Composites Science and Technology 153 (2017) 1–6

Contents lists available at ScienceDirect

Composites Science and Technology

journal homepage: http://www.elsevier.com/locate/compscitech

A flexible and transparent thin film heater based on a carbon fiber /heat-resistant cellulose composite

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ARTICLE INFO

Article history:
Received 18 July 2017
Received in revised form
5 September 2017
Accepted 11 September 2017
Available online 29 September 2017

Keywords:
Carbon fibers
Flexible composites
Mechanical properties
Thermal properties

ABSTRACT

The thin flexible film heater made of carbon fibers is widely considered to be an ideal material for the use as self-heating devices because of its safe, low-cost, no noises, small size and fast heating as well as energy saving. Presently thin flexible film heater is mostly fabricated by mixing method using the long cellulose fibers as film-forming materials and carbon fibers as self-heating materials, which mostly suffer from opaque or uneven heating field. In this work, we firstly reported a flexible and transparent thin film heater (FTFH) composed of carbon fibers and regenerated cellulose. The use of regenerated cellulose for membrane materials brings high transmittance, strong adhesion, fast temperature response and high generated temperature. More importantly, the FTFH using novel carbon fibers as self-heating materials and regenerated cellulose as membrane materials show a rapid heating response (12 s), higher power density (2577 w/m^2) and long-term stability of generated temperature (162.3 °C).

1. Introduction

In recent years, design and fabrication of FTFH have attracted significant attentions from both academic and industrial interests [1]. Due to high efficiency in converting electric energy into heat energy, FTFH is widely used in snow-cleaning device, thermal therapies or heating outdoor displays [2–4]. Several emerging materials, including indium tin oxide (ITO), carbon nanotube, metal grids, and graphene have been studied as self-heating material in FTFH [5–8]. However, their inferior performance and high-cost limit its further application in the next-generation devices. Compared to the other self-heating material in FTFH, carbon fibers were widely considered to be an ideal self-heating materials for use in FTFH because of its high generated temperature, low density, high strength, easy processing and low-cost [9]. In particular, much effort has been focused on incorporating carbon fibers into membrane materials, such as cellulose and resin [10,11]. Compared with the other thin flexible film heater, carbon fibers paper (CFP) has been widely used as the thin flexible film heater due to its easy handling, low-cost and high heat generation efficiency [12]. Whereas, research efforts at CFP have encountered numerous problems because the long carbon fibers with high-surface-energy made the CFP opaque and uneven temperature field [13]. Among known strategies for FTFH based on a carbon fiber as the self-heating material, the main membrane materials for it are resin or plant fiber [14]. However, there are several obstacles for these membrane materials to overcome before wide application in thin flexible film heater. For example, the resin can be easily fabricated as a uniform film, but the properties of resin made the thin flexible film heater inflexible [15]. The thin flexible film heater using plant fiber as membrane materials have shown excellent heating performance. However, the poor adhesion of carbon fiber network with the plant fibers made the thin flexible film heater poor mechanical properties and the long fibers made it opaque [16]. Yet, the flexible and transparent thin film heater with high transmittance (>90%), strong adhesion and fast temperature response can be obtained by means of comprising Ag nanowires (NWs) in an ultrathin polyimide (PI) foil, the process of this flexible and transparent thin film heater is complex and the cost is huge [17]. Contrary to the other membrane materials, the regenerated cellulose is advantageous for practical applications of the flexible and transparent thin film heater, since it will lead to much higher transparency and tensile strength [18]. For this reason, the work presented herein is devoted to obtaining a flexible and transparent thin film heater based on the carbon fiber/heat-resistant cellulose composite by using a very simple and low-cost method.

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https://doi.org/10.1016/j.compscitech.2017.09.033
0266-3538/

In this paper, we reported a FTFH using regenerated cellulose as a membrane material and carbon fibers as self-heating material. Our FTFH has the following advantages over previously reported materials: (1) The FTFH exhibited uniform heating and rapid thermal response because the regenerated cellulose with a large number of hydroxyl made the carbon fibers disperse evenly. (2) Tensile property and optical transmission have been greatly improved owing to the regenerated cellulose with small size. (3) The FTFH can reach to higher power density and generated temperature.

2. Materials and methods

2.1. Materials

Commercial eucalyptus dissolving pulp was used as the raw cellulose source material was purchased from Guangzhou Chenhui Paper Co., Ltd. (China). The 6 mm-length carbon fibers were provided by Shanghai Lishuo Co., Ltd. (China); N, N-Dimethylacetamide (DMAC), lithium chloride (LiCl), supplied by Aladdin-Reagent Co., Ltd. (Shanghai China). Commercial eucalyptus dissolving pulp, DMAC, LiCl, acetone, were used to prepare regenerated cellulose. All the reagents were used as received without further purification.

2.2. The dissolution of regenerated cellulose

The solvent of cellulose was synthesized by according previously reported literature [19]. A specified amount of eucalyptus dissolving pulp was pre-treated by immersing it in acetone for 5 min at 25 °C and subsequent dissolution of cellulose in 8% LiCl/DMAc (wt/v) solvent through continuous stirring under the anhydrous condition. The process to fully dissolve the cellulose fibers in the LiCl/DMAc took about 3 h at 40 °C. Then the cellulose solution was added dropwise into the stirring deionized water at a rate of 20 ml/min to produce the regenerated cellulose. The deionized water was stirred at 5000 r/min to generate strong shear forces and prevent agglomeration using an emulsification machine (Shanghai Yingdi Automation Inc., China). By doing this, the solution of regenerated cellulose was obtained.

2.3. Fabrication of FTFH

The method of fabrication for FTFH is briefly described as below. The solution of regenerated cellulose and carbon fibers were dispersed evenly in deionized water by agitating the mixture using a mixer (Shanghai Yingdi Automation Inc., China). The FTFH was prepared by filtering above mixture onto a glass filter with a nylon fabric membrane (pore size: 38 μm). The obtained wet FTFH was repeatedly washed with deionized water to eliminate the residual LiCl or DMAc and then was sandwiched between two stacks of regular filter paper to dry under o.4 MPa pressure at 110 °C for 5 min. The FTFH with base density of 37 g/m^2 was obtained using the above-mentioned method.

2.4. Characterizations

The structure of the FTFH was studied with a scanning electron microscope (SEM). JEM-100CXII at an accelerating voltage of 10 kV. Prior to the examination, the surface of the specimen was coated with a thin layer of gold, ~20 nm.

A KajaaniFS300 Fiber Analyzer was used to quantitatively analyze the dimensions of the regenerated cellulose fibers in the DI water solution and the original cellulose fibers in DI water solution.

The mechanical strength of samples was measured using a universal tensile tester (Instron5565, Instron instruments Inc. USA). Samples were firstly cut to 15.0 mm × 50.0 mm and placed in constant temperature and humidity chamber at (50 ± 1) % relative humidity (RH) and (23 ± 1) °C for 24 h to ensure the stabilization of their water content before characterization.

UV-Vis spectrometer with an integrating sphere (UV-9000 Shanghai Yuanyi Inc. China) with an integrating sphere was used to measure the total transmittance of the FTFH in a wavelength range of 400 nm−900 nm.

The resistances of the FTFH were recorded by a multimeter (VC890D, China). Samples were firstly cut to 80.0 mm × 80.0 mm and placed in constant temperature and humidity chamber at (50 ± 1) % relative humidity (RH) and (23 ± 1) °C for 24 h to ensure the stabilization of their water content before characterization.

The temperatures and heat distributions of the FTFH were measured using CEM infrared thermal image (DT-980, China). Samples were firstly cut to 80.0 mm × 80.0 mm and placed in constant temperature and humidity chamber at (50 ± 1) % relative humidity (RH) and (23 ± 1) °C for 24 h to ensure the stabilization of their water content before characterization. The applied DC voltage was supplied by a power supply (HY3005ET, China) to the heater through two copper conductive tapes pasted at the FTFH edges.

3. Results and discussion

3.1. Reaction principle analysis

Cellulose is ideal membrane materials for FTFH due to its easy processing, low-cost, good chemical and biodegrading properties [20]. However, since the original CFP is made of cellulose fibers with diameters of ~20 mm, there are intrinsic disadvantages regarding original CFP. The difference in refractive index between air in pores and the cellulose fibers results in the opaque of the original CFP [21]. The long cellulose fibers also limit its overall mechanical properties due to the tiny amounts of hydrogen bonds. Compared to the CFP made of long cellulose fibers, the FTFH made of regenerated cellulose is recently attracting great attentions by providing excellent mechanical properties, optical transparency [22]. A homogeneous cellulose solution was obtained for preparing the FTFH [23–25]. Subsequently, the regenerated cellulose was obtained by a debonding procedure. The obtained cellulose solution was slowly dropped into the stirring deionized water under the action of high-speed shear force. The hydrogen bond network between the cellulose chains broke again due to the attack from water molecules. The dissolution of regenerated cellulose was prepared because of the interactions between the solvent [26]. Afterwards, the carbon fibers with an average length of 6 mm were homogeneously dispersed in the solution of regenerated cellulose. The dispersing performance of carbon fibers is usually unsatisfactory due to carbon fibers with less active groups [27]. In our work, the solution of regenerated cellulose provides an excellent environment for dispersion of carbon fibers because regenerated cellulose has many hydroxyl bonds. The solution with many hydroxyls made the carbon fibers more difficult to flocculate. After that, the wet FTFH was obtained by random deposition of the nylon fabric membrane through vacuum filtration. The FTFH have homogeneous structures, glossy and excellent mechanical properties with acceptable elastic properties. The reasons for that is the regenerated cellulose with many hydroxyl bonds is considered as the adhesive to bond the neighboring carbon fibers. It was noted that the produced fines (fiber length less than 200 μm) in the regenerate process increased the packing density and filled in the voids between carbon fibers, which undoubtedly increased the packing area and caused the FTFH to exhibit high mechanical strength, acceptable elastic properties and transparency (Fig. 1).

3.2. Properties of transparent paper

In order to investigate the properties of the samples, the structure of the samples was examined with a scanning electron microscope (SEM). The differences from the ideal structure of original paper to the FTFH samples were distinctly observed from SEM images by scanning cross-sections and fracture surface. The original paper was loosely composed of large pores and long cellulose fibers. The difference in refractive index between air in pores and the basic cellulose results in the opaque of the original paper [28]. The images of original paper made by commercial eucalyptus dissolving pulp through the traditional method were shown (Fig. 2a–b). Compared with the images of the film only made from regenerated cellulose (RCF), there is a significant difference between two kinds of film. The film only made from regenerated cellulose (RCF) has no obvious pores on the surface as shown in the SEM image (Fig. 2c). The flat smooth surface of the RCF indicated that the regenerated cellulose is small in size. The regenerated cellulose with small size is highly desirable for achieving good optical and mechanical properties of the resulting thin flexible heater. This RCF also is denser than the original paper sample because the space between regenerated cellulose is smaller than the long cellulose fibers. The images of FTFH were shown (Fig. 2e–f). It is clear that the carbon fibers and the regenerated cellulose are well combined with each other. The regenerated cellulose in small size filled out the pores between the carbon fibers and glued on the skeleton of the carbon fibers made the thin flexible film heater is more compact [29]. When the dosage of carbon fibers increased, this made the crossed network in the film more intensive, which led to the increase in the generated temperature while the transmittance declined for the FTFH.

The fiber dimensions for both regenerated cellulose fibers and original pulp fibers were presented in Table 1. The regenerated cellulose fibers have an average fiber length of 0.84 mm and an average fiber width of 12.09 μm, which is slightly smaller than original pulp fibers. Regenerated cellulose fibers have a higher curl index (24.56%) but a lower kink index (1063.58 1/m) compared to original pulp fibers, which explain the curvy and weavable nature of the regenerated cellulose fibers. The fine content of the regenerated cellulose fibers (fiber length less than 200 μm, 32.33%) is much than higher than that of original pulp fibers (8.31%). Therefore, the basic hydroxyl groups on the surface of the regenerated cellulose fibers enable them to bond tightly through hydrogen bonding pathways, which is similar to the original paper and nanopaper.

The optical properties of RCF, the FTFH-4% and the FTFH-8% were compared with a conductive nanopaper [30] (Fig. 3c). The transmittance of the RCF made by regenerated cellulose at 550 nm reaches up to 63%, which is lower than the conductive nanopaper (81%). The conductive nanopaper is a transparent film made of network-forming nanocellulose fibers and Ag nanowire. These materials are several micrometers long with a diameter of 4–50 nm [31]. As the fiber diameter decreases, the optical transmittance of the film increases [32]. In comparison, although the transmittance of the RCF is lower than the conductive nanopaper, the method of fabrication RCF is greatly easier than the conductive nanopaper and the cost is lower. The meaning of the FTFH-4% is that the sample contains 4% carbon fiber, and the same suits for FTFH-8%. As the content of carbon fibers increases, the optical transmittance of the FTFH decreases. When the content of the carbon fibers is 8%, the transmittance of the film decreases to 16%. The reason for the decrease in transparency of FTFH is that the light is hard to penetrate through carbon fibers network.

The mechanical properties of FTFH play a significant role for the comprehensive device. To examine the suitability of the samples for industrial applications, the mechanical properties of the original paper, the RCF, the FTFH -4% and the FTFH -8% were determined (Fig. 4a). It can be seen that the RCF made from regenerated cellulose caused the most excellent tensile strength, which was suggested by the highest tensile stress (34 MPa). As the content of carbon fibers increases, the tensile stress of the FTFH decreases. The reason for the decrease in the tensile stress of the FTFH is that the carbon fibers with less active groups are difficult to intertwine with regenerated cellulose fibers. The XRD patterns from experiments for the sake of comparison between the original paper and the RCF (Fig. 4b). The XRD patterns of the original paper exhibit obvious diffraction peaks at 14.86° and 22.75°, which correspond to the crystalline form of cellulose-I. The RCF shows the diffraction peaks of cellulose II as indicated by the appearance of a board peak at $2\theta = 20.2°$. During regeneration, the dissolving of cellulose fibers in a solvent, which is the main polymorphic modification of cellulose, causes a rearrangement of the crystal packing of chains from native cellulose I to cellulose II.

The performance of the power density of FTFH was compared with CFP that composed of long cellulose fibers and carbon fibers with the same length (Fig. 5b) [33]. And the resistance of FTFH and the CFP with the content of carbon fibers ranging from 4% to 12% were shown (Fig. 5c). As the content of carbon fibers increases, the power density of FTFH increases and the resistance of the FTFH decrease. It can be observed that the FTFT exhibited a higher power density compared to the CFP. When the content of carbon fibers is 12%, the power density can reach to 2577 w/m^2, which is higher than the CFP. The improved power density of the FTFH could be mainly attributed to the compact contact between carbon fibers and good adhesion of the carbon fibers to the substrate. The generated temperature of the FTFH with the carbon fibers content

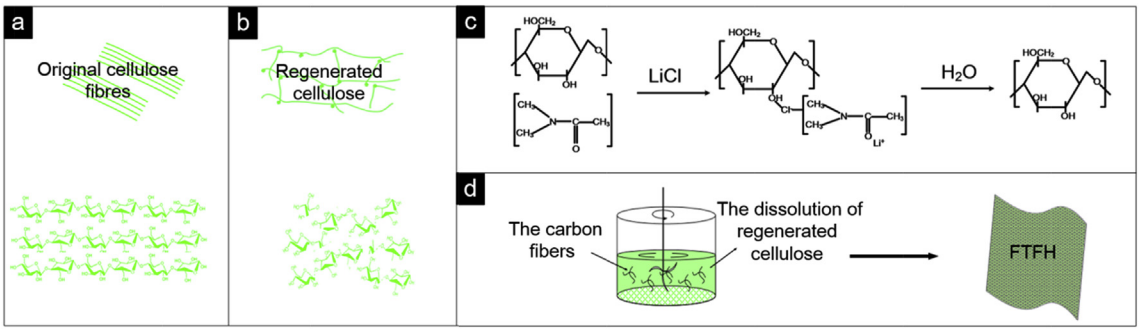

Fig. 1. Schematic illustration of the procedure of FTFH. (a) The structure of original cellulose fibers. (b) The structure of regenerated cellulose. (c) The dissolution of regenerated cellulose and the carbon fibers. (d) The FTFH.

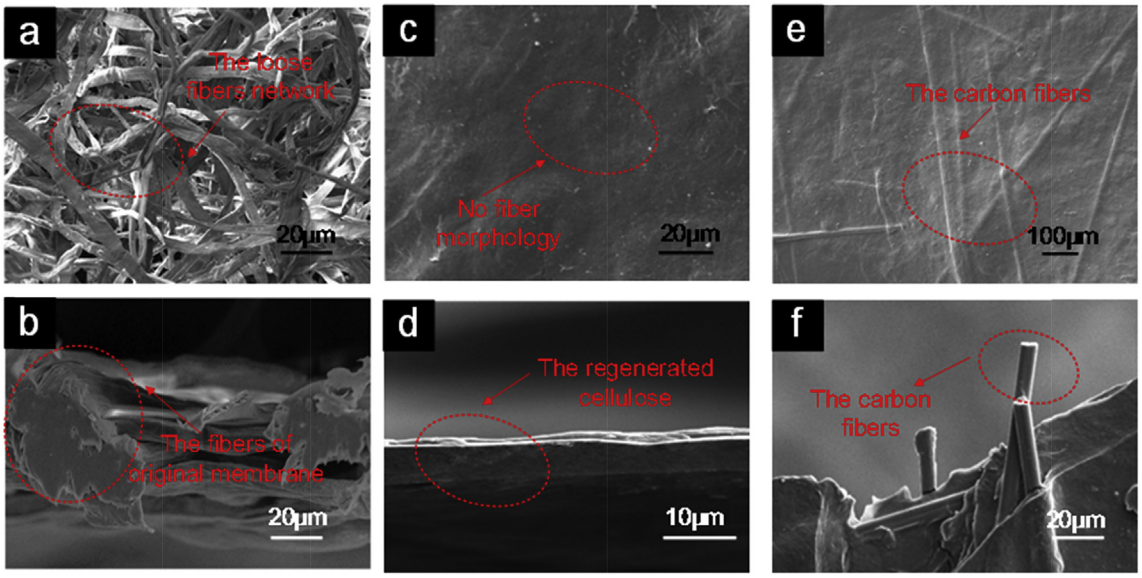

Fig. 2. SEM images of the film samples. SEM micrographs of cross-sections: (a) Original paper. (c) RCF. (e) FTFH; SEM micrographs of the fracture surface. (b) Original paper. (d) RCF. (f) FTFH.

Table 1
Fiber dimension of regenerated cellulose and the original pulp fibers.

	Average length(mm)	Average width (μm)	Average curl Index (%)	Average kink Index (1/m)	Fine content (%)
Regenerated cellulose	0.84	12.09	24.56	1063.58	32.33
Original pulp fibers	2.40	25.80	18.51	1334.82	8.31

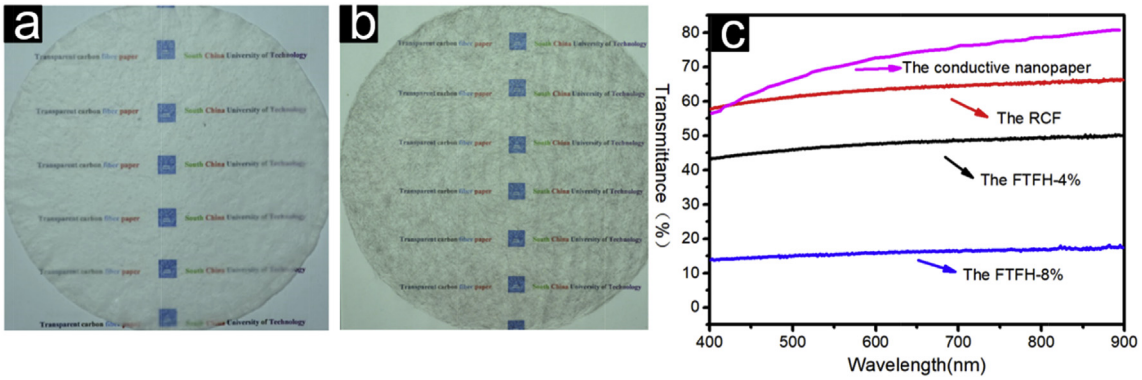

Fig. 3. The digital images of the RCF, the FTFH-4% and the optical transmittance plot for the conductive nanopaper, RCF, the FTFH-4% and the FTFH -8%.

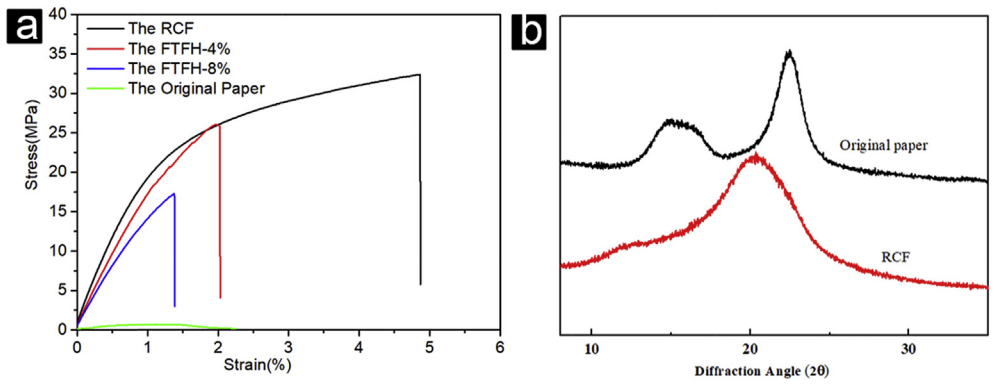

Fig. 4. (a)Stress-strain curves of the Original paper, the RCF, the FTFH -4% and the FTFH -8%. (b)X-ray diffraction profiles of original paper and the RCF.

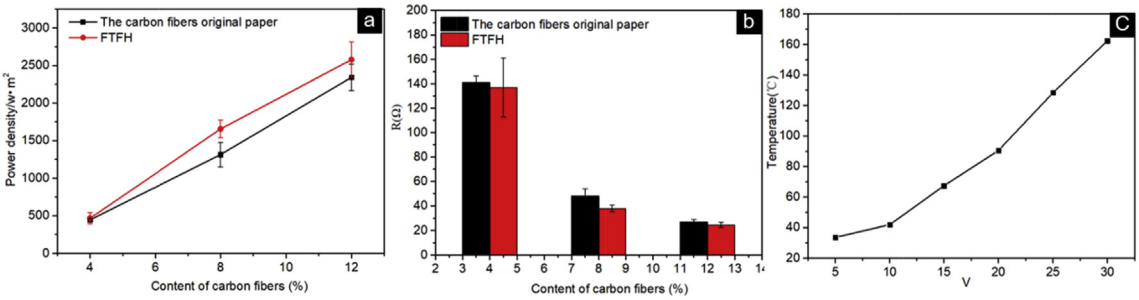

Fig. 5. (a) A digital image of FTFH which illustrates its excellent optical and conductive properties. (b) The power density of FTFH and the CFP; (c) the resistance of FTFH and the CFP with the content of carbon fibers ranging from 4% to 12%; (d) the generated temperature of the FTFH with the carbon fibers content of 12% at various voltages ranging from 5 V to 30 V.

of 12% at various voltages were shown (Fig. 5d). When the voltages were set as 5, 10, 15, 20, 25 and 30 V, the FTFH can reach to the temperature of 33.5, 41.9, 67.5, 90.6, 128.3 and 162.3 °C, respectively. Therefore, the same FTFH could be used to meet different temperature requirements by simply applying an appropriate voltage. Contrary to the long cellulose fibers, the network of carbon fibers can be formed easily due to the regenerated cellulose with small size in FTFH. These results indicate that the use of regenerated cellulose as membrane materials has enormous potential in the fabrication of FTFH.

The temperature evolution of the FTFH and the CFP with carbon content fibers of 4%, 8% and 12% were shown in Fig. 6, respectively. All samples were driven at 20 V for 12 s. One of the intriguing properties of the thin flexible film heater is their high heat generation efficiency. By passing the current through the FTFH, they could be quickly heated from room temperature to a steady temperature of 36.3 °C, 59.9 °C and 88.2 °C, respectively. The presence of high generated temperature will be desirable for self-heating heater devices. As the content of carbon fibers increases, the generated temperature of the FTFH increases. The results indicated that the generated temperature of the FTFH is tuned depending on the content of the carbon fibers. Compared with the CFP with the same length and content of carbon fibers, the generated temperature of FTFH is significantly higher than it. Its good heating performance was attributed to its low resistance of the FTFH, which was improved by introducing regenerated cellulose with small size enabling enhanced overlapping between the carbon fibers. Therefore, we propose that the FTFH with industrially scalable could be fabricated, revealing a bright application prospect in a large-areas heating production, such as heating in outdoors displays, defogging in vehicles, or thermal therapies.

The response time is also an important part to evaluate the performance of the thin flexible film heater. Both the FTFH and the CFP can reach to saturation temperature in 12 s. The results proved that the carbon fibers are good self-heating material to use in the thin flexible film heater. The stability of the resistance of the thin flexible film heater is essential for the sake of safety. To examine the stability of the resistance of FTFH for safety, the change of the resistance of the FTFH along the time was determined (Fig. 7b). The resistance is no obvious change along the time. Only when the first thirty minutes the resistance slightly increased due to the rise of the temperature of the FTFH. Then the resistance maintains a steady value in about two hours. The properties of the FTFH meet the requirement of the use of the thin flexible film heater due to shorter response time, the stability of the resistance and higher generated temperature.

4. Conclusions

The FTFH with high optimal transmittance, excellent tensile strength, and higher generated temperature has been successfully fabricated using novel carbon fibers as self-heating material and regenerated cellulose as membrane materials. The FTFH based on carbon fibers-regenerated cellulose composite generated hot temperature saturation up to 162 °C at 30 V, exhibits shorter response time and long-term stability of resistance. The performance attributes of FTFH are all higher than the previously reported CFP. These results suggest that the use of regenerated cellulose as membrane materials has enormous potential in the fabrication of FTFH. For device applications, the FTFH should be a suitable material as outdoor display panels, defrosters in vehicles, and other devices experiencing large temperature variations. This work is expected to be helpful for the development of the flexible and transparent thin film heater for the fabrication of large-scale, uniform film heaters.

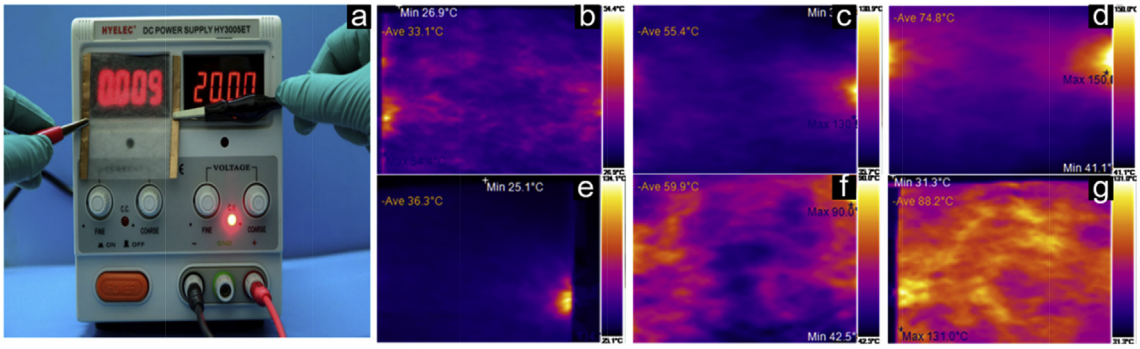

Fig. 6. (a) Digital image of the FTFH with carbon fibers content 2%; The infrared thermal images of the film samples, the CFP with carbon fibers content of (b) 4%, (c) 8% and (d) 12%, the FTFH with carbon fibers content of (e) 4%, (f) 8% and (g) 12%.

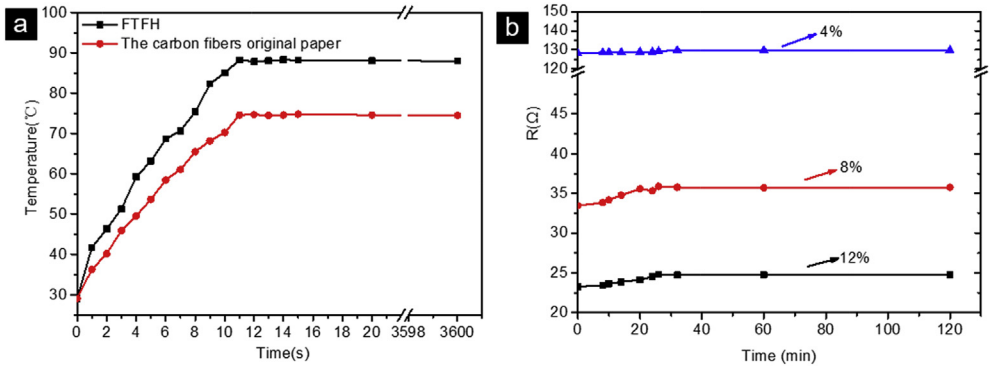

Fig. 7. (a) The generated temperature - response time of the FTFH and the CFP with the content of 12% at 20 V; (b) the resistance - time of the FTFH with various carbon fibers content.

Acknowledgments

This work was supported by funding received from State Key Laboratory of Pulp and Paper Engineering (Grant No. 2016C08), Guangzhou Science and Technology Plan Project (Grant No.201704030066), kindly supported by Guangdong Province Youth Science and Technology Innovation Talents (Grant No. 2014TQ01C781) and the Fundamental Research Funds for the Central Universities (Grant No.2017ZD087).

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