

Interfacial Modification and Dispersion of Short Carbon Fiber and the Properties of Composite Papers as Gas Diffusion Layer for Proton Exchange Membrane Fuel Cell (PEMFC)

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Short carbon fibers (SCF) were modified with oxidation and coupling treatment to improve their water-wettability and bonding properties. Four types of dispersants were studied and discussed. Short carbon fibers/plant fiber (PF) composite papers were prepared by papermaking techniques. Scanning electron microscopy (SEM) and tests to determine zeta potential, absorbance, tensile index, and conductivity were carried out to investigate the modified effect of SCF and the interfacial properties. Modification experimental results showed that the surface grooves were deepened and new superficial grooves were generated by the liquid acid oxidation. The reaction with the silane coupling agent provided higher density and more uniform distribution on the SCF surface than that of organic titanate, and it obviously increased the roughness and the absolute value of zeta potential. After modification, the hydrophilic properties and dispersion in aqueous solutions were improved, the SCFs could form a good mechanical grip with plant fibers, and the conductivity and physical strength of SCF/PF composite papers were enhanced. It was shown that there was obvious adhesive binding at the fiber overlap nodes by the SEM analysis. It was confirmed that the improvement of physical properties of composite paper can be ascribed to the interfacial enhancement.

Keywords: Short carbon fiber; Interfacial modification; Plant fiber; Dispersion; Paper

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INTRODUCTION

A fuel cell is a power generation module that converts the chemical energy stored in the fuels and oxidants directly into power using a non-burning electrochemical reaction (Barbir 2005). Among the various fuel cells, the proton exchange membrane fuel cell (PEMFC) has been considered as an alternative power source for transport and stationary and portable fields (Barbir and Yazici 2008; Meidanshahi and Karimi 2012). The PEMFCs have captured much attention during the past few decades because of their advantages, which include high energy conversion efficiency, fast activation, low emissions, low noise, and low working temperature (Litster and McLean 2004; Wild *et al.* 2004).

A schematic diagram of the general configuration of a PEMFC is shown in Fig. 1. Electrodes for a PEMFC have, in general, two- or three-layered structures. A gas diffusion layer (GDL) is embedded between the catalyst layer and the gas flow channel in a PEMFC, providing a channel for the transport of fuel and the transmission of current. The GDL mostly functions as (i) a gas diffuser, (ii) a current collector, and (iii) a physical support, thus determining the catalyst utilization and the overall performance (Mathias *et al.* 2003; Williams *et al.* 2004; Barbir 2005). A GDL typically consists of a macroporous substrate.

The advantages of carbon fiber (CF), such as its high conductivity and corrosion resistance, make it well-suited for the special environment inside a PEMFC. Woven carbon cloth and carbon fiber paper are currently the most common materials used to produce GDLs because of their high gas permeability and electronic conductivity (Wang *et al.* 2007; Ko *et al.* 2008). Although carbon fiber fabric is prone to warping and shrinking in fuel cells, carbon fiber paper offers excellent size stability.

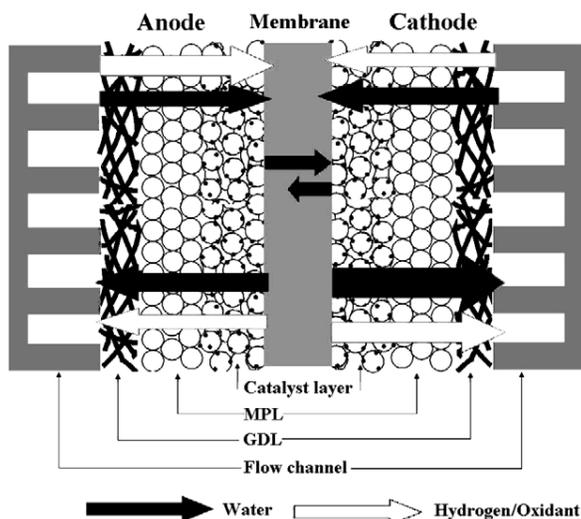


Fig. 1. Schematic diagram of a PEMFC

However, CFs have smooth surfaces, inferior wettability, and less chemically active groups, which show poor dispersion and weak bonding (Edie 1998; Ageorges *et al.* 1999; He 2004). There are thus almost no chemical bonds or effective physical interactions, such as van der Waals attraction, hydrogen bonding, and mechanical interlocking, between CF and other fibers. When CFs are applied without previous surface treatment, an increase in the carbon fiber fraction leads to both conductivity enhancement and mechanical performance reduction.

It is generally accepted that the modification of CFs can improve the interfacial properties by creating mechanical interlocking, increasing the fiber surface area, and generating chemical bonding (Pittman *et al.* 1997; Ma *et al.* 2014). These characteristics are directly affected by the content of polar functional groups, and chemical bonding plays the most important role in the interfacial adhesion in most situations (Arash *et al.* 2010; Zhao and Huang 2011). In recent years, extensive research has centered around the introduction of additional oxygenated species (*e.g.* COOH, OH, and C=O) onto the fiber surface to increase hydrophilicity, enhance strong non-covalent interactions, or introduce repulsion force among fibers through processes such as oxidation treatment (Zielke *et al.* 1996; Pittman *et al.* 1997), chemical grafting (Peng *et al.* 2012; Chukov *et al.* 2014), electrochemistry (Cao *et al.* 2005; Ishifune *et al.* 2005), plasma treatment (Ma *et al.* 2011; Park *et al.* 2012), and high-energy irradiation (Xu *et al.* 2007). Improving the interface properties of CFs effectively and conveniently is still a challenge and is dependent on the functional groups at the surface (Zhao and Huang 2011; Liu *et al.* 2013; Ma *et al.* 2014).

As a result, there is an urgent need to find a balance between the mechanical properties and the electrical conductivity. This paper focused on the interfacial modification of SCF combining oxidation treatment and coupling treatment in an attempt

to improve the interfacial properties between SCF and PF. The relationship between the properties of composite papers and the SCF fraction was systematically investigated. The aim is to improve the performance of SCF/PF composite papers and meet the requirement of GDL of PEMFC.

EXPERIMENTAL

Materials

Short carbon fibers with an average diameter of approximately 8 μm and length of 3mm were obtained from the Japanese Toho Co. Ltd.(Japan). Bleached soft wood pulp for the plant fiber was provided by the Yongtai Paper-Making Mill (China). Silane coupling agent (KH-550) and organic titanate coupling agent (TMC-102) were supplied by the Hangzhou Feidian Coupling Agent Co., Ltd. (China).Sulfuric acid (H_2SO_4), potassium chlorate (KClO_3), sodium dodecyl sulfate(SDS), cetyltrimethyl ammonium bromide (CTAB), non-ionic surfactant Polysorbate 80(PS-80), cationic polyacrylamide(CPAM), and other chemicals were analytical reagents and supplied by Hangzhou Huipu Chemical Agent Co., Ltd. (China).

Methods

Interfacial modification

For liquid acid oxidation, the SCFs were first washed in acetone to remove surface adhesives and contamination and then dried at 80 $^\circ\text{C}$. The oxidation was achieved by heating SCFs in 40 wt% (weight concentration) H_2SO_4 and 15 wt% KClO_3 at 85 $^\circ\text{C}$ for 90 min. After oxidation, the modified SCFs were thoroughly washed with distilled water, and then dried at 50 $^\circ\text{C}$. Under heating, the oxidation agent reaction was as follows:



The gas thermal oxidative modification was carried out at 400 $^\circ\text{C}$, and isothermal holding was performed for 60 min in a static air atmosphere.

For the coupling agent treatment, a silane coupling agent and organic titanate coupling agent were used. The coupling agent aqueous solution was mixed at 1 wt% consistency. After the coupling agent hydrolyzed, the modified SCF was dipped in the hydrolyzed silane solution for 4 h, and then dried at 80 $^\circ\text{C}$ for 24 h.

Sample preparation

The SCFs, PFs, and agents were mixed at different ratios with a certain consistency and dispersed by a high-shear dispersion device. Some suspension samples were put aside, and the supernatants were taken to measure absorbance; some suspension samples were tested to determine the zeta potential, and some samples were used to form paper sheets in a rapid handsheet former.

Fiber suspensions were prepared according to the proportions that are shown in Table 1.

Table 1. Composition of Fiber Suspension

Item	Short carbon fibers (wt%)	Plant fibers (wt%)	Dispersant agent (wt%)	Concentration (wt%)
Ratio	40	60	0.3	0.1

Sample testing

The SCFs and paper sheets were analyzed by scanning electron microscopy (SEM) (SIRION, FEI Co. Ltd., Netherlands) with acceleration voltage of 15 kV. Proper portions of the SCF suspensions were subjected to zeta (ζ) potential measurements using an FPA zeta potential detector (AFG Co. Ltd., German), which operates according to the streaming potential principle. The supernatants were taken to determine the absorbance using a UV-visible spectrophotometer at the wavelength of 650nm. The physical strength of paper handsheets was determined following the TAPPI standard methods (TAPPI T494 om-13, Tensile properties of paper and paperboard). The conductivity of the paper was measured by a MS8218 precision meter (Shenzhen Huayi instrument Co. Ltd., China), and the square resistance was calculated.

RESULTS AND DISCUSSION

Oxidative Modification

The surface morphology of the SCF was investigated by SEM. Images of untreated and oxidized fibers are shown in Fig. 2. The zeta potential, absorbance values, and conductivity of the papersheets are shown in Table 1. Gas thermal oxidation and liquid acid oxidation were compared according to the SEM images and the data in Table 2.

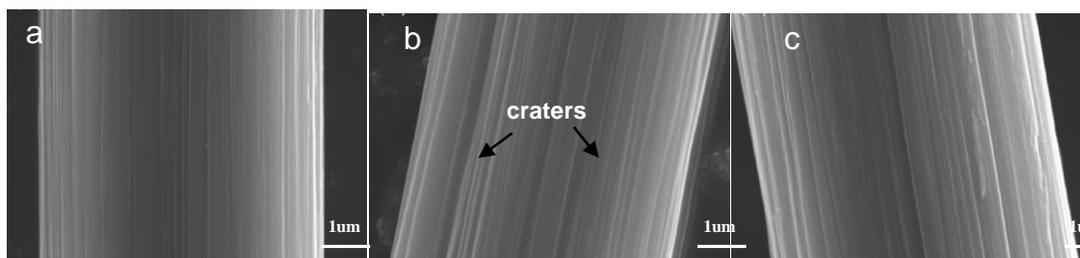


Fig. 2. SEM images of (a) untreated SCF, (b) SCF after isothermal holding at 400 °C for 1 h, and (c) SCF after H₂SO₄/KClO₃ treatment for 1.5h

As expected, SCF before modification presented a comparatively smooth surface with few defects (Fig. 2a). This would certainly have detrimental effects on good interfacial performance because of the formation of weak bonds between SCF and CF. Oxidative treatment changed the surface morphology of SCF significantly. The grooves were caused by the removal of amorphous carbon and defective layers on the borders of fibers, as reported by Chukov *et al.* (2014). As a result, the exfoliation of fibrous bands began. The thermal oxidation of fibers resulted in a well-developed surface with the formation of craters with sizes of less than 1 μm (Fig. 2b), pointing to etching with more graphite structures. Comparing Figs. 2b and 2c, the surface grooves were deepened and new superficial grooves were generated by the liquid acid oxidation. Thus, the changes in the morphology of the SCF surface were more pronounced and the liquid oxidation had a stronger effect on the fiber structure than the thermal oxidation. These changes helped to increase surface area and enhance the mechanical interlocking effect, leading to good mechanical binding between SCF and PF.

Table 1 shows that there was a strong increase in the absolute value of zeta potential, indicating effective change on the surface after oxidative modification. The value

of the zeta potential of the suspension became more negative by 21.2 mV, or 160%, with liquid modification, a result that depended on the functional groups at the surface and aided the dispersion of SCF. Meanwhile, a substantial enhancement in absorbance was observed, and the value increased by 73%. Higher absorbance can indirectly lead to better dispersion conditions. Good dispersion of SCF would aid in the formation of a conductive network and improve the conductivity of composite paper. Gas thermal oxidation created a hole in the carbon fiber surface and etched the trench to increase the roughness. The liquid acid oxidation of carbon fibers increased the surface oxygen content, mostly because of the increase in hydroxyl and carbonyl groups, as described in the literature (Zielke *et al.* 1996; Pittman *et al.* 1997). Because these oxygen-containing functional groups were increased, they compensated for micro-cracks on the carbon fiber surface to some extent. These functional groups were ionized in water, increasing the electric double layer on the carbon fiber surface and the zeta potential absolute value.

Modification with Coupling Agent

Based on liquid-phase oxidative modification, the carbon fibers were further modified with coupling agent.

Table 2. Effect of Different Modification Methods

Modified method	Untreated	Gas oxidation	Liquid oxidation	TMC-102	KH-550
Zeta potential(mV)	-13.5	-27.4	-31.7	-40.3	-45.9
Absorbance	0.243	0.340	0.421	0.481	0.542
Square resistance(Ω/\square)	572.36	422.12	396.41	378.65	346.32

It can be seen from Table 2 that, after coupling modification with KH-550 and TMC-102, both the absolute value of zeta potential and the absorbance increased obviously. The absolute value of zeta potential with KH-550 treatment increased 14.2 mV (45%), and the absorbance improved by 29%. This indicated that coupling modification could change the double-layer of the fiber, and the dispersion of SCF was improved further. The active groups of coupling agent could be hydrolyzed and combined with the remnants of hydroxyl and carboxyl groups on the carbon fiber surface in the solution, as reported in the literature (Liu *et al.* 2013). It is inferred that the coupling agent became attached to the carbon fiber surface and formed a bridge between inorganic SCF and organic PF, improving the lyophilic performance and the dispersion of SCF. As for the electrical conduction of paper, the square resistance of liquid oxidation was lower than that of gas oxidation. After coupling modification with KH-550 and TMC-102, the square resistance could be decreased further.

The surface morphology of SCFs, including liquid acid oxidation and additional coupling agent treatment, is shown in Fig. 3. After coupling, the surface morphology of SCF changed significantly (Figs. 3e and 3f). There were still wide and deep grooves along the axial direction of the SCF both before and after coupling, which means that coupling modification did not fill the grooves of the fiber surface because of the amount of the absorption. Prior to the coupling process, the oxidation treatment introduced some polar functional groups on the surface of SCF (Park *et al.* 2012; Servinis *et al.* 2013). For instance, carboxyl groups on the surface of SCF can form hydrogen bonds with new functional groups and provide a firm attachment of the coupling agent to the fiber surface. To compare

Figs. 3e and 3f, KH-550 provided higher density and more uniform distribution on the SCF surface. The interaction between SCFs and PFs by KH-550 coupling was higher than that of TMC-102. This result can be verified by the square resistance data.

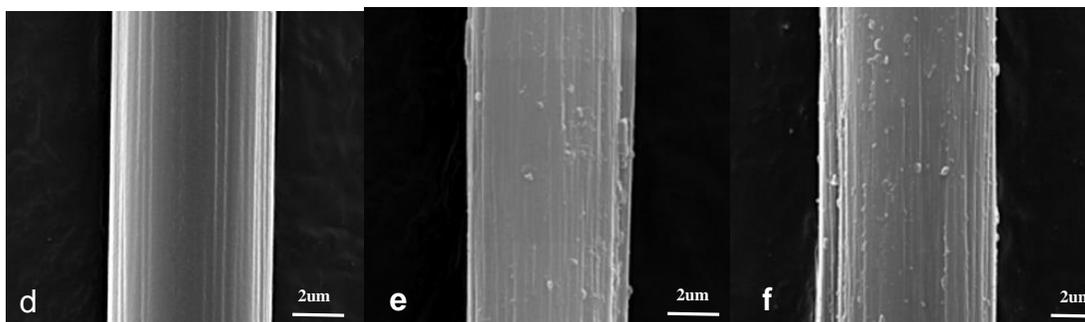


Fig. 3. SEM images of (d) SCF after liquid acid oxidation, (e) TMC-102 treatment, and (f) KH-550 treatment

Effect of Dispersant

Figure 4 shows that without dispersants, there was flocculation and a significant layering phenomenon in the SCF suspension. When SDS was added (Fig. 4e), the layering phenomenon of the SCF was weakened, but the flocculation increased. When CTAB (Fig. 4d) was added, the layering phenomenon of the SCF almost disappeared, while there was still fiber flocculation. When PS-80 (Fig. 4b) was added, the carbon fiber was evenly dispersed, and there was almost no flocculation. However, after a short duration, the suspension displayed an uneven deposition. When CPAM (Fig. 4c) was added, the carbon fiber quickly formed small regular areas of flocculation, and the whole suspension was uniformly distributed. Meanwhile, the deposition was also even and the deposition time doubled.

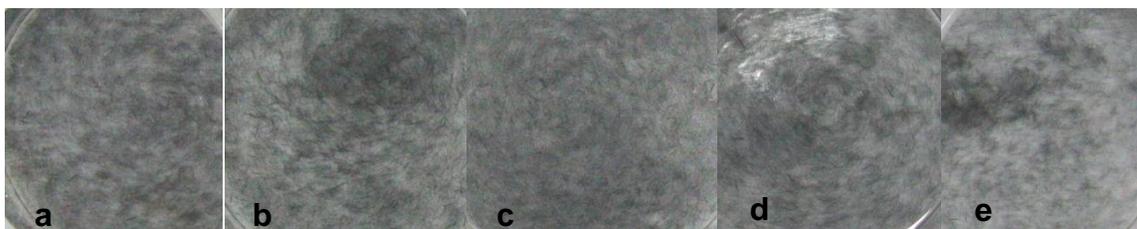


Fig. 4. Dispersants applied to SCF suspensions; (a) SCF without dispersant, (b) SCF treated with PS-80, (c) SCF treated with CPAM, (d) SCF treated with CTAB, and (e) SCF treated with SDS

Figure 5 shows that, taking into account the corresponding papersheet surface morphology, the number of black clumps of SCF after the addition of CTAB (Fig. 5d) was slightly reduced, while the number of black clumps by adding SDS (Fig. 5e) increased. The papersheets with added CPAM (Fig. 5c) and PS-80 showed a uniform formation, with almost no black clumps on the paper.

It can be seen from Table 2 that the absolute value of the zeta potential was minimal by adding SDS, and least negative after adding the cationic surfactant CTAB. According to the charge-control theory, the dispersion of ionic surfactants increases with increasing zeta potential. This can explain the function of CTAB and SDS. However, there was an unusual phenomenon in the experiments. Although the value of the zeta potential

was reduced by adding CPAM, the dispersion of the SCFs and the uniformity of papersheets were excellent. Therefore, the charge-control theory may not apply to CPAM. It was discovered that when CPAM was added, the carbon fibers quickly formed a large number of micro flocculations, and the micro flocculations maintained a regular distribution for some time. Thus, at the macro level the SCFs can show a better dispersion. The addition of PS-80, a non-ionic surfactant, changed the value of the zeta potential. The PS-80 improved the dispersion performance of SCFs greatly because of its more hydrophilic groups, *i.e.*, polyoxyethylene. The SCFs were covered with PS-80, and its hydrophilicity was enhanced significantly.

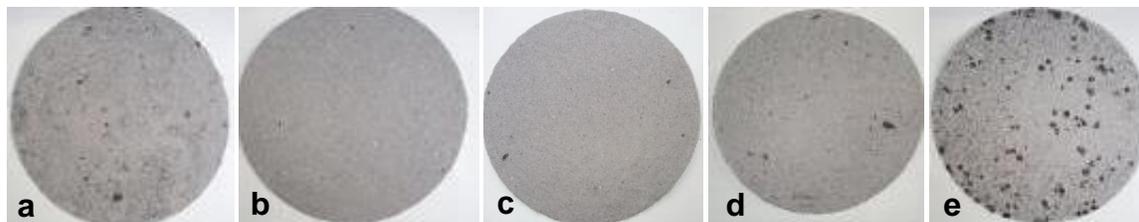


Fig. 5. The carbon fiber composite paper-sheets with different dispersants. (a) SCF without dispersant, (b) SCF treated with PS-80 (c) SCF treated with CPAM, (d) SCF treated with CTAB, and (e) SCF treated with SDS

Table 3. Characteristics of SCF Suspension with Different Dispersants

Item	Untreated SCF	PS-80	CPAM	CTAB	SDS
Zeta potential (mV)	-13.5	-34.2	-22.9	-3.92	-18.4
Absorbance	0.243	0.384	0.421	0.336	0.272
Tensile index (N·m/g)	15.53	21.37	23.83	18.75	13.12
Square resistance (Ω/\square)	572.36	414.58	397.68	472.36	665.71

The absorbance results coincided with the suspension conditions in Fig. 3. The absorbance value of CPAM was the highest, followed by PS-80 followed, and the value with SDS was the least. The dispersion performance increased with increasing absorbance value. The tensile index was determined using the sheet surface morphology in Fig. 3. When the fibers were dispersed more uniformly, the physical strength performance of the paper sheets was improved. There were no black clumps on the paper after adding CPAM or PS-80, and the physical strength value was the highest.

Considering the above experiments, SCFs were first modified by liquid oxidation and coupling treatment, then SCFs and PFs were mixed with CPAM and dispersed by the dispersion device. The pulp was used to form papersheets. The surface morphology of the paper was investigated by SEM, as shown in Fig. 6.

Figure 6 reveals that SCFs and PFs were distributed uniformly, forming a continuous network and porous structure. There was obvious adhesive binding at the fiber overlap nodes. The improvement in the physical properties of composite paper can be attributed to the binding reinforcement. The results in Table 3 also showed that with the addition of SCF, the composite papers exhibited enhanced conductivity.

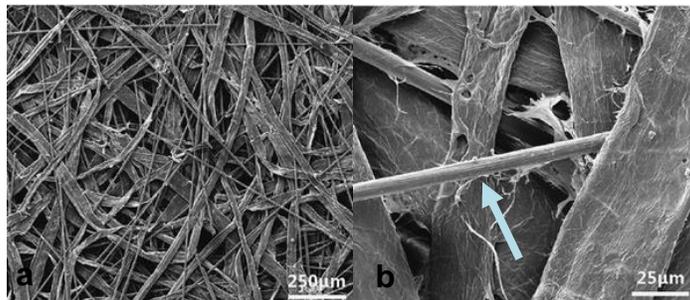


Fig. 6. SEM images of the SCF / PF composite papers (40 wt% of SCF)

CONCLUSIONS

1. Oxidation modification can change the surface morphology of SCFs and improve the hydrophilic properties and dispersion. The surface grooves were deepened and new superficial grooves were generated by liquid acid oxidization. The absolute value of the zeta potential of the suspension increased by 21.2 mV (160%) with liquid modification, which greatly benefit the dispersion of SCFs. Meanwhile, the value of absorbance increased by 73%, and higher absorbance can indirectly lead to better dispersion conditions. Good dispersion of SCFs benefit the formation of conductive networks and improve the conductivity of composite papers.
2. The KH-550 was coated uniformly on the fiber surface through chemical reaction, and it increased the fiber surface polarity and roughness. The absolute value of the zeta potential after KH-550 treatment increased by 14.2 mV (45%), and the absorbance increased by 29%. The KH-550 provided higher density and more uniform distribution on the SCF surface. The interaction between SCFs and PFs was higher with KH-550 than it was with TMC-102. This indicated that the SCF dispersion improved further. The active groups of the coupling agent can be hydrolyzed and combine with the remnants of hydroxyl and carboxyl groups on the carbon fiber surface in solution.
3. There was much flocculation and a significant delamination in the SCF suspension without additives. Papersheets with the addition of CPAM and PS-80 showed a uniform formation, and there were almost no black clumps in the paper. Although the value of the zeta potential with CPAM was markedly reduced, the dispersion of SCF and the uniformity of the papersheets were excellent. Therefore, the charge-control theory was not suited for CPAM. It was discovered that when CPAM was added, the carbon fibers formed a large number of micro flocculations quickly, and the micro flocculations retained a regular distribution for quite some time. Thus at the macro level the SCFs showed a better dispersion.
4. The SEM analysis revealed that SCFs and PFs were distributed uniformly and formed a continuous network and porous structure. There was obvious adhesive binding at the fiber overlap nodes. The improvement in the physical properties of composite paper can be attributed to the binding reinforcement.

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