

Research Article

Mechanism of Permanganate Biosorption by Fermentation Biowaste

Namgyu Kim,¹ Sang-Hoon Lee,² Hanui Yang,³ and Donghee Park³ 

¹Environment Solution Research 2 Team, GS Engineering & Construction Corp., 33 Jong-ro, Jongno-gu, Seoul 03159, Republic of Korea

²Water Environmental Engineering Research Division, National Institute of Environmental Research, Incheon 22689, Republic of Korea

³Department of Environmental and Energy Engineering, Yonsei University, 1 Yonseidae-gil, Wonju 26493, Republic of Korea

Correspondence should be addressed to Donghee Park; dpark@yonsei.ac.kr

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Biosorptive treatment of industrial wastewater contaminated with heavy metals has been recognized as one of the most effective green tools, competing with traditional physical/chemical treatment processes. This study delves into the detailed investigation of the biosorbent prepared from fermentation biowaste, particularly focusing on the mechanism behind permanganate (Mn(VII)) removal. Various parameters including pH, biosorbent dosage, initial concentration, and temperature were examined. Among these factors, solution pH emerged as the most crucial in removing Mn(VII) using the biosorbent. The significant removal of Mn(VII) was attributed to both reduction and adsorption, as confirmed by X-ray photoelectron spectroscopy. Depending on the experimental conditions, the removal of Mn(VII) was influenced not only by simple adsorption but also by oxidation-reduction and precipitation processes. This study not only advances our understanding of biosorptive treatment but also highlights the promising potential of fermentation biowaste-based biosorbents for effective Mn(VII) removal.

1. Introduction

Water pollution is a significant global environmental challenge. Wastewater often carries heavy metals, which, even at low concentrations, pose health risks to humans and ecosystems due to their high toxicity and persistence in the environment [1–3]. Heavy metals, such as lead, cobalt, copper, and manganese, can cause serious health effects on both flora and fauna [4–6]. Manganese (Mn) is present in industrial wastewater but also occurs naturally. It is sourced from the Earth's crust and can be found in the atmosphere, soil, and water [7, 8]. Although less toxic than other heavy metals in water, manganese is typically removed for aesthetic reasons and to address potential distribution system issues [9]. Precipitates of oxidized manganese can obstruct water pipes, leading to increased head loss, transportation costs, and the proliferation of bacteria, which can alter the taste and odor of water [10]. These issues arise when soluble manganese concentrations exceed 0.02 mg/L. Consequently, the

EPA has established a secondary maximum contaminant level of 0.05 mg/L for manganese, based on considerations of staining and taste, which influence consumer acceptance of water quality [11]. Furthermore, most countries enforce legal regulations on manganese to safeguard against staining and the metallic taste in water.

In recent times, a range of techniques have been explored for the removal of Mn, encompassing oxidation, precipitation, (ad)sorption, ion exchange, membrane, and biological approaches [12–14]. However, certain methods render the efficient removal of Mn from wastewater financially burdensome. For instance, precipitation fails to completely eliminate manganese and may lead to secondary pollution. Ion exchange, membrane filtration, and biological methods can incur significant costs, particularly when treating large volumes of wastewater with low Mn concentrations [15]. These limitations have spurred research efforts to develop more effective processes for manganese removal. In recent years, biosorption has emerged as a promising alternative to

traditional physical and chemical treatments. Biosorption, utilizing inactive and nonliving microbial biomass as adsorbents, stands out for its cost-effectiveness and superior performance compared to other adsorbents [16–18]. Most studies on manganese biosorption demonstrate that Mn(II) can be effectively removed from the aqueous phase through an adsorption mechanism, wherein cationic Mn(II) ions bind to negatively charged groups present in biomaterials. It is worth noting that manganese exists in a diverse range of oxidation states, with the most common being +2, +3, +4, +6, and +7, although states ranging from –3 to +7 have been observed [19]. Manganese was found to have widespread application as a transition metal in various industrial alloys, particularly in the production of stainless steels. Notably, in aqueous systems, permanganate (Mn(VII)) serves as a widely utilized oxidizing agent, extensively employed in organic synthesis, sterilization, disinfection, and bleaching processes [20, 21]. Furthermore, Mn(VII) predominantly exists as an anion species in aqueous environments [22]. Due to its specific chemical nature, it cannot be effectively removed through a general adsorption mechanism; however, a significant portion can be eliminated through redox processes [23].

To the best of our knowledge, no prior studies have offered a comprehensive understanding of the precise mechanism behind Mn(VII) removal by biosorbents. In this investigation, we employed a biosorbent designed to target anionic species, effectively binding Mn(VII) in its anionic form within aqueous systems. The biosorbent used in this study was fabricated from fermentation biowaste, as detailed in previous work [24, 25]. Notably, this biosorbent has been modified with polyethylenimine (PEI), possessing numerous amine groups, which significantly enhances its performance in anion adsorption. In order to elucidate the mechanism of Mn(VII) removal, we conducted a systematic exploration of the influence of key factors including pH, initial Mn(VII) concentrations, biosorbent dosage, and temperature.

2. Materials and Methods

2.1. Materials. Fermentation biowaste was obtained from Daesang Co. in a slurry with a moisture content of 85%. Chitosan (Samchun, Korea), PEI (Habjung Moolsan, Korea), and glutaraldehyde (Junsei, Japan) were purchased to immobilize and cross-link the bacterial biomass biosorbents, respectively. A pure analytical-grade Mn(VII) solution was prepared by dissolving KMnO_4 (Samchun, Korea) in distilled water. The pH was adjusted by adding NaOH (Samchun, Korea) or H_2SO_4 (Samchun, Korea). All other reagents were of analytical grade.

2.2. Preparation of Biosorbent. The fermentation biowaste utilized in the experiment consisted of *Corynebacterium glutamicum*, a widely employed organism in biotechnological amino acid production. To create the biosorbent, chitosan served as a binder to immobilize the fermentation biowaste. This process involved mixing 5 g of chitosan with 130 mL of biowaste in a 5% (v/v) acetic acid solution. The mixture was thoroughly stirred until achieving homogeneity. Subsequently, the mixture was extruded through a spinneret,

and the resulting fibers were allowed to immerse in a 2.0 mol/L NaOH solution for 24 hours. Following this, the biosorbent was suspended with 3 g of PEI in 100 mL of distilled water for 6 hours. Then, a 0.6% (v/v) glutaraldehyde solution was introduced as a cross-linker while being stirred for 2 hours. For a more comprehensive procedure, please refer to the previous study [24]. Ultimately, PEI-coated biosorbent fibers measuring 0.2 to 0.5 mm in thickness and 5 to 10 mm in length were employed.

2.3. Adsorption Studies. The removal efficiency of Mn(VII) by the biosorbent was assessed by monitoring the time-dependent concentrations of Mn(VII) and total manganese in a batch system. Test solutions were prepared by dissolving precise quantities of analytical-grade KMnO_4 in deionized and distilled water. Batch experiments were conducted using 230 mL plastic bottles with a working volume of 200 mL. These bottles were horizontally agitated on a shaker at 200 rpm for a duration of 6 hours. To investigate the influence of pH, the pH of the solutions was varied within the range of 2.0 to 6.0. The biosorbent dosage was maintained at 1.0 g/L. Temperature effects were examined across a range of 10 to 40°C. To assess the impact of biomass concentration, biomass concentrations of 0.5, 1.0, 1.5, and 2.0 g/L were employed. For initial Mn(VII) concentrations, values of 50, 100, 150, and 200 mg/L were used, all at a pH of 3.0 and room temperature (20°C). Solution pH adjustments were made by adding H_2SO_4 or NaOH (0.1 mol/L or 1.0 mol/L). It is worth noting that the change in working volume resulting from the addition of H_2SO_4 or NaOH was negligible.

2.4. X-Ray Photoelectron Spectroscopy Analysis. The valence states of Mn bound to the biosorbent were analyzed using X-ray photoelectron spectroscopy (XPS) from Thermo Scientific Inc., UK, equipped with a K-alpha and an Al Ka μ -focused monochromator (1486.6 eV). Manganese-laden biosorbent was generated by exposing it to 100 mg/L of Mn(VII) at pH levels of 2 and 4 for 24 hours. Before mounting for XPS analysis, the biosorbent underwent several washes with deionized and distilled water, followed by freeze-drying. For reference, $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ (Samchun, Korea) and KMnO_4 (Samchun, Korea) were employed as Mn(II) and Mn(VII) compounds, respectively.

2.5. Analysis of Metal Ions. The pink color of Mn(VII) was assessed at 525 nm using a spectrophotometer (Optizen 1412 V, Mecasys Co., Ltd., Korea) for concentration measurement. Total manganese was analyzed using an inductively coupled plasma-optical emission spectrometer (ICP/IRIS, Thermo Jarrell Ash Co., USA) after filtration through a 0.20 μm membrane.

3. Results and Discussion

3.1. Effect of pH on Mn(VII) Removal by the Biosorbent. Solution pH was the most influential factor compared with temperature, biosorbent dosage, and the quantity of target removed. The pH affects the solution chemistry of a metal itself, the activity of functional groups on the biosorbent, and the competition with coexisting ions in solution. Some

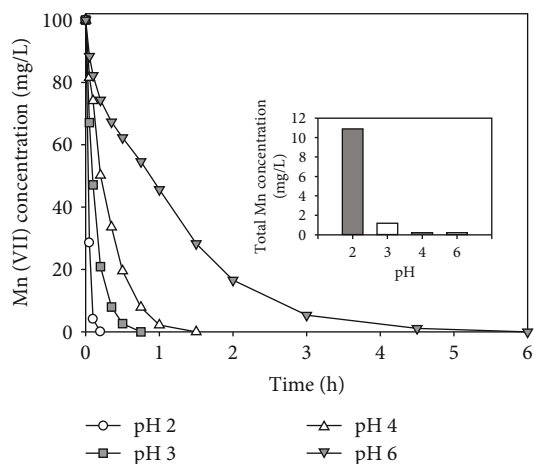
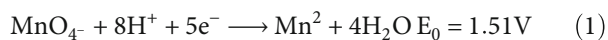


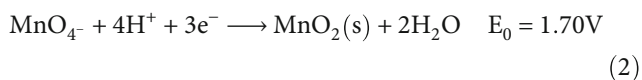
FIGURE 1: Dynamics of Mn(VII) removal by the biosorbent at various pH values.

functional groups (amide, amine, carboxylate, and hydroxyl groups) are known as dependable options for binding metals in biosorbents. Among these, the amine and hydroxyl groups are relatively effective in removing anionic metal species through electrostatic interactions or hydrogen bonding. The biosorbent used in this study has abundant amine and hydroxyl groups due to the presence of chitosan and PEL, which were used for its manufacture. For this reason, the biosorbent is an effective option for anion adsorption, as reported previously [24–26]. During anion adsorption, the adsorption rate and uptake amount increased as solution pH decreased, because amine and hydroxyl groups can be easily protonated with positive charges at a low pH [27]. As shown Figure 1, the Mn(VII) removal rate also increased with decreasing pH. However, unlike general adsorption kinetic behavior, Mn(VII) was completely removed in all experiments, even at a relatively high pH after an extended period. The strong influence of pH on Mn(VII) species suggests that it is a critical factor. Manganese's various oxidation states (Mn^{6+} , Mn^{5+} , Mn^{4+} , Mn^{3+} , and Mn^{2+}) depend on the pH of the aqueous system. The following equations show the oxidation states of manganese according to pH.

In strong acid solution,



In acidic-neutral pH,



Mn(VII) is reduced to a colorless +2 oxidation state as an Mn(II) (Mn^{2+}) ion in a strong acid (Eq. (1)). Total manganese concentrations were high at a low pH (Figure 1). These results indicate that some of the Mn(VII) was reduced to Mn(II) when brought into contact with the biosorbent at a low pH. Mn(VII) is a heavy metal that exists as an anion in water. At the same time, Mn(VII) was bound to positively charged groups in the biosorbent. This adsorption mecha-

nism was observed at all pH values. After adsorption and until the Mn(VII) was removed below the lower limit of detection, the concentration of the total Mn gradually decreased as the pH increased, and only low concentrations of total Mn remained in a solution of pH 4. According to Eq. (2), Mn(VII) is reduced to $\text{MnO}_2(\text{s})$ in the form of a precipitate at a neutral pH during adsorption. Manganese ions were present at the lower limit of detection as most were removed through filtering before ICP analysis.

3.2. Mn(VII) Removal Mechanism. To examine the Mn(VII) removal mechanism onto the biosorbent, Mn(VII) and total manganese concentration profiles were investigated at a pH of 2 and 4, respectively (Figure 2). The concentration of total manganese in an aqueous phase sharply decreased for 0.2 h and then gradually increased. First, the concentration of Mn(VII) and the concentration of total Mn were almost the same until 0.1 hour into the adsorption process. This indicates that Mn(VII) anions were bound first by the positively charged groups, such as the ammonium groups and the protonated amino groups on the biosorbent surface, via electrostatic attraction. After 0.1 h, a difference arose between the total manganese concentration and Mn(VII) concentrations as the Mn(VII) was removed not only by adsorption but also through reduction. During adsorption, the concentration of total manganese increased, which indicates that small amounts adsorbed on the biosorbent were released into the aqueous phase. Some release of manganese ions from the biosorbent was due to breakdown of the biosorbent itself at a low pH. According to previous reports, this biosorbent exhibits poor acid resistance [28]. Manganese released again after adsorption was reduced to Mn(II) and remained in the aqueous solution, increasing the concentration of total manganese. For this reason, the biosorption mechanism of anionic Mn(VII) is known to be an adsorption-coupled reduction at low pH condition. However, the removal behavior of Mn(VII) and the total manganese removal were the same in the case of pH 4. Mn(VII) was initially removed because MnO_4^- was adsorbed quickly on the biosorbent at a pH of 4. After 0.3 h, the removal rate was slower than before because most of the Mn(VII) was removed by precipitation with MnO_2 . The removal of manganese in high pH (>4) conditions was accompanied by a combination of reactions, including oxidation-reduction, adsorption, and precipitation.

Through additional surface analysis, the removal mechanism of Mn(VII) by the biosorbent was observed in more detail. Analysis by XPS was employed to further investigate the valence state of the manganese bound on the biosorbent surface before and after adsorption. Low-resolution XPS spectra of the manganese-unloaded biosorbent indicated that no other elements associated with biosorbent surfaces other than carbon, nitrogen, and oxygen made significant contributions (data not shown). High-resolution spectra of the Mn-laden biosorbent indicated that significant contributions were made by the manganese bound on the biosorbent (Figure 3). According to previous reports, significant manganese $2\text{p}_{3/2}$ peaks appear at binding energies of 640.6–642.4 eV [29]. For accurate analysis, the Mn(II) and Mn(VII)

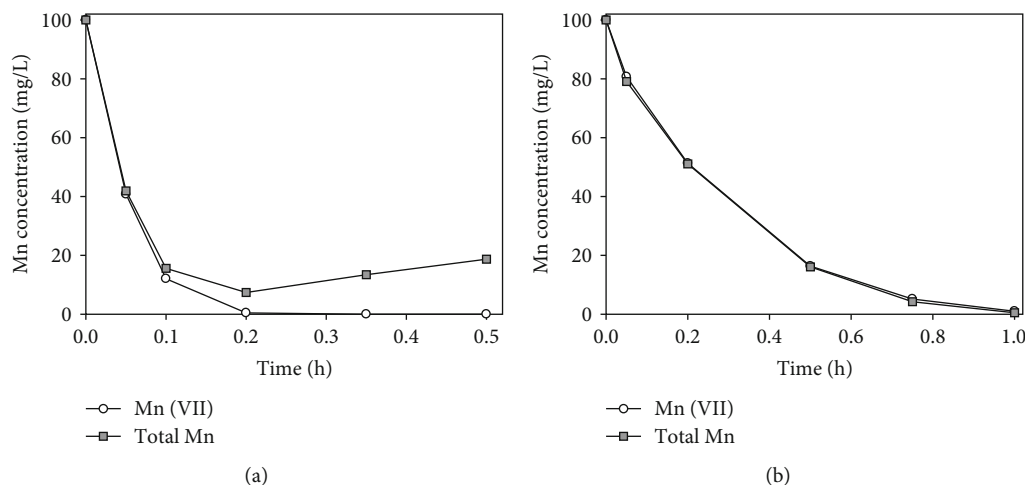


FIGURE 2: Mn concentration profiles during Mn(VII) removal at (a) pH 2.0 and (b) pH 4.0.

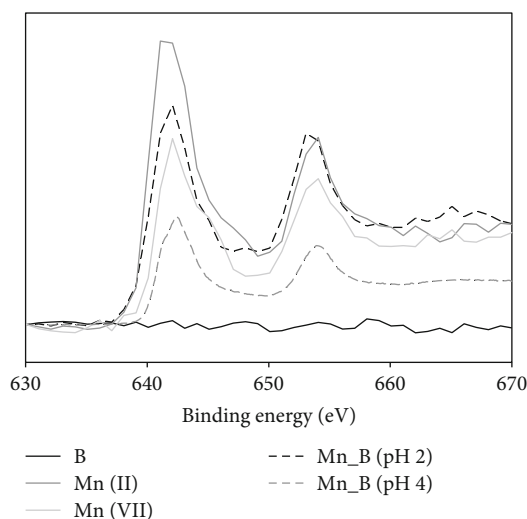
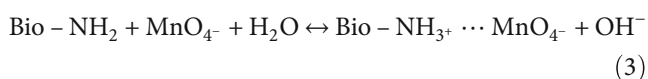
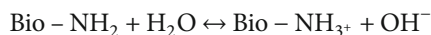


FIGURE 3: XPS spectra of biosorbent (B), the Mn-laden biosorbent (Mn_B), and standard Mn chemicals.

reference materials were compared with the biosorbent and adsorbed biosorbent at different pH values (2.0 and 4.0). Peak analysis confirmed that Mn(VII) was attached at both pH 2 and pH 4. Through a different anion adsorption mechanism in the biosorbent, Mn(VII) removal also occurred directly through Mn(VII) anions binding onto the positively charged surface moieties of the biosorbents involving electrostatic interaction in both pH conditions.



As mentioned above, manganese was removed by adsorption simultaneously with oxidation-reduction at pH 2.

3.3. Effect of Biosorbent Dosage and Initial Mn(VII) Concentration on Mn(VII) Removal. Four different biosor-

ben doses ranging from 0.5 to 2.0 g/L were employed to investigate the influence of dose on adsorption capacity. It was observed that both Mn(VII) and total Mn were effectively removed across all dose concentrations, with the highest removal rate observed at a high dose and a pH of 3 (Table 1). Additionally, it was noted that the removal time of Mn(VII) extended with an increase in biosorbent dose (Figure 4). This phenomenon can be attributed to the augmentation of adsorbent surface area and the availability of active sites on the biosorbent surface [30]. However, upon comparing the time required for the removal of all Mn(VII) with the removal rate, it was observed that the rate of increase did not proportionally escalate at concentrations exceeding 1.5 g/L. This discrepancy suggests that the rate of adsorption significantly diminished as contact time increased, indicating that the solution had reached a steady state.

While initial concentration experiments are commonly employed to investigate the adsorbent isotherm, in the case of Mn(VII) removal, the primary mechanism involves redox and precipitation. Consequently, at high concentrations, Mn(VII) was entirely eliminated, rendering the determination of an isotherm unattainable. For this reason, we examined the concentration of Mn(VII) over time at various initial concentrations, ranging from 50 to 200 mg/L (Figure 5). Notably, the Mn(VII) removal rate exhibited an increase in correlation with the initial Mn(VII) concentration. However, it is worth noting that the removal rate did not show a proportional increase at concentrations exceeding 150 mg/L.

3.4. Effect of Temperature on Mn(VII) Removal. Figure 6 illustrates the impact of temperature on the removal of Mn(VII) by the biosorbent over time, demonstrating complete removal within the temperature range of 283 to 313 K. With rising temperature, the Mn(VII) removal rate exhibited a gradual increase (small figure in Figure 6). It is important to note that the effect of temperature on adsorption differs depending on whether it involves physisorption or chemisorption. In this experiment, the adsorbent, as

TABLE 1: Final total Mn concentration at various adsorption conditions (temperature, dosage, and initial concentration) after an equilibrium state.

	Temperature (K)	Dosage (g/L)	Initial Mn(VII) concentration (mg/L)	Final total Mn concentration (mg/L)
Effect of dosage	293	0.5	100	1.43
	293	1.0	100	1.19
	293	1.5	100	0.59
	293	2.0	100	N.D
Effect of initial Mn(VII) concentration	293	1.0	50	0.98
	293	1.0	100	1.193
	293	1.0	200	3.091
	293	1.0	300	3.567
Effect of temperature	283	1.0	100	1.64
	293	1.0	100	1.14
	303	1.0	100	2.73
	313	1.0	100	4.60

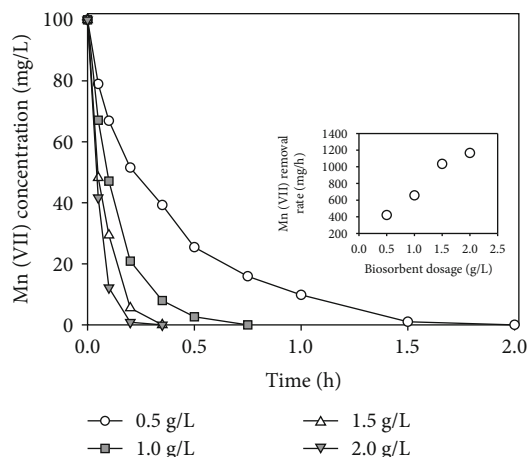


FIGURE 4: Effect of biosorbent dosage on Mn(VII) removal.

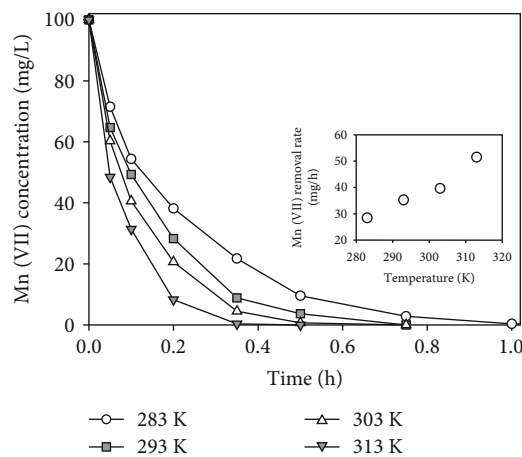


FIGURE 6: Effect of temperature on Mn(VII) removal.

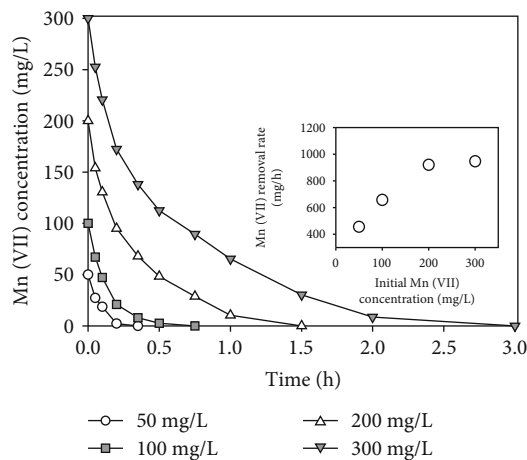


FIGURE 5: Effect of initial Mn(VII) concentration on Mn(VII) removal.

determined by BET measurement, lacks an external surface, indicating that the primary adsorption mechanism is chemisorption rather than physisorption. Chemisorption tends to occur more readily at higher temperatures and increases as the temperature rises [31]. This observation suggests that the removal of Mn(VII) by the biosorbent entails an endothermic reaction.

4. Conclusion

The biosorbent prepared from fermentation biowaste was successfully used to remove the anionic Mn(VII) from aqueous solutions. Solution pH was the most important factor determining Mn(VII) removal using the biosorbent. Based on the experimental results, we proposed an adsorption-coupled reduction mechanism for Mn(VII) removal at a low pH (≤ 2). Removal of Mn(VII) at a high pH (≥ 4) is accompanied by a combination of oxidation-reduction, adsorption, and precipitation. Therefore, understanding the

exact mechanism of Mn(VII) removal is more important than simply determining the amount of removal.

Data Availability

All data generated or analyzed during this study are included in this published article.

Conflicts of Interest

All authors declare that the authors have no competing interests as defined by Springer or other interests that might be perceived to influence the results and/or discussion reported in this paper.

Authors' Contributions

Namgyu Kim participated in formal analysis, investigation, and data curation and wrote the original draft. Sang-Hoon Lee contributed to the methodology and reviewed and edited the manuscript. Hanui Yang participated in formal analysis and contributed to the methodology. Donghee Park contributed to the methodology, conceptualized and supervised the study, and reviewed and edited the manuscript.

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