.C. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the National Natural Science Foundation of China (22172194, 21972167, 21872173).

**Data Availability Statement:** All data generated or analyzed during this study are contained within the article and Supplementary Materials.

**Conflicts of Interest:** The authors declare no conflict of interest.

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