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Heat Treatment Effect on the Surface Properties of Carbon Cloth Electrode for Microbial Fuel Cell

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In this study, we investigate the effect of heat treatment on the surface properties of carbon cloth electrodes and on the power generation efficiencies of microbial fuel cells (MFCs) configured with the heat-treated carbon cloth electrodes. Water contact angle measurements show that the hydrophobic surfaces of the carbon cloth became super-hydrophilic after heat treatment at a temperature above 500 °C, making it suitable for bacterial propagation. X-ray photoelectron spectrometry revealed that the signal of the C-O functional group of the carbon cloth electrodes increased in intensity after heat treatment. The MFCs configured with heat-treated carbon cloth electrode exhibited high power density of 16.58 mW/m^2, whereas that of the untreated MFCs was only 8.86 mW m^2. Compared with chemical modifications, heat treatment does not use any environmentally unsound acidic or toxic solutions during modification and are promising for manufacturing large-scale MFC stacks.

Keywords: microbial fuel cell, heat treatment, surface properties, carbon cloth, super-hydrophilicity.

1. INTRODUCTION

In recent years sustainable and renewable energy sources have been widely investigated and developed to prevent and overcome the current energy crisis with fewer environmental concerns. Microbial fuel cells (MFCs) are potential renewable energy sources because they can convert biodegradable organic substances into energy by the action of electrochemically active bacteria during wastewater treatment [1 – 4]. These electrochemically active bacteria could generate bioelectricity in MFCs [5]. However, the amount of attached bacteria and the electrochemical performance of MFCs are closely related to the surface properties of the anodes [6, 7]. Therefore, it is crucial to choose appropriate anode material to enhance the power generation of MFCs. Carbonaceous electrodes, such as carbon paper, graphite rod, carbon cloth, carbon mesh, carbon felt, and carbon brush, have been demonstrated to be appropriate anode materials because of their high conductivity, biocompatibility, chemical stability, and low cost [8, 9]. Nevertheless, these carbonaceous electrodes normally exhibit hydrophobic surfaces, which impedes the colonization of electrochemically active bacteria and electron transfer from bacteria to anode.

To enhance the electron-transfer efficiency of MFCs, many researchers have modified the carbonaceous electrode surface through ammonia treatment [10], acid soaking [11], and electrochemical oxidation treatment [12, 13]. It has been demonstrated that coating oxides or nanomaterials on the surface of carbonaceous electrodes can improve the power generation efficiency of MFCs [14 – 20]. Besides, several chemical-free methods were also used to modify the surface properties of carbonaceous electrodes. Chang et al. reported that surface modification of carbon cloth electrodes simply by applying atmospheric-pressure plasma jets [21] or candle soot [22] could effectively increase the power-generation efficiency of MFCs. Yang et al. [23] demonstrated that heat pre-treatment at appropriate temperatures on the carbon brush anodes could also enhance the power generation of MFCs. The aim of this study was to investigate the surface properties of carbon cloth electrodes modified by heat-treatment at different temperatures using a normal furnace in an ambient atmosphere in terms of the power generation capability of the MFCs configured with heat-treated carbon cloth electrodes.

2. EXPERIMENTAL PROCEDURES

The construction of the MFCs used herein was described in detail elsewhere [21, 22]. The anodes and air cathodes of the MFCs were made of carbon cloths, purchasing from CeTech, Taiwan. The carbon cloth anodes were heat-treated by directly placing them in a pre-heated furnace set at different temperatures (400, 500, and 550 °C) and the heating time was 30 min. The heating time was set at 30 min because the heat-treatment effect was not significant when the carbon cloths were heat-treated at 500 °C for less than 30 min. On the other hand, some carbon cloths were damaged when they were heat-treated at 550 °C for more than 30 min. Besides, the carbon cloth was only heated to 550 °C because its surface was damaged when the heat-treated temperature was set at 600 °C. After heat treatment, the carbon cloths were cooled inside the furnace to room temperature and then used to construct the MFCs. The microbe used in this study was *Aeromonas hydrophila*. *Aeromonas hydrophila* was a kind of electrochemically active bacteria possessing the ability of dye decolorization and bioelectricity generation for MFCs applications [24 – 26]. The surface wettability values of the heat-treated carbon cloths were determined using a First Ten Ångstroms

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FTA125 contact-angle instrument. The surface chemical compositions of the heat-treated carbon cloth electrodes were evaluated using a Thermo Scientific (VGS) K-Alpha X-ray photoelectron spectrometer (XPS) with a monochromatic Al Kα radiation source (1468.6 eV). The C 1*s* and O 1*s* spectra of the carbon cloths were obtained in steps of 0.05 eV. The surface morphologies of the heat-treated carbon cloths after soaking in the MFC chambers were determined using scanning electron microscopy (SEM; Tescan 5136MM). The power density and polarization curves of the MFC were studied using a WonAtech ZIVE SP1 electrochemical workstation. The internal resistance of the MFC was determined by electrochemical impedance spectroscopy (EIS).

3. RESULTS AND DISCUSSION

3.1. Wettability measurements

Fig. 1 presents the determined water contact angle results of the untreated carbon cloth and those heat-treated at 400, 500, and 550 °C for 30 min. The untreated carbon cloth and the one heat-treated at 400 °C exhibited high water contact angle values of 131.5 ± 1.9° and 131.0 ± 2.4°, respectively, indicating that the surfaces of both carbon cloths are hydrophobic.The water contact angles of the carbon cloths heat-treated at 500 and 550 °C both approach zero, indicating that the carbon cloths became super-hydrophilic when the heat-treatment temperatures exceeded 500 °C. Super-hydrophilicity refers to the phenomenon of excess hydrophilicity. The contact angle of water is approaching to zero degrees on a super-hydrophilic surface. It has been reported that hydrophilic surfaces are easily colonized by electroactive microbes and are more suitable for electroactive biofilm formation [27, 28]. Therefore, bacteria colonization should be easier on the hydrophilic surface of carbon cloths heat-treated at 500 and 550 °C than on the hydrophobic surfaces of the untreated and 400 °C heat-treated carbon cloths.

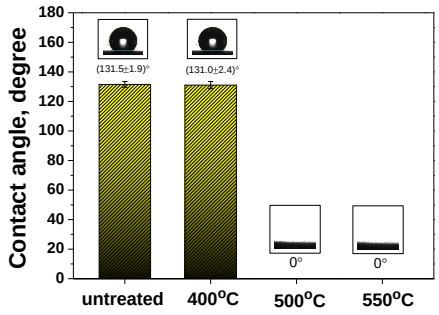

Fig. 1. Water contact angles of untreated and heat-treated carbon cloths

To evaluate the duration of the super-hydrophilicity of the heat-treated carbon cloths, they were exposed in an ambient environment (at 26 °C temperature and 60 % relative humidity) after the heat-treatment. Fig. 2 plot the water contact angle values of the untreated carbon cloth and of those heat-treated at 400, 500, and 550 °C for 30 min, respectively, as a function of standing time. Fig. 2 a and b show that the water contact angles of both the untreated and the 400 °C heat-treated carbon cloths were maintained at ~130° over 700 h. Fig. 2 c shows that the water contact angle of the 500 °C heat-treated carbon cloth was nearly 0° immediately after heat-treatment; however, its super-hydrophilicity deteriorates after exposure to ambient air for ~108 h, after which the water contact angle increased significantly and was maintained at ~130°. Fig. 2 d reveals that the 550 °C heat-treated carbon cloth had a water contact angle of ~0° immediately after heat-treatment, and its super-hydrophilicity was maintained for approximately 648 h.

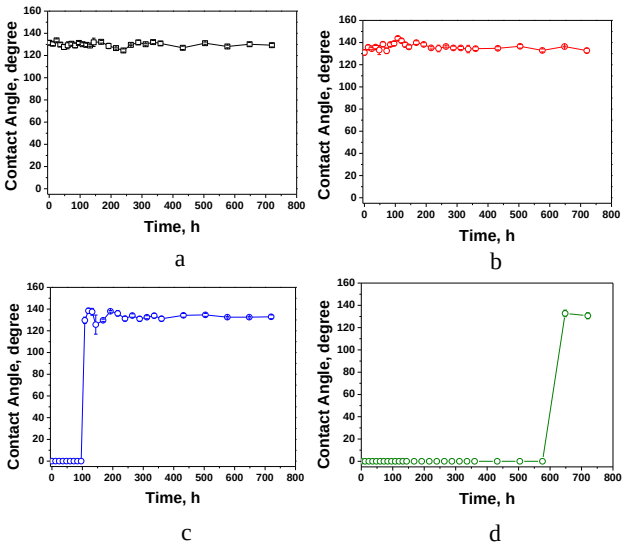

Fig. 2. Water contact angles: a – untreated carbon cloth electrode and those heat-treated at b – 400 °C; c – 500 °C; d – 550 °C as a function of exposure time in the ambient environment

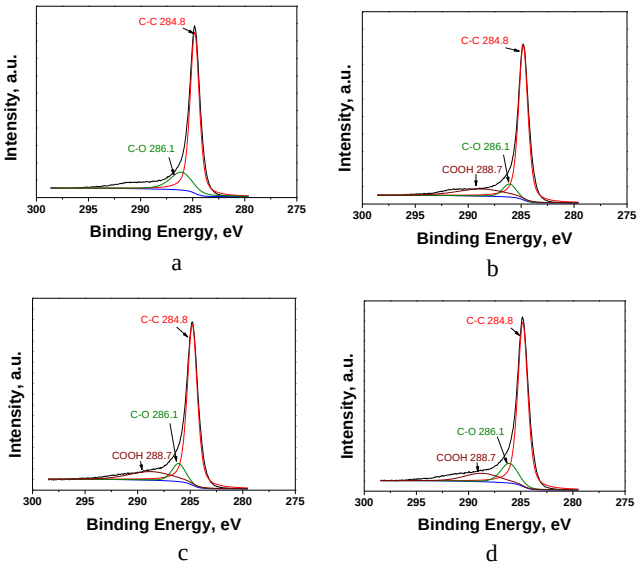

Fig. 3. XPS C 1*s* spectra of the surfaces: a – untreated carbon cloth electrode and those heat-treated at b – 400 °C; c – 500 °C; d – 550 °C

3.2. XPS measurements

Fig. 3 shows the C 1*s* XPS spectra of the untreated carbon cloth and those heat-treated at 400, 500, and 550 °C for 30 min, respectively. As shown in Fig. 3 a, the C 1*s* characteristic peak of the untreated carbon cloth can be deconvoluted into a major C–C peak at ~284.8 eV and a minor C–O peak at ~286.1 eV. Fig. 3 b reveals that the C 1*s*

characteristic peak of the 400 °C heat-treated carbon cloth also comprises C–C and C–O peaks, similar to those of the untreated carbon cloth; however, there an additional COOH peak appears at ~288.7 eV. Fig. 3 c and d show that the C 1*s* characteristic peaks of the carbon cloths heat-treated at 500 and 550 °C were similar to that of the one heat-treated at 400 °C.

Fig. 4 shows the O 1*s* high-resolution XPS spectra of the untreated carbon cloth and those heat-treated at 400, 500, and 550 °C for 30 min, respectively. Fig. 4 a reveals that the O 1*s* characteristic peak of the untreated carbon cloth was extremely weak. Fig. 4 b shows that the 400 °C heat-treated carbon cloth exhibited significant O 1*s* characteristic peaks, which could be deconvoluted into C–O, C=O, and C–OH peaks at ~531, 532, and 533 eV, respectively. Fig. 4 c and d show that the O 1*s* characteristic peaks of the 500 and 550 °C heat-treated carbon cloths were comparable to that of the 400 °C heat-treated carbon cloth, but had greater intensities. According to Fig. 4, it can be seen that the amount of C–O and COOH functional groups on the surface of carbon cloths could be effectively increased after heat treatments.

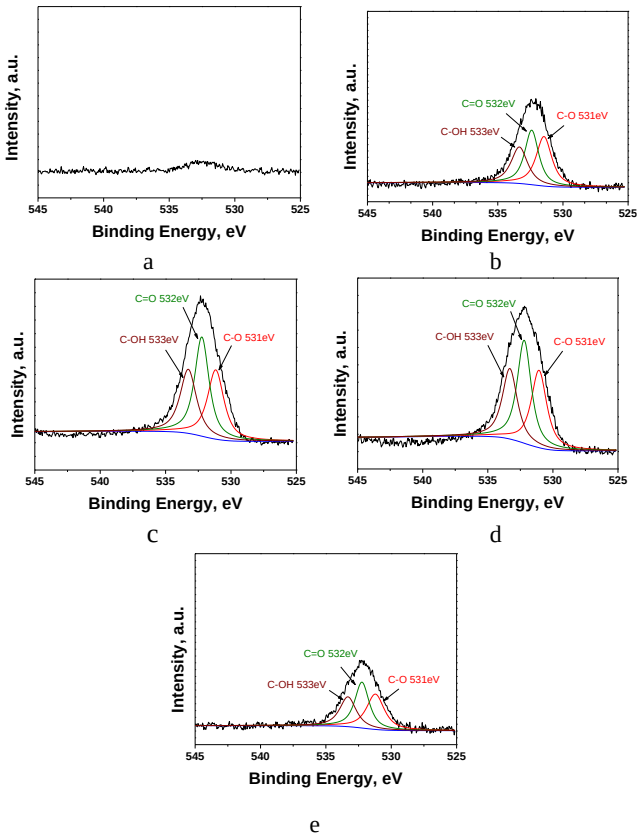

Fig. 4. XPS O 1*s* spectra of the surfaces: a – untreated carbon cloth electrode and those heat-treated at b – 400 °C; c – 500 °C; d – 550 °C; e – XPS O 1*s* spectrum of the carbon cloth heat-treated at 500 °C determined after exposure in ambient environment for 120 h

Fig. 4 e shows the O 1*s* characteristic peak of the 500 °C heat-treated carbon cloth exposed to ambient environment after 120 h. Compared with the XPS results shown in Fig. 4 b, Fig. 4 e shows that the intensity of the O 1*s* characteristic peak of the 500 °C heat-treated carbon cloth decreased, indicating that some C–O, C=O, and C–OH functional groups gradually disappeared during the exposure. Accordingly, we suggest that the C–O, C=O, and C–OH functional groups are more likely a physical adsorption on the surface of carbon cloth, rather than a strong chemical attachment. After 120 h exposure time, the amount of the residual oxygen-containing functional groups on the surface of 500 °C heat-treated carbon cloth was comparable to that of the hydrophobic 400 °C heat-treated carbon cloth. Since the 500 °C heat-treated carbon became hydrophobic after 120 h exposure time, we suggest that the heat-treated carbon cloth could maintain its super-hydrophilicity only if the amount of oxygen containing functional groups on the surface of carbon cloth exceeds a critical value. This explains why the contact angles of the 500 and 550 °C heat-treated carbon cloths were sudden increased after exposed for more than 108 h and 648 h in the ambient environment, respectively.

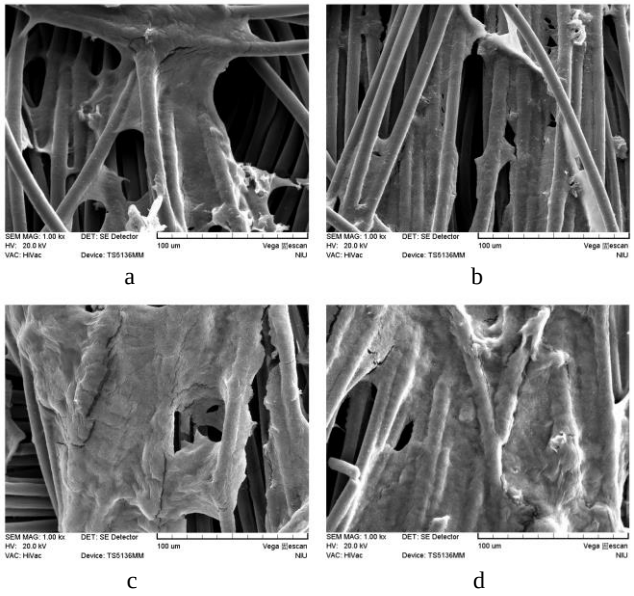

Fig. 5. SEM images: a – untreated carbon cloth electrode and those heat-treated at b – 400 °C; c – 500 °C; d – 550 °C after soaking in the chambers of MFCs for 24 h

3.3. Microbial colonization

Fig. 5 shows the SEM images of the untreated carbon cloth and those heat-treated at 400, 500, and 550 °C, respectively, after soaking in the MFC chambers for 24 h. Fig. 5 a and b show that some microorganisms and biofilm segments colonize on the surfaces of the untreated and 400 °C heat-treated carbon cloths. Fig. 5 c and d show that there are more abundant microorganisms on the surfaces of the 500 and 550 °C heat-treated carbon cloths. Fig. 5 demonstrates that heat treatments at 500 and 550 °C facilitates the colonization of microorganisms on the super-hydrophilic surface of the carbon cloth electrodes.

3.4. Power generation performance

Fig. 6 a presents the power density response curves of MFCs configured with electrodes of untreated carbon cloth and those heat-treated at 400, 500, and 550 °C. According to Fig. 6(a), the highest power densities of the MFCs were determined as 8.86 ± 0.34, 9.78 ± 0.10, 13.78 ± 0.60, and 16.58 ± 0.48 mW m^{-2}, respectively. Fig. 6 a demonstrates

that the power-generating efficiencies of MFCs could be improved by heat treatment at 500 or 550 °C. Fig. 6 b shows the EIS analysis results of the MFCs configured with untreated and heat-treated carbon cloth electrodes. As shown in Fig. 6 b, each MFC shows a single capacitive loop. These single capacitive loops could be fitted by the constant-phase-element (CPE) circuit model, comprising a CPE in parallel with a charge-transfer resistance (R_{CT}), as shown in Fig. 6 c. The impedance of the CPE can be calculated using $Z_{CPE} = \frac{1}{T(j\omega)^{\varphi}}$ [29]. The Z-View® software was adopted in this study for fitting the impedance of the CPE; φ is the CPE-P and T is the CPE-T. Table 1 lists the calculated values of R_S, CPE-T, CPE-P, and R_{CT} of the MFCs configured with the untreated and heat-treated carbon cloth electrodes. The R_{CT} values of the MFCs configured with the untreated carbon cloth electrode and with those heat-treated at 400, 500, and 550 °C were determined as ~1823, 1477, 1010, and 912 Ω, respectively. Since the R_{CT} value correlates to the resistance of the electron transfer efficiency from bacteria to the electrode [30], Fig. 6 b suggests that heat treatment at 500 or 550 °C could effectively improve the power generation efficiencies of the MFCs.

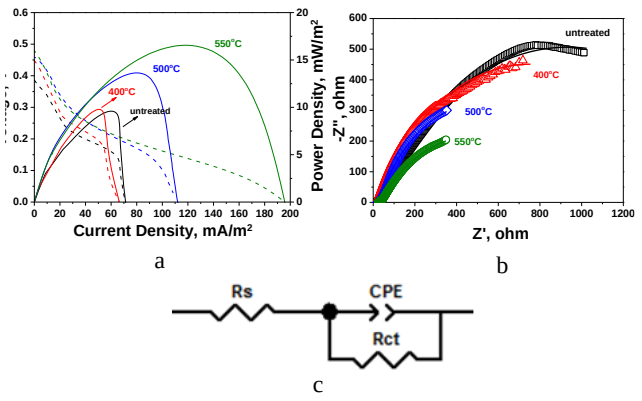

Fig. 6. a – power density response curves; b – EIS results of the untreated and heat-treated MFCs; c – equivalent circuit model

Table 1. R_S, CPE-T, CPE-P, and R_{CT} of the MFCs configured with the untreated carbon cloth electrode and those heat-treated at 400, 500, and 550 °C

Samples	*R*s, Ω	CPE-T	CPE-P	*R*ct, Ω
Untreated	29.52	0.0044	0.6459	1823
Heat-treated at 400 °C	27.78	0.0076	0.6980	1477
Heat-treated at 500 °C	21.81	0.0211	0.7309	1010
Heat-treated at 550 °C	15.77	0.0128	0.5938	912

According to the electrochemical results presented in Fig. 6, the MFCs configured with the 400, 500 or 550 °C heat-treated carbon cloth electrodes showed superior power densities and lower total internal resistances than those configured with the untreated carbon cloth electrode. Besides, experimental results demonstrated that the heat-treatment temperature has a significant impact on the electrochemical performances of the MFCs. Compared to those of the MFCs configured with the 500 and 550 °C heat-treated carbon cloth electrodes, the MFC configured with the 400 °C heat-treated carbon cloth electrode exhibited a lower power density of 9.78 ± 0.10 mW m^{-2} and a higher R_{CT} of 1477 Ω. This verified that insufficient heat-treatment temperature is not advantageous to the surface modification of the carbon cloth. The superior power generation efficiency of the 500 and 550 °C heat-treated modified MFCs was attributed to their super-hydrophilic surfaces, which is due to the presence of sufficient C–C, C–O, and C–OH functional groups on the surfaces of these heat-treated carbon cloths that favor bacteria colonization and biofilm formation. Additionally, the carboxyl functional groups formed on the surfaces of these heat-treated carbon cloths facilitate electrons transfer capability owing to the hydrogen bonding with the membrane-bound peptide bonds in bacterial cytochromes associated with intracellular electron transfer chain [13].

Fig. 6 also demonstrates that the power density of the MFC configured with the 550 °C heat-treated carbon cloth electrode (16.58 ± 0.48 mW m^{-2}) is higher than that of the MFC configured with the 500 °C heat-treated carbon cloth electrode (13.78 ± 0.60 mW m^{-2}). This is because of the presence of abundant hydrophilic functional groups on the surface of the 550 °C heat-treated carbon cloth. Consequently, the 550 °C heat-treated carbon cloth exhibited a considerably longer super-hydrophilicity duration time (648 h) than the 500 °C heat-treated carbon cloth (108 h). However, when the heat-treatment temperature was increased to 600 °C, the carbon cloth fractured and could not be used to configure the MFCs. According to our study, the heat treatment at appropriate temperatures can effectively facilitate bacterial colonization on the carbon cloth electrode, thereby increasing the power generation efficiencies of the MFCs.

4. CONCLUSIONS

This study demonstrated that the surface modification of carbon cloth electrodes through heating at appropriate temperatures using a normal furnace in an ambient air atmosphere can effectively improve the electrochemical performances of MFCs. Both the untreated and 400 °C heat-treated carbon cloths were hydrophobic, whereas, the 500 and 550 °C heat-treated carbon cloths were super-hydrophilic. The super-hydrophilicity of the 500 and 550 °C heat-treated carbon cloths can be attributed to the abundant formation of C–O and COOH functional groups on the carbon cloth surfaces. The amount of these functional groups gradually decreases over time, indicating that the oxygen containing functional groups were more likely a physical adsorption on the surface of carbon cloth, rather than a strong chemical attachment. The MFCs configured using the super-hydrophilic carbon cloth electrodes exhibited a superior electrochemical performance. According to our study, heat-treatment at approximately 550 °C for 30 min without the use of any chemicals is a rapid, cost-effective, and convenient surface modification method for carbon cloth electrodes used in MFCs.

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