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Preparation of PANI/PPy/CC Hydrogel Electrode and Its Performance in Microbial Fuel Cell

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Abstract

In order to improve the overall performance and practicability of MFC, anode materials with excellent conductivity, good corrosion resistance, large specific surface area and good biocompatibility should be selected. Modification can not only provide a large specific surface area and good biocompatibility for the growth of biofilm, but also make the anode surface easier for microbial colonization and substrate transfer, thus greatly reducing the polarization loss. Polyaniline (PANI) and polypyrrole (PPy), as the two most popular conductive polymers, can significantly reduce the internal resistance of the reactor and reduce the electron transfer impedance. It shows excellent performance in the process of electron transfer, and the synthesis method is relatively simple, so it is widely used in the study of MFC anode material modification.In this paper, polyaniline and polypyrrole were combined to construct a composite hydrogel electrode with aniline and pyrrole monomers at different molar ratios, forming a composite electrode with an inner layer of carbon cloth (CC) material and an outer layer of hydrogel material with good electrical conductivity. The electrochemical performance and power generation performance of the electrode were tested by various characterization and testing techniques. Different comparative experiments were used to optimize the electrode preparation conditions and determine the optimal doping ratio.

Keywords

Microbial fuel cell, Anode modification, Polyaniline, Polypyrrole, Electricity production performance.

1. INTRODUCTION

With the development of the global economy and the acceleration of urbanization and industrialization, the speed of energy consumption is also accelerating, and the energy crisis is gradually intensifying. At the same time, the total amount of industrial wastewater discharged in China every year is huge. These wastewater components are complex and difficult to degrade, seriously endangering human production and life[1] The development of sustainable and pollution-free energy has become the only way for human beings to seek long-term development. In order to solve the two serious problems of energy shortage and water pollution, wastewater resource utilization has become a sustainable development direction of wastewater treatment in the future[2].

Microbial fuel cell (MFC), as a new type of energy conversion device with both sewage purification and energy recovery capabilities, has great application prospects in the fields of

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environment, new energy, medicine, ocean and even aerospace[3]. MFC is an el ectrochemical fuel cell that directly converts the chemical energy of organic compounds into electrical energy through the catalytic reaction of external electrons or anode breathing bacteria[4]. As a technology for simultaneous treatment of wastewater and generation of green energy, it has attracted more and more attention in recent years[5]. The working principle of MFC is shown in Fig. 1.In the anode chamber, organic pollutants are degraded by electrogenic microorganisms, releasing electrons and protons during the metabolic process. Electrons are directly or indirectly transferred to the anode, and then reach the cathode through an external circuit to form a current, and an oxidation reaction occurs. In the cathode chamber, the protons produced by the anode pass through the proton exchange membrane to the cathode and react with O_2 and electrons to form H_2O[6].

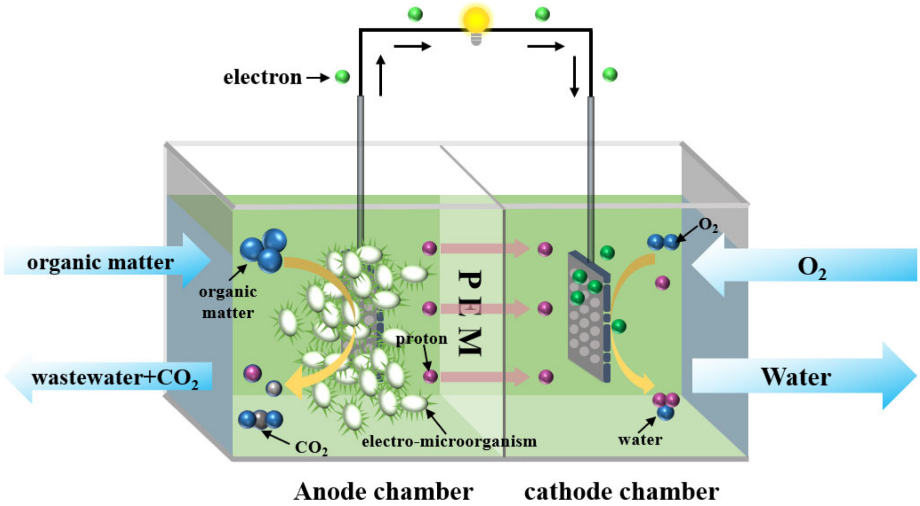

Figure 1. Microbial fuel cell working principle diagram

It can be seen that the anode material is directly involved in the oxidation reaction catalyzed by microorganisms. The microorganisms adsorbed on the anode surface played a decisive role in the electricity generation of MFC[7]. The anode material affects the performance of MFC by affecting the formation of biofilm and bioelectrochemical reaction. The biofilm formed on the anode surface can improve the conductivity between the substrate and the electrode. In the MFC system, the anode is not only a conductor, the anode material needs to be conducive to the attachment and growth of the electrogenic bacteria, which helps the electron transfer to the anode surface. At the same time, it should have the characteristics of small internal resistance, relatively stable potential, good biocompatibility and high chemical stability[8]. In order to improve bacterial adhesion and electron transfer, anode surface modification has become a research hotspot in the field of MFC research[9].

Polyaniline can be used as a modified material to directly improve the anode performance of MFC, or indirectly improve the anode performance by affecting the formation of anode biofilm. Polyaniline has a high specific surface area and good biocompatibility, which is conducive to the attachment of electrogenic bacteria and provides a new selective environment for bacterial metabolism. Moreover, polyaniline can be used as an intermediary to change the transfer path of electrons from the strain to the anode surface, making it easier for the strain with low electron transfer ability to achieve electron transfer[10] As a heterocyclic conjugated conductive polymer, polypyrrole is widely used in electrode materials, anti-corrosion materials, sensors and other fields. As an anode material for MFC, polypyrrole is often combined with carbon materials and anthraquinone materials to modify MFC anode materials[11].

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Hydrogel is a kind of polymer with three-dimensional network structure obtained by crosslinking hydrophilic polymer chains, which has high hydrophilicity and adjustable physical and chemical properties. This makes it have great potential application value in the field of biology and medicine[12][13]. Conductive hydrogels can be obtained by adding conductive materials based on hydrogels. Compared with traditional hydrogels, conductive hydrogels have the characteristics of both hydrogel materials and conductive materials, and have good biocompatibility and unique conductivity[14]. Conductive polymer hydrogels headed by polypyrrole hydrogels and polyaniline hydrogels have shown broad application prospects in recent years[15].

2. PREPARATION OF PANI/PPY/CC HYDROGEL ELECTRODE

(1) Configuration Solution A: 0.602 mL, 0.401 mL and 0.201 mL of aniline were added to the three reagent bottles respectively, and 1.205 mL of pyrrole monomer was added to the above reagent bottles. Stir in ice water for 10 min to make the solution mixed evenly. 2.506 mL of phytic acid was added respectively and stirred in ice water for 5 min. Subsequently, 5 mL of isopropanol solution was slowly added to each of the three reagent bottles and magnetically stirred for 20 min.

(2) Configuration solution B: Weigh 2.508 g ammonium persulfate in the reagent bottle, add 6 mL deionized water, magnetic stirring for 10 min to dissolve.

(3) Three treated carbon cloths were placed in solution A respectively, and waited for 15 min to make the carbon cloth completely wet in solution A. Subsequently, solution B was slowly added to solution A in an ice water bath.

(4) After placing the mixed solution for a week, the completely reacted carbon cloth hydrogel electrode was taken out and immersed in deionized water to remove impurities until the water was colorless.

The PANI/PPy/CC hydrogel electrode was prepared as shown in Fig. 2.

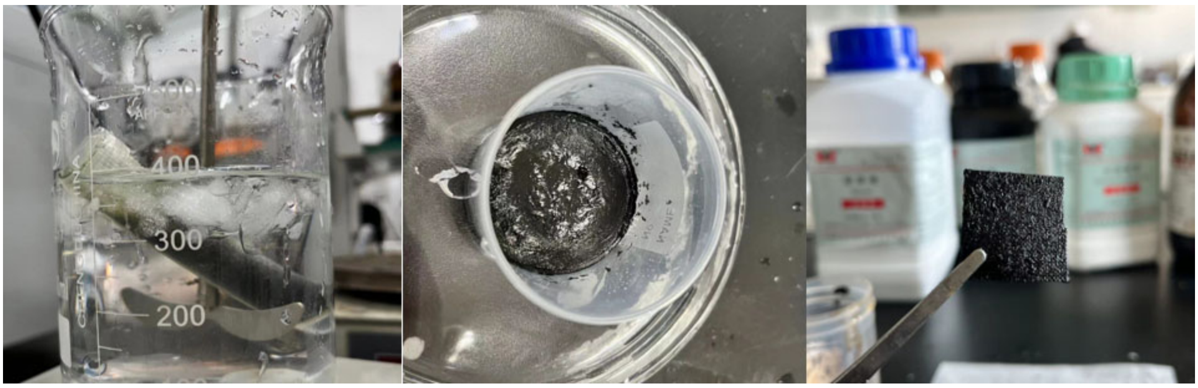

Figure 2. Preparation of PANI/PPy/CC hydrogel electrode

3. CHARACTERIZATION OF HYDROGEL ELECTRODE

3.1. Scanning electron microscope analysis

Fig. 3 is the scanning electron microscope image of CC, PANI/PPy/CC hydrogel electrode under different multiples. Among them, (a) (b) (c) is the SEM diagram of CC electrode. It can be seen from the diagram that CC is composed of many carbon fibers on the surface, which are connected with each other, and the surface of carbon fiber is smooth and delicate, with clear lines. (d) (e) (f) is the SEM image of PANI/PPy/CC hydrogel electrode. It can be seen from the figure that the PPy nanospheres on the surface of the PANI/PPy/CC hydrogel electrode material

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are closely connected with the PANI sheet structure. PPy particles were accumulated on the surface of PANI, and the surface was rough. Compared with the untreated CC electrode, the PANI/PPy modified CC electrode increased the specific surface area of the carbon fiber, and the increase of the specific surface area provided more space for the attachment and growth of the electricity-producing microorganisms[16]. With the increase of the amount of electricity-producing microorganisms on the electrode surface, the electricity production performance of MFC was also improved accordingly.

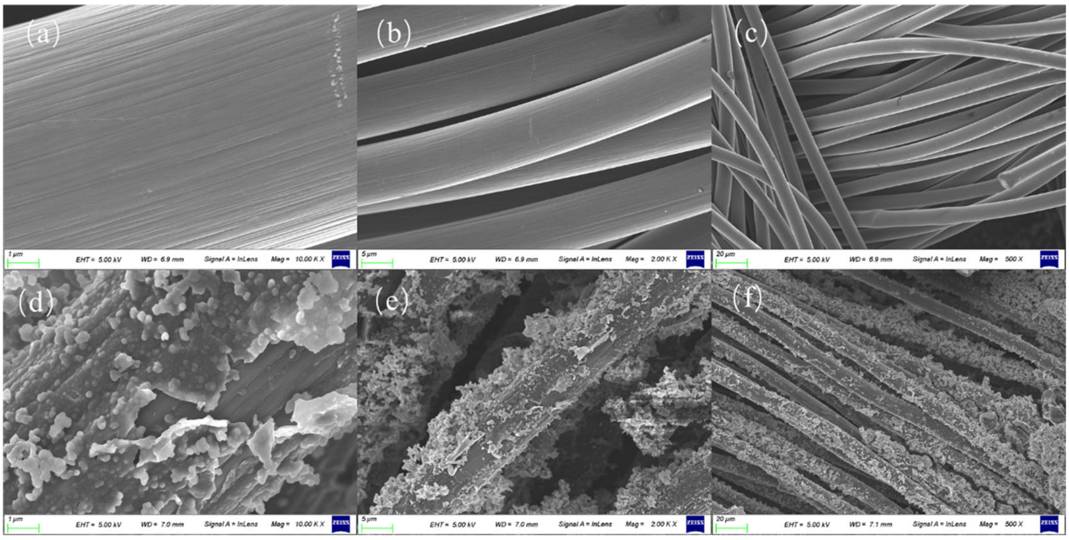

Figure 3. SEM images of different hydrogel electrodes

3.2. Infrared absorption spectroscopy analysis

Fig. 4 is the infrared absorption spectra of PANI/CC, PPy/CC and PANI/PPy/CC hydrogel electrodes.

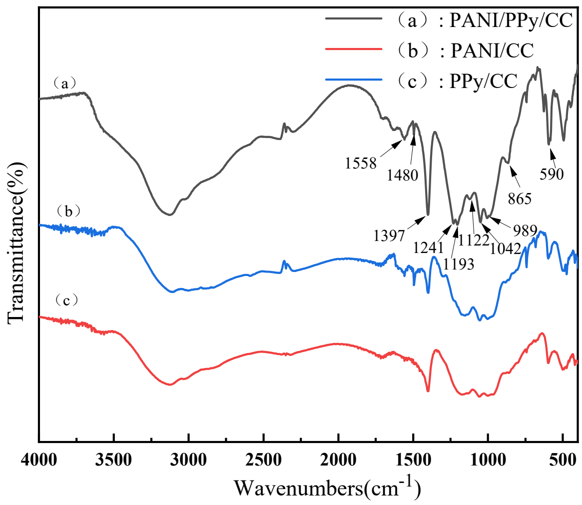

Figure 4. FTIR spectra of different electrode materials

In the figure, (a), (b) and (c) correspond to PANI/PPy/CC, PANI/CC and PPy/CC, respectively. In the PANI/PPy/CC spectrum, the C=C stretching vibration absorption peak is at 1558 cm^{-1}, and the C-H ring deformation vibration peaks are at 1042 cm^{-1} and 989 cm^{-1}. The above can explain the existence of PPy characteristic peaks in PANI/PPy/CC hydrogel electrode. The

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absorption peak at 1480 cm^{-1} is the absorption peak of semiquinone and quinone, 1193 cm^{-1} is attributed to the bending vibration of C-H plane, 865 cm^{-1} is the ring deformation vibration absorption peak of substituted benzene ring, and 590 cm^{-1} is the stretching vibration absorption peak of benzene ring. These absorption peaks indicate that PANI characteristic peaks exist in the PANI/PPy/CC hydrogel electrode. Compared with PPy/CC, the position of the absorption peak of PANI/PPy/CC was obviously shifted back, and the absorption peaks at 1397 cm^{-1} and 1042 cm^{-1} were also enhanced to a certain extent. The main reason is that these two are both the characteristic absorption peaks of the pyrrole ring and the vibration absorption peaks of the aromatic ring skeleton. Compared with PANI/CC, the peak intensity ratio of quinone structure and benzene ring structure in PANI/PPy/CC composites increased, indicating that the quinone structure in the composites increased. This is mainly because the quinone ring of PANI and the π bond in PPy form a conjugated structure[17].

3.3. Performance testing of hydrogel electrodes

3.3.1 Contact angle test

The hydrophilic ability of the analytical electrode can be characterized by measuring the water contact angle of different electrodes. The water contact angle test results are shown in Fig. 5. In the figure, (a), (b), (c), (d) are the water contact angles of unmodified carbon cloth electrode, 1:2, 1:4, 1:6 PANI/PPy/CC hydrogel electrode. It can be seen from the figure that the surface contact angle of the unmodified carbon cloth electrode is 93.302 °, while the water contact angles of the PANI/PPy/CC hydrogel electrodes at 1:2,1:4 and 1:6 are 71.434 °, 23.311 ° and 48.214 °, respectively. Through the contact angle test of PANI/PPy/CC hydrogel electrode, the surface contact angle of carbon cloth electrode modified by polyaniline and polypyrrole was significantly reduced. In the experiment, it was found that the electrode can absorb water instantly, has good water absorption performance, and the hydrophilic ability has been greatly improved. The electrode is easier to contact with the anode nutrient solution, which facilitates the capture of nutrients by microorganisms. The good water absorption of the electrode can help the microorganism to better adhere to the electrode surface, not easy to fall off, and help to improve the polarization resistance and electricity generation performance of MFC.

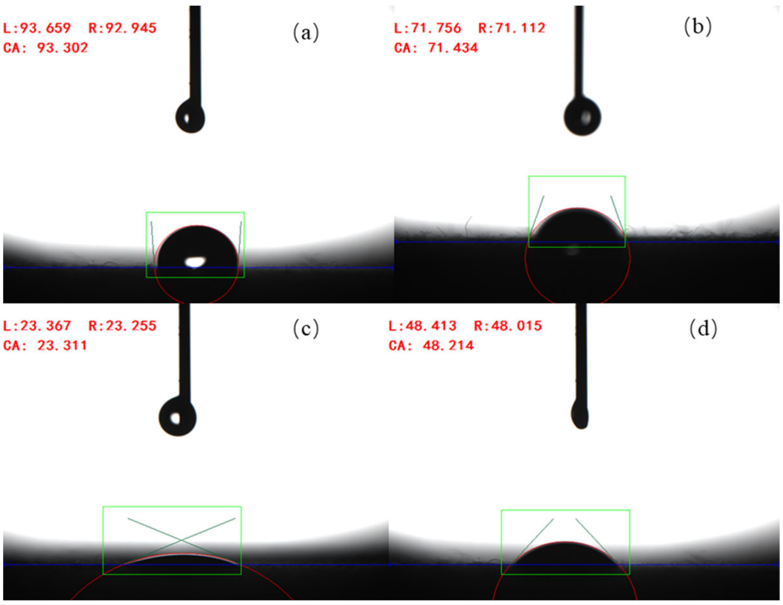

Figure 5. Contact angle of different anode surfaces

3.3.2 Swelling performance test

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In order to analyze the effect of different aniline monomer ratios on the swelling properties of PANI/PPy/CC hydrogel electrodes, the swelling properties of hydrogel electrodes with different aniline monomer contents were tested. Fig. 6 is the water absorbency of hydrogels with different PANI content. It can be seen from the figure that as the soaking time increases, the water absorption rate of different hydrogels also increases. The swelling ratio of carbon cloth electrode without polyaniline and polypyrrole increased from 5.4 g/g to 10.6 g/g, the swelling ratio of 1:2 PANI/PPy/CC hydrogel electrode increased from 5.1 g/g to 10.1 g/g, the swelling ratio of 1:4 PANI/PPy/CC hydrogel electrode increased from 5.5 g/g to 9.7 g/g, and the swelling ratio of 1:6 PANI/PPy/CC hydrogel electrode increased from 5.5 g/g to 9 g/g. With the increase of doping ratio, the swelling ratio of the electrode decreased gradually, when the soaking time was about 800 min. The swelling properties of the hydrogels are close to equilibrium.

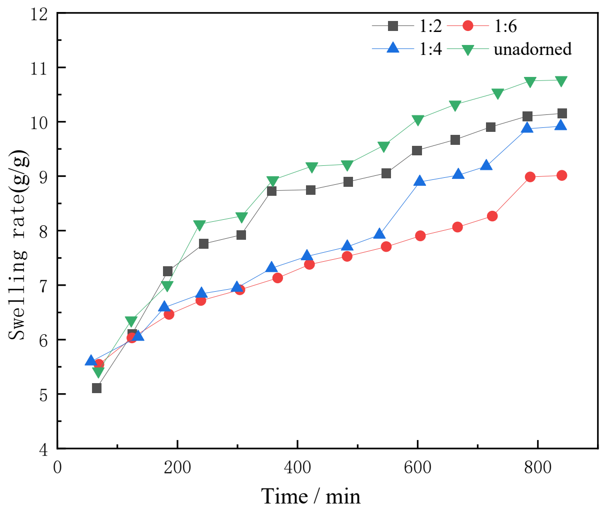

Figure 6. The swelling rate of PANI/PPy/CC hydrogel electrodes with different AN content changed with time

3.4. Performance test of different electrodes in MFC

3.4.1 Cyclic voltammetry test

Fig. 7 is the CV scanning curve of different proportion hydrogel electrodes. It can be seen from the scanning diagram that the modified electrodes have two pairs of redox peaks. The order of the area enclosed by the CV curve of the electrode is 1:4 > 1:6 > 1:2 > unmodified. The area of the unmodified CC is the smallest in the four groups of electrodes, indicating that the specific capacitance of the CC electrode is very small, and there is no redox peak during the test. The CV curve area of PANI/PPy/CC composite hydrogel electrode is the largest at the ratio of 1:4, indicating that the electrode has the highest specific capacitance. It can be seen from the CV curve that due to the addition of aniline, the specific capacitance of the 1:2 PANI/PPy/CC hydrogel electrode MFC was significantly higher than that of the unmodified carbon cloth electrode MFC. When the proportion of aniline is increased to 1:4, the specific capacity of the battery is the highest. When the aniline ratio continues to increase to 1:6, the specific capacitance of the battery is lower than that of the 1:4 aniline addition. Because, polyaniline from the initial cryptogreen imine to emerald green imine, and finally until the complete oxidation of aniline black. The electrode has the best conductivity and capacitance performance when polyaniline is in the semi-oxidized state.

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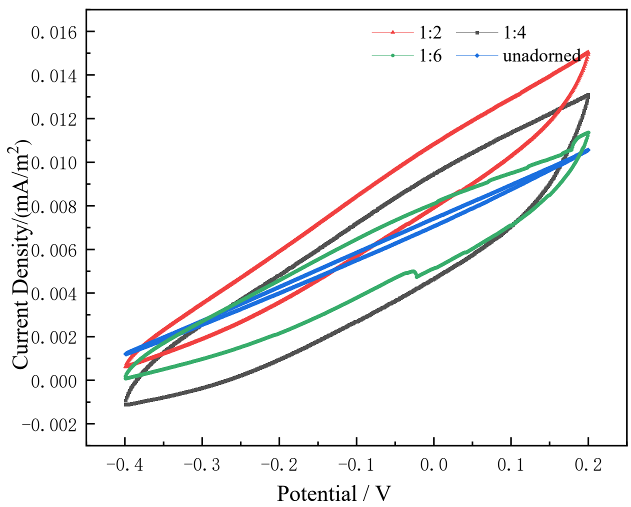

Figure 7. Cyclic voltammetry curves of composite hydrogel electrodes with different AN ratios

Fig. 8 is the CV curve of the hydrogel electrode at a ratio of 1:4 at five scanning rates. It can be seen from the figure that by continuously increasing the scanning rate, the current density of the electrode is also increasing and the area of the CV curve is gradually increasing when the potential is constant. The maximum current density increased from 0.014 mA/m^2 at a scan rate of 5 mV/s to 0.026 mA/m^2 at a scan rate of 100 mV/s, an increase of nearly 2 times. Tests show that the electrode has fast response speed and good conductivity. The CV curves of the five groups of scanning speeds show a symmetrical leaf shape, indicating that the PANI/PPy/CC hydrogel electrode has good reversibility.

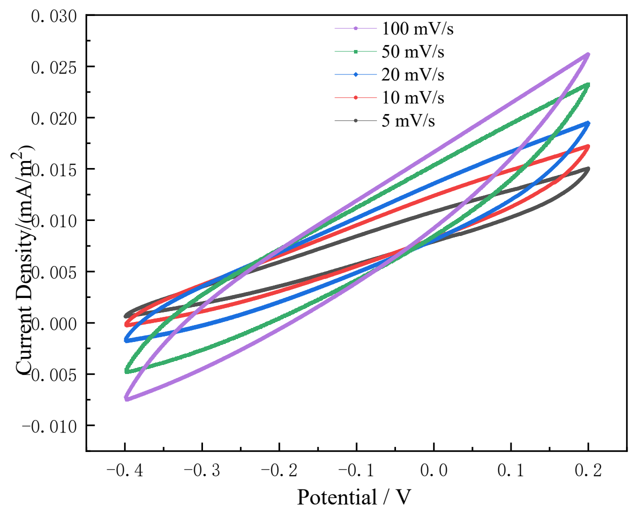

Figure 8. CV curves of hydrogel electrode at different scan rates

3.4.2 AC impedance test

Fig. 9 shows the AC impedance spectra of PANI/PPy/CC hydrogel electrodes with different ratios under the condition of stable open circuit voltage. Before the test, the MFC needs to be open for 2 hours. When the open circuit voltage remains stable, the electrochemical test is

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performed through a three-electrode system. The EIS diagrams of hydrogel electrodes at different ratios all contain high-frequency semicircles and low-frequency oblique lines[18]. The electrochemical reaction process of the electrode is mainly reflected in the high frequency region, while the diffusion process of the electrode is mainly reflected in the low frequency region. The intersection point of the starting point of the semicircle in the high frequency region and the X axis represents the sum of the solution resistance and the electrode internal resistance, which is represented by R_1. The diameter of the semicircle in the high frequency region represents the electron transfer resistance, which is represented by Rct. The oblique line in the low frequency region represents the Warburg impedance, which can reflect the diffusion of the electrolyte in the electrode.

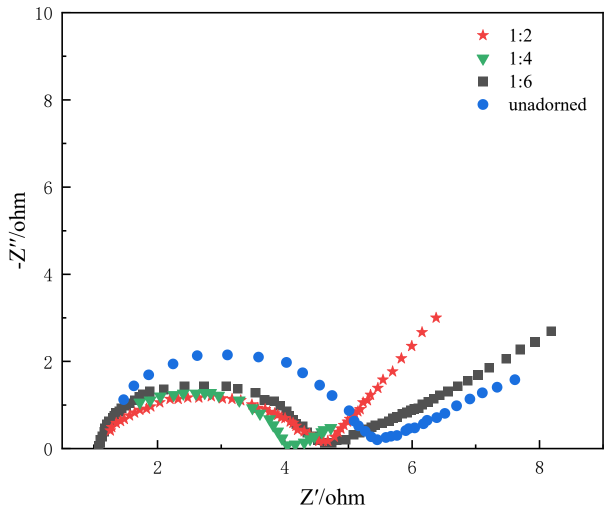

Figure 9. EIS diagram of composite hydrogel electrode under different AN ratios

Fig. 10 is the fitted equivalent circuit diagram. The electron transfer resistance Rct of four hydrogel electrodes can be obtained by ZView software fitting. From Table 1, it can be seen that the Rct resistance data decreases first and then increases in the high frequency region. When the doping ratio is 1:4, the Rct resistance of PANI/PPy/CC hydrogel electrode is 3.24 Ω, which is smaller than that of other groups of electrodes. At this time, polyaniline and polypyrrole have a synergistic effect, which reduces the electron transfer impedance, facilitates the transfer of charge, and is conducive to improving the conductivity of the electrode. In the low frequency region, the slope of the straight line is not very different, indicating that the diffusion impedance of the hydrogel electrodes prepared at different ratios is similar.

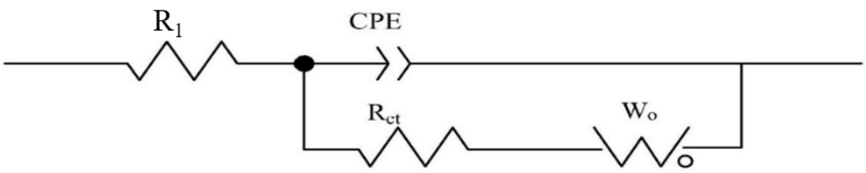

Figure 10. Equivalent circuit diagram

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Table 1. Impedance fitting data of different electrodes

Electrode	Unmodified	1:2	1:4	1:6
R_1 (Ω)	1.21	1.12	0.86	1.06
Rct (Ω)	3.86	3.56	3.24	3.63

3.4.3 Polarization curve test

The four groups of hydrogel electrodes were used as the anodes of the MFC device, and the device was started and tested for electricity generation after several cycles of stable operation. The four batteries were opened separately, and the potassium ferricyanide solution in the cathode chamber was replaced to keep the cathode potential consistent. When the potential is stable, the external voltage of the battery is obtained by changing different external resistances, and the power density curve and polarization curve of the battery are further calculated. Fig. 11 is the polarization curve of MFC with PANI/PPy/CC hydrogel electrode as MFC anode under different AN ratios.

The highest point represents the open circuit voltage (OCV) of the four groups of batteries. The open circuit voltages of the unmodified, 1:2,1:4, and 1:6 groups of MFCs were 740.1 mV, 760 mV, 780.5 mV, and 751.3 mV, respectively. The OCV of MFC at a ratio of 1:4 is higher than other ratios. Mainly because when the cathode potential is stable, the OCV of the battery will be affected by the microbial population. As shown in Fig. 11, the degree of decrease in the polarization curve of the four groups of MFCs also showed significant differences. With the increase of current density, the MFC output voltage of 1:4 PANI/PPy/CC hydrogel anode decreased slowly, and the polarization degree was the smallest. On the contrary, with the increase of current density, the MFC output voltage of the hydrogel electrode MFC at the ratio of 1:6 shows a rapid downward trend, and the polarization degree of the battery is the largest.

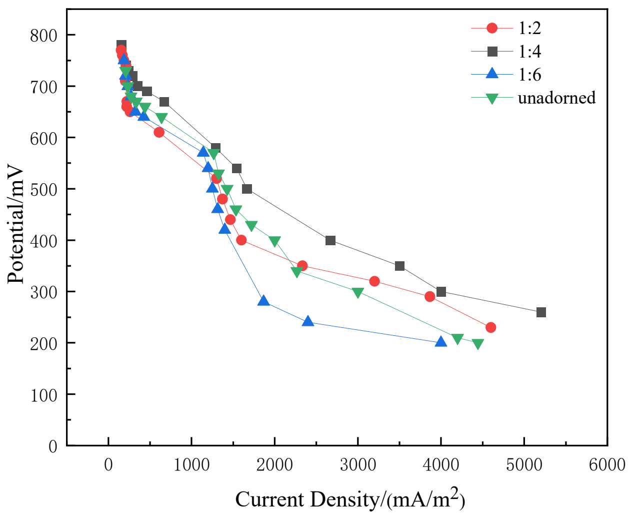

Figure 11. Polarization curves of MFC under different electrodes

3.4.4 MFC cycle voltage test

Fig. 12 is the periodic curve of the output voltage of the hydrogel electrode as the MFC anode under different AN ratios. Starting from the inoculation of electrogenic microorganisms, the anolyte and catholyte were replaced when the cell voltage was lower than 100 mV, and each replacement was recorded as an operating cycle of the cell. The multi-cycle operation curve of

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MFC can reflect the relationship between the output voltage of the battery and the time, and can analyze the growth and reproduction of the electrogenic microorganisms on the electrode and the electrochemical performance of MFC.

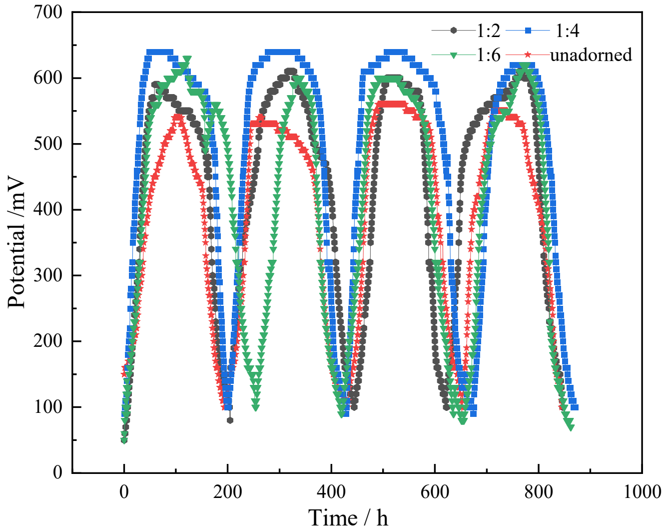

Figure 12. Periodic voltage curves of MFCs with different anodes under 1000 Ω external resistance

When the battery is just running, the electricity-producing microorganisms attached to the anode need to undergo a certain period of domestication. The biofilm on the electrode surface is not stable, resulting in fluctuations in the output voltage. After a period of domestication, the output voltage becomes stable, and the electricity-producing microorganisms can transfer electrons to the electrode in time. During each operation cycle, the output voltage of all batteries shows a trend of rising first and then decreasing. The maximum output voltages of unmodified, 1:2, 1:4 and 1:6 MFCs were 561.42 mV, 603.56 mV, 645.85 mV and 620.15 mV, respectively. At the same time, it can be seen that the MFC voltage of 1:4 PANI/PPy/CC hydrogel electrode rises fastest with time and reaches the peak first. The MFC voltage of the unmodified carbon cloth electrode rises slowly with time. This situation may be due to the low biocompatibility of the unmodified electrode itself and the small surface area compared with the other three groups of modified electrodes. It is impossible to guarantee the number of electricity-producing microorganisms attached to the electrode surface and the growth and metabolism effect, which is not conducive to the transfer of electrons from the cell membrane to the electrode surface, so the output voltage is naturally low. It can also be seen that the first cycle running time of 1:6 PANI/PPy/CC hydrogel electrode MFC is significantly longer than other electrodes, which is about 260 h. This may be due to the high proportion of polyaniline and polypyrrole, which makes the electrogenic microorganisms accumulate in a short time and fully degrade the organic substrate in the anode chamber. Comparing the four groups of output voltage curves with time, it can be seen that the 1:4 PANI/PPy/CC hydrogel electrode has the highest output voltage, and the one-time running time can be maintained at about 200 h, which is higher than other groups of electrodes. It shows that the stability of the electrode is good and the power generation performance is the best under this ratio.

3.4.5 MFC power density curve test

Fig. 13 is the power density curve of MFC with different proportions of hydrogel electrode as anode. The MFC power densities of the four groups of hydrogel electrode materials with

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different proportions were 2002.05 mW/m^2,2187.5 mW/m^2,2019.6 mW/m^2 and 1848.75 mW/m^2, respectively. Compared with the unmodified electrode, the power density of MFC in the three groups of 1:2,1:4 and 1:6 increased by 8.29 %, 18.32 % and 9.24 %, respectively. The power density of the electrode is the highest at the ratio of 1:4. When the polyaniline content is higher or lower than this ratio, the output power begins to decrease. It can be seen from the figure that although the electrochemical activity of polyaniline is high, it will cause a large internal resistance when the content is too high. At a ratio of 1:4, it can not only maintain the good electrochemical activity of the electrode, but also promote the electron transfer between the electrode and the electricity-producing microorganisms, thereby improving the electricity generation performance of MFC. Therefore, PANI:PPy=1:4 was selected as the optimal doping ratio of the composite hydrogel.

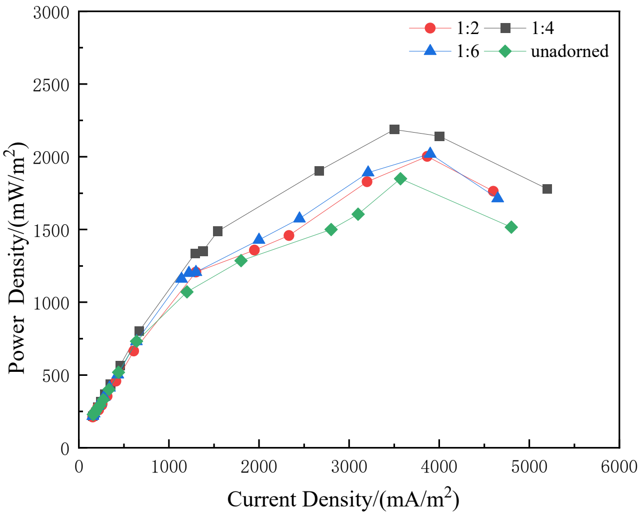

Figure 13. Power density curves of MFC under different electrodes

3.4.6 The effect of external resistance on the electrical performance of MFC production

Fig. 14 shows the variation of MFC output voltage with time of 1:4 PANI/PPy/CC hydrogel electrode under different external resistances. As shown in the figure, when the external resistance is fixed at 500 Ω, the maximum output voltage of MFC is 471.88 mV. When the external resistance is 1000 Ω, the maximum output voltage is 648.52 mV ; when the external resistance is 200 Ω, the maximum output voltage is 372.05 mV. It can be seen from the figure that the output voltage of MFC has a stable period under different external resistances, which proves that the MFC started with 1:4 PANI/PPy/CC hydrogel electrode can provide long-term stable output voltage for external loads. When the organic substrate in the anode chamber is exhausted, the output voltage of MFC shows a rapid downward trend. The smaller the external resistance is, the faster the organic substrate is consumed. Therefore, reducing the external resistance can promote the degradation of organic substrates in MFC.

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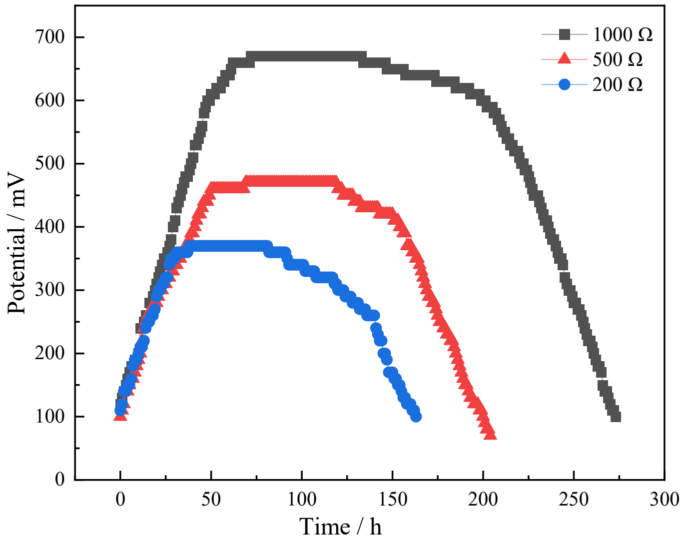

Figure 14. MFC output voltage curve under different external resistance

4. CONCLUSION

(1) Different proportions of PANI/PPy/CC hydrogel electrodes were prepared. Through SEM test, it was found that the surface of PANI/PPy/CC hydrogel electrode was rougher, which facilitated the attachment of electricity-producing microorganisms. The water contact angles of unmodified, 1:2, 1:4 and 1:6 electrodes were 93.302 °, 71.434 °, 23.311 ° and 48.214 °, respectively. AC impedance were 3.86 Ω, 3.56 Ω, 3.24 Ω, 3.63 Ω; through comparative experiments, it can be seen that the contact angle of the 1:4 PANI/PPy/CC hydrogel electrode is smaller than that of the other groups of electrodes, the hydrophilicity is the best, the electron transfer impedance is the smallest, and the electron transfer between the electrode and the electrogenic microorganism is promoted. Therefore, 1:4 is the optimal doping ratio.

(2) In the performance test of MFC, it was found that the maximum output power of MFC under the four groups of unmodified, 1:2, 1:4 and 1:6 was 1848.75 mW/m^2,2002.05 mW/m^2,2187.5 mW/m^2 and 2019.6 mW/m^2, respectively. The maximum output voltages were 561.42 mV, 603.56 mV, 645.85 mV and 620.15 mV, respectively. The experimental results show that the MFC has the maximum output power density at the ratio of 1:4.

(3) There is a significant positive correlation between the output voltage of MFC and the external resistance. The external resistance decreases and the output voltage decreases. The reduced external resistance will increase the voltage loss and reduce the battery running time.

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