**Optimum parameters for humic acid removal and power production by Al–air fuel cell electrocoagulation in synthetic wastewater**

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**ABSTRACT**

Although humic acid (HA) is a complex natural organic matter, it can potentially harm the environment and human health. In this study, aluminum–air fuel cell electrocoagulation (AAFCEC) was used to remove HAs from water while generating electricity. Initial pH, electrolyte concentration, HA concentration electrode distance and external resistance were investigated to determine the power generation and removal efficiency. The results showed that the better performance of power generation has been acquired in the alkaline solution and larger electrolyte concentration and short electrode distance. Further, Al–Ferron complexation timed spectrophotometry was used to determine the Al speciation distribution in the solution under different parameters. The power density of the cell reached 313.47 mW/cm² for the following conditions: 1 g/L NaCl concentration, 3 cm electrode distance, 20 Ω external resistor, and pH 9. After about an hour electrolysis, the optimum removal rate of HA was above 99%. The results demonstrated that the AAFCEC is an efficient and eco-friendly water treatment process, and it could be further developed and disseminated in the rural areas and households.

**Key words:** aluminum–air fuel cell electrocoagulation, humic acid, power generation, wastewater treatment

**HIGHLIGHTS**

- This Al-air fuel cell electrocoagulation can be used to treatment wastewater without external power source.
- The output power density can reach up to 427.59 mW/cm².
- The form of Al in the solution have been studied by Al-Ferron complexation timed spectrophotometry.
- The removal rate of HA was above 99% after about an hour electrolysis.

**1. INTRODUCTION**

Humic acids (HAs) are complex natural organic matter formed from the remains of various plants and animals by anaerobic respiration of microorganisms in water, soil, and other environments (Barhoumi et al. 2019; Son et al. 2019). HAs exist in large quantities in water, accounting for 40–90% of the soluble organic pollutants in water (Soleimani et al. 2019). The main HA components are carbon, hydrogen, oxygen, nitrogen, and a small amount of phosphorus, sulfur, and other elements. HA has no fixed molecular structure. It is generally believed that the core structure of HA consists of aliphatic and aromatic parts, surrounded by various functional groups, such as hydroxyls, phenols, carboxyls, and ketones (García et al. 2020; Liu et al. 2020; Xie et al. 2020). These functional groups can adsorb organics and heavy metal ions in the water to form chelates that are difficult to remove (Zhang et al. 2020; Zhou et al. 2021). A large amount of HA can significantly affect the color, smell, and taste of water. In addition, HA in drinking water can form trihalomethanes, haloacetic acids, and other disinfection byproducts during the disinfection process, and these substances can potentially endanger human health (Wongrueng et al. 2018; Song et al. 2019; Zhao et al. 2020). Therefore, HAs should be removed from the water to reduce its potential harm to the environment and human body.

There are several techniques to remove HAs from water, including adsorption (Derakhshani & Naghizadeh 2018; Jampa et al. 2020), membrane filtration (Hwang et al. 2013), advanced oxidation (Yin et al. 2020), ion exchange (Wang et al. 2009),
and coagulation (Xu et al. 2016; Saxena et al. 2019). However, these methods have some disadvantages, such as long processing time, high processing cost, difficult process control, and secondary pollution. Considering these problems, a more effective removal method is needed. According to previous research (Kac et al. 2017; Ghenemy et al. 2020), electrocoagulation (EC) is a technology that can effectively remove HA. EC is a simple and effective electrochemical process in which coagulants are produced by the electrolysis of metal anodes. As an eco-friendly water treatment technology, EC is widely used to remove various pollutants from water bodies, such as arsenic (Sik et al. 2017; Mohor et al. 2018), chromium (Martín-Domínguez et al. 2018), cadmium (Vasudevan et al. 2011), phosphate (Dura & Breslin 2019), nitric acid salts (Berkani et al. 2019), dyes (Eyvaz et al. 2009), fluorides (Silva et al. 2018), and chemical oxygen demand (Elazzouzi et al. 2017). Compared with traditional chemical coagulation, EC can generate coagulants through the electrolysis of metal anodes without the addition of chemicals. EC has the advantages of simple equipment requirement, convenient operation, easy control, and short running time. However, the operation of EC requires a large amount of electrical energy, which limits its development. Therefore, it is necessary to reduce the power consumption of EC, thereby facilitating the application of this technology.

To reduce the EC power consumption, this study combines the concepts of metal–air fuel cells and electrocoagulation to design an aluminum–air fuel cell electrocoagulation (AAFCEC) equipment. The device, which consists of an aluminum anode and an air cathode, can generate electricity from the anodic oxidation of aluminum without an external power source, and it can also produce an aluminum coagulant. As a new type of energy cell, the aluminum–air fuel cell has the advantages of high specific energy and specific power, less harm to the environment, simple cell structure, safety, and reliability (Katsoufis et al. 2020; Wu et al. 2020a). The aluminum plate is used as the electrode because aluminum has advantages such as abundant reserves, low price, and nontoxicity (Wu et al. 2020b). In addition, the aluminum salt produced in the reaction is an effective coagulant and can potentially be used to remove pollutants from water. Aluminum–air batteries can generate electricity because of the low oxidation potential of aluminum and the relatively high reduction potential of oxygen. There is a potential difference between the two electrodes, which causes the electrons to continuously flow from negative to positive, thereby forming a continuous current. The reaction formula is shown in (1)–(3) (Goel et al. 2020).

\[
\text{Anode: } \text{Al}(s) + 3\text{OH}^- (aq) \rightarrow \text{Al(OH)}_3(s) + 3\text{e}^- \quad E_0 = -2.35\text{V} \\
\text{Cathode: } \text{O}_2(g) + 2\text{H}_2\text{O(l)} + 4\text{e}^- \rightarrow 4\text{OH}^- (aq) \quad E_0 = +0.40\text{V} \\
\text{Overall reaction: } 4\text{Al}(s) + 3\text{O}_2(g) + 6\text{H}_2\text{O(l)} \rightarrow 4\text{Al(OH)}_3(s) \quad E_0 = -2.75\text{V}
\]

The purpose of this study is to explore the feasibility and optimal conditions of an AAFCEC for the removal of HA from wastewater and to investigate the potential power generation of its cell. We investigate the influence of different parameters on electricity generation performance to determine the optimum conditions for power generation. We also determine the form of Al in the solution by using Al–Ferron complexation timed spectrophotometry. Subsequently, we investigate the removal efficiency of HA under different reaction conditions to determine the optimum removal conditions. Finally, we compare the results and proposed an appropriate set of parameters for an optimum balance between HA removal efficiency and power generation. In this study, aluminum-air cells were directly applied to removal of HAs from water, and the species of aluminum in electrolyte is also explored, which to some extent has innovation in water treatment technology. Compared to traditional EC, AAFCEC is more economical and environmentally friendly without external power supply.

2. EXPERIMENT AND METHODS

2.1. Sample

The HA sample used in the experiments was artificially configured. HA (98%, Shanghai Macklin Biochemical Technology Co., Ltd) was dissolved in deionized water in a beaker and continuously stirred with a magnetic agitator. The obtained solution was then filtered through a 0.45 μm filter membrane. And the obtained filtrate was the HA stock solution, which was refrigerated. In the experiments, sodium chloride solution was used as the electrolyte. And 0.1 M HCl and 0.1 M NaOH were used to adjust the pH of the solution to prevent other ions from being introduced and affecting the experimental results. All other reagent used in the experiments were of analytical grade.
2.2. Experimental setup and procedures

The schematic diagram of the experiment is shown in the Figure 1. The experimental device was a rectangular parallelepiped with size of 180 mm × 30 mm × 180 mm, which was hollow inside in a cylinder format with diameter of 15 mm and height of 3 cm. The front and rear sides were composed of square organic glass plates that formed a closed container. The effective volume of the device was 800 mL. The aluminum plate anode (100 mm × 100 mm × 0.25 mm) was polished with sandpaper before the experiments and cleaned with absolute ethanol. The air electrode was composed of carbon black and activated carbon. Polytetrafluoroethylene was used as a binder. Platinum and copper were added as catalysts to the carbon powder of the catalytic layer. The carbon powder was laminated and fixed by a roller press on a nickel metal net. Peristaltic pump was used to keep internal circulation of the electrolyte at the rate of 50 mL/min in order to facilitate adequate mixing of coagulant and HAs.

In the experiment, a static aluminum–air fuel cell was constructed. Its power generation was explored by adjusting different parameters, such as NaCl concentration (0.5, 1, 2, and 3.5 g/L), initial pH (5, 6, 7, 8, and 9), electrode distance (3, 6, 9, and 12 cm), and external resistance (10, 20, and 50 Ω), which provided a technical basis for the subsequent experiments. Simultaneously, the total aluminum concentration and the aluminum form distribution were investigated under different conditions to determine the relationship between electricity production and aluminum production. The HA water sample was then placed in a beaker, and its pH (5, 6, 7, 8, and 9), NaCl concentration (0.5, 1, 2, and 3.5 g/L), and initial concentration (5 mg/L, 10 mg/L, 20 mg/L, and 40 mg/L) were adjusted to determine the removal efficiency. Subsequently, 10 mL HA solution was directly sampled from the beaker using a syringe at reaction times of 15, 30, 45, 60, 120, and 180 min. The samples were filtered through a 0.45 μm filter membrane, and their respective HA contents were measured. All the experiments were triplicate at least, and the mean was been reported.

2.3. Analysis and calculation

The voltage of the resistor was collected by a data acquisition card connected to a computer. The data acquisition card collected voltage data every 0.5 s, and all voltage values were stored in the computer for subsequent processing.

Linear sweep voltammetry was used to measure the power density and polarization curves using an electrochemical workstation with an aluminum anode as the working electrode, an air cathode as the counter electrode, and a saturated calomel electrode as the reference electrode. The measurement range was set from the open circuit voltage (−0.7 to −0.8 V) to 0 V, and the sweep gradient was set to 0.01 V. The current density (i) and power density (P) were calculated using the following Equations (4) and (5) (Chen et al. 2015):

\[ i = \frac{U}{RA} \]  
\[ P = iU \]

where U is the voltage (mV), R is the external resistance (Ω), and A is the surface area of the anode (cm²). Therefore, considering the current density as the abscissa and the power density as the ordinate, the power density curve was obtained.

The aluminum form was determined by Al-Ferron complexation timed spectrophotometry. The aluminum form can be divided into three types: Ala, Alb, and Alc. Ala is monomeric species, and it's the products of instantaneous reaction including...
Al monomers, dimer, and trimer; Alb is a medium polymer species, and it is generated within 120 min such as \( \text{Al}_{13}(\text{AlO}_4\text{Al}_{12}(\text{OH})_{24}({\text{H}_2\text{O}})_{7}^+) \); Alc is the aluminum in the species of sol or gel, molecular weight normally larger than 3,000 Da (Chen et al. 2017; Wu et al. 2019). UV254 was measured with an ultraviolet spectrophotometer at a 254 nm wavelength to represent the HA concentration.

3. RESULTS AND DISCUSSION

3.1. Electrochemical analysis

3.1.1. Effect of electrolyte concentration

The voltage values according to time for different NaCl concentrations are shown in Figure 2. Evidently, as the concentration of NaCl increases, the voltage also increases. When the NaCl concentration was 0.5, 1, 2, and 3.5 g/L, the initial voltages were 145, 207, 257, and 306 mV, respectively. This may be attributed to the solution conductivity, which increased with increasing electrolyte concentration (Maitlo et al. 2018). Therefore, the electron transfer rate increased correspondingly. In the first 15 min of reaction, the voltage increased slightly, after which it gradually stabilized. The stable voltages were 167, 229, 306, and 342 mV.

As shown in Figure 3(a), the maximum power density of the cell increased significantly as the electrolyte concentration increased. When the NaCl concentration was 0.5, 1, 2, and 3.5 g/L, the corresponding maximum power densities were 108.97, 169.36, 272.08, and 427.59 mW/cm². These results are consistent with the changes in the anode potential. Figure 3(b)
shows the polarization curves for different NaCl concentrations. When the NaCl concentration increased, the trend of the polarization curve was relatively smooth. This implies that for the same current density, the power generation is better at higher electrolyte concentrations.

Usually, the aluminum electrode surface is covered with a corrosion-protective film composed of oxide or hydroxide. The existence of this protective film hinders the charge transfer between electrode and solution, increasing the ohmic resistance and anode electrode potential. Therefore, proper removal of this protective layer is conducive to the electrode reaction. According to previous studies (Kim et al. 2017, 2018; Maitlo et al. 2019b), NaCl is one of the most effective electrolytes for such removal. The Cl\(^-\) in the solution reacts with the protective film to accelerate the dissolution rate of the aluminum anode, as shown in Equations (6)–(10) (Hubdar et al. 2018). These reactions contributed to the breakdown of the corrosion protective layer from the aluminum anode electrode surface. Therefore, NaCl reduces the ohmic resistance of the battery and improves the electron transfer efficiency. Furthermore, previous study has proved that the increase of the ionic strength with 1–6\% NaCl induced a decrease in the electrolyte resistance and an increase in the maximum power density (Huang et al. 2010). These conclusions are consistent with our experimental results.

\[
\begin{align*}
\text{Al}_2\text{O}_3 + 6\text{Cl}^- + 6\text{H}^+ & \rightarrow \text{AlCl}_3 + \text{H}_2\text{O} & \text{(6)} \\
\text{Al(OH)}_3 + \text{Cl}^- & \rightarrow \text{Al(OH)}_2\text{Cl} + \text{OH}^- & \text{(7)} \\
\text{Al(OH)}_2\text{Cl} + \text{Cl}^- & \rightarrow \text{Al(OH)}\text{Cl}_2 + \text{OH}^- & \text{(8)} \\
\text{Al(OH)}\text{Cl}_2 + \text{Cl}^- & \rightarrow \text{AlCl}_3 + \text{OH}^- & \text{(9)} \\
\text{AlCl}_3 + \text{Cl}^- & \rightarrow \text{AlCl}_4 & \text{(10)}
\end{align*}
\]

### 3.1.2. Effect of initial pH

We explored the effects of initial pH on the power generation of the Al–air fuel cells. The changes in voltage according to time are shown in Figure 4. Weak acid or weak alkaline electrolytes were more conducive to electricity generation than that in the neutral electrolyte. In contrast, neutral electrolyte led to a relatively poor power generation performance. For an initial pH of 9, the voltage was the highest, and the corresponding stable voltage reached 247 mV. When the initial pH was 5, the stable voltage reached 244 mV. The cell showed the worst performance at pH 7. Overall, the voltage was not significantly different at different initial pH values.

Similar results were observed for the power density curve and polarization curve. As shown in Figure 5(a), for initial pH of 9, the power density was the maximum, reaching 326.10 mW/cm\(^2\). At pH 5, the second highest power density of 313.47 mW/cm\(^2\) was obtained. For initial pH of 7, the power density was the smallest, at 267.807 mW/cm\(^2\). The relative anode potentials at different initial pH values presented similar results. As shown in Figure 5(b), the anode potential for weak acid and alkaline electrolytes increased slowly, which is consistent with the results described above.

![Figure 4](image-url) | Voltage according to operation time for different initial pH.
In other words, the electricity generation performance of the aluminum–air fuel cell was better in weak acid and alkaline electrolytes. This may be concerned to the behavior of anodic corrosion in the weak acid and alkaline electrolyte. The Al anode is amphoteric, which is soluble in the acid as well as in alkali solutions. In the acid solution, the solubility of Al$^{3+}$ accelerated the chloride attack. And in the alkaline electrolyte, OH$^-$ will react with the passivation layer. Therefore, the protective layer is thinned by the chemical dissolution (Ma et al. 2013a). Consequently, the cells obtained superior power generation. However, in a neutral electrolyte, the passive layer of the aluminum sheet hardly reacted so that it is quite stable (Ma et al. 2013b) Accordingly, the electricity generation performance was relatively poor.

3.1.3. Effect of electrode distance and external resistance

To explore the effect of electrode distance on cell power generation, experiments were performed with electrode spacing of 3, 6, 9, and 12 cm. Figure 6 shows that the voltage decreased significantly with the increase in electrode distance. For the electrode distance of 3 cm, the voltage reached its maximum, at approximately 300 mV. For electrode spacing of 12 cm, the stable voltage reached only 190 mV. This occurred because the internal resistance increased with the electrode distance, thereby increasing the energy consumption (Hashim et al. 2017a, 2017b). We used voltammetry to roughly measure the cell internal resistance; the results are shown in Figure 7. The slope of the fitted volt–ampere characteristic curve was used to roughly estimate the internal resistance of the fuel cell. For electrode distances of 3, 6, 9, and 12 cm, the internal resistances were approximately 12, 18, 25, and 32 Ω, respectively. Therefore, electrode spacing significantly impacts the aluminum–air cell, and we set the electrode spacing to 3 cm in the subsequent experiments to obtain a better power generation.

We investigated the influence of external resistors on the power generation of the aluminum–air cell. The voltage and power according to time at different external resistances are shown in Figure 8. The highest voltages were obtained under external resistance of 50 Ω, reaching 420 mV; however, the output power was very low at that resistance. In contrast, lower voltages and higher powers were obtained at 10 Ω. It can be explained as that the external resistors controls the ratio between voltage and electric current (Wang et al. 2009). The current in the close circuit would decrease at a higher external resistor. Thus, the lower output power was achieved under external resistance of 50 Ω. Therefore, to provide a considerable output power and ensure a high stable voltage, an external resistance of 20 Ω was used in the subsequent experiments.

3.2. Aluminum speciation

Al species are crucial in the EC process as they determine the behavior and efficiency of Al-based coagulants (He et al. 2016; Kong et al. 2021). Equations (1)–(3) show that the anode dissolves to generate Al$^{3+}$ into the solution, and Al$^{3+}$ is hydrolyzed to form various forms of compounds or polymers. The aluminum species is essential to determine whether the EC can remove HAs. This study investigates the optimum operating parameters according to the distribution of aluminum speciation in the solution.
Figure 9 shows the distribution of aluminum speciation according to time for different NaCl solution concentrations at pH 5. As the reaction progressed, the concentrations of Ala, Alb, and Alc gradually increased. The concentration of Alc increased quickly and reached the highest amount at the end. The contents of Ala and Alb were relatively low, and their growth was relatively slow, and it even presented a downward trend after some time. This can be explained by the fact that Ala and Alb in the
Figure 8 | (a) Voltage and (b) output power according to time for different external resistance.

Figure 9 | Aluminum speciation distribution according to time for NaCl solution concentrations of (a) 0.5, (b) 1, (c) 2, and (d) 3.5 g/L.
solution may be continuously converted into Alc as the reaction progresses (Yan et al. 2007). The final Al concentration indicates that all aluminum species in the solution increased with the NaCl concentration. When the NaCl concentration reached 3.5 g/L, the concentrations of Ala, Alb, and Alc reached 1.131, 8.145, and 16.147 mg/L, respectively.

To explore the effect of pH on the Al species, the initial solution pH was adjusted to 5, 6, 7, 8, and 9 using dilute hydrochloric acid and dilute sodium hydroxide. In the experiments, a 3.5 g/L NaCl solution was used as the electrolyte. Figure 10

Figure 10 | Aluminum speciation distribution according to time for different initial pH of (a) 5, (b) 6, (c) 7, (d) 8, and (e) 9.
shows that the concentration of all Al species in the solution have increased. The total aluminum concentration was the highest at pH 9. As described in Section 3.1, the fuel cell presented the best electricity production performance at same pH. In general, the total aluminum concentration did not significantly change according to pH. The influence of pH on battery reaction is mainly reflected in distribution of aluminum species. It is found that the Ala is the most unstable species. As the pH increases, this species would quickly transform into Alb and finally Alc. Similar results has been reported before (Wang et al. 2004).

We also observed that the concentration of Alc was higher when the electrolyte was weakly alkaline. At pH 9, the concentration of Alc reached 20.163 mg/L. The content of Ala and Alb in the weakly acidic electrolyte was relatively higher than that in the weakly alkaline electrolyte. This may be attributed to the hydrolysis reaction of Al$^{3+}$. According to previous studies (Hu et al. 2012), the main hydrolysis products of Al$^{3+}$ are different at different pH values. This may be explained by the presence of H$^+$, which inhibits the conversion of Ala to Alc in an acidic solution. Therefore, Al$^{3+}$ and AlOH$_2^+$ were dominant in the solution. Moreover, as the pH value of the solution increases, the increase in OH$^-$ promoted the formation of Al(OH)$_3$, and the concentration of Alc gradually increased.

3.3. HA removal

According to previous studies electrolyte (Maitlo et al. 2019a; Zaied et al. 2020), concentration is an important factor of EC. As shown in Figure 11(a), the HA removal efficiency significantly improved with increasing NaCl concentration. When the NaCl concentration was 3.5 and 2 g/L, the HA removal was over 90% in approximately 30 min of reaction. After
approximately 40 min, the HA removal rate was approximately 95% for all samples, except for those with NaCl concentration of 0.5 g/L.

This phenomenon can be explained by the higher ion concentration in the solution, which significantly increases the conductivity, thereby significantly improving the transfer rate of electrons on the electrode surface (Tanattı et al. 2018). Correspondingly, the dissolution rate of the anode also increases, as well the amount of aluminum flocculant. Cl⁻ has the potential to break down the protective layer at the surface of the metal anode electrodes to remarkably increase the current density (Kim et al. 2015). In addition, as described in 3.2, the concentration of the NaCl solution also affects the speciation distribution of Al. As the concentration of the NaCl solution increases, the total aluminum concentration, especially the concentration of Alc, also significantly increases. According to previous studies (Garcia-Segura et al. 2017), Al(OH)₃ is the main coagulant in Alc, and it is essential for the electro-flocculation process. According to our experiments, when the NaCl concentration is greater than 1 g/L, most HAs can be removed. Therefore, considering the economic benefits, the subsequent experiments were performed using 1 g/L NaCl solution.

The effect of initial HA concentration is shown in Figure 11(b). We prepared HA samples of 5, 10, 20, and 40 mg/L to investigate the effect of different HA concentrations on the removal efficiency. After 3 h of reaction, the HA removal rate in the solution at different initial concentrations surpassed 96%. When the HA concentration was less than 20 mg/L, the removal rate was higher than 90% after 60 min. For this reaction time, the 40 mg/L HA sample presented a removal rate smaller than 50%. Therefore, 20 mg/L was the best initial HA concentration, and it was used in the subsequent experiments.

The pH can significantly affect the EC process, which can affect the Al hydrolysate and HA structure (Antonio et al. 2020; Koparal et al. 2008). The effect of HA removal at different pH values is shown in Figure 11(c). The initial pH value presented a certain effect on the HA removal. After the reaction proceeded for 120 min, the UV₂₅₄ values all dropped to a lower level, which indicated that the HAs were removed. Although the HA removal efficiency in the acidic solution was slightly higher than that in the alkaline solution, the overall difference was not significant. A relatively higher removal efficiency of HA was observed at pH 5. This is also consistent with the results of previous studies (Merma et al. 2020). This phenomenon can be explained by the formation of a gel layer on the surface of the aluminum anode under high pH and high HA concentration. At pH 5, the presence of H⁺ can inhibit the formation of the gel layer. In addition, as mentioned in Section 3.1, the aluminum-air fuel cell has a relatively good power generation at pH 5. Furthermore, the total aluminum concentration in the solution is relatively high at this pH. Therefore, pH 5 was considered the optimum pH value.

4. CONCLUSIONS

In this study, we demonstrate that AAFCEC can be used for HA removal in the wastewater without external power supply. We determined the optimal AAFCEC parameters for power generation and HA removal. The result indicated that the battery can achieve the max power density of 427.59 mW/cm² at the condition of 3.5 g/L NaCl concentration, pH 9, and initial HA concentration of 20 mg/L. Based on economic aspects and HA removal, we adjusted the relevant parameters to 1 g/L NaCl concentration, pH 5, and initial HA concentration of 20 mg/L, at which the power density reached 313.47 mW/cm², and the HAs removal rate was over 99%. Our results indicate that AAFCEC is efficient water treatment for HAs removal. And this device has a great advantage on wastewater treatment of rural areas or households because of its indispensable nature for external power supply. Further work on the application of an AAFCEC system as treatment technology for more complex water pollutants require explored, such as arsenic, heavy metal, dyes, fluorides, natural organic. This technology will be developed into a cost-effective, efficient and environmentally friendly water treatment technology in the future.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

CONSENT TO PUBLISH

Not applicable.

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AUTHOR CONTRIBUTIONS
Conceptualization, Wei Wei; Formal analysis, Shuguang Zhu and Wei Wei; Funding acquisition, Wei Wei; Methodology, Haoyang Gong; Project administration, Haoyang Gong; Resources, Lin Sheng and Dong Zhou; Validation, Wei Wei; Writing – original draft, Haoyang Gong.

CONFLICTS OF INTEREST
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

VAILABILITY OF DATA AND MATERIALS
All data generated or analysed during this study are included in this published article [and its supplementary information files]. And that the content of the article and the experimental data are authentic.

DATA AVAILABILITY STATEMENT
All relevant data are included in the paper or its Supplementary Information.

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