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Original Article

Transparent conductive far-infrared radiative film based on cotton pulp (CP) with carbon fiber (CF) in agriculture greenhouse

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ABSTRACT

There are many types of transparent conductive films, the most common made by depositing indium-tin-oxide (ITO) on ultra-thin glass substrate by physical or chemical methods. ITO films are good conductor of electricity, achieving low resistivity of 10^{-4} Ω cm which is similar to metal. This however results in high power dissipation rate of 1500–6000W/m^2 when connected to 220–240V, which results in high surface temperatures of 300–1000 °C. In this study, a transparent conductive film is developed specifically for agriculture heating, application of mercerization of CP-CF film (mixed conductive carbon fiber (CF) with cotton pump (CP) substrate), and depositing acrylics. For suitability in agricultural heating, the resistivity is designed to be around 40–50 Ω mm, which is much higher than typical ITO films. The newly developed CP-CF transparent film has an average Edge-to-Edge Resistance of 545.65Ω, light transmittance of 67.9% and has a heating capability of 88.70 W/m^2 via far-infrared light. The film has improved light transmittance and is suitable for deployment as part of the retaining structure of agricultural greenhouses as it allows adequate sunlight penetration for the necessary photosynthesis of crops. The analysis evidence showed promising result and can solve the long-term problems of agriculture in seasonable regions such as northern China during the winter. The developed CP-CF film conveys improved energy efficient replacement to conventional greenhouse control to meet the optimal light transmittance and required temperature needs for ~~of~~ crop growth.

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1. Introduction

Membrane materials can be distinguished by their electrical conductivity and categorized into conductive and non-conductive materials. In respect of optical properties, membrane materials can be further categorized as either transparent or opaque. In the current market, transparent conductive films are typically composed of glass or polyester plastic (PET) as the transparent film material, and metal oxide as the transparent conductive material. The transparent conductive films are generally used in optoelectronic applications. This paper focuses on the study of transparent conductive film made of cotton pulp and carbon fiber for far-infrared radiation heating in agriculture greenhouse applications.

Cotton pulp is pure cellulose and is widely used in paper-making industries to produce fine and soft paper [1]. Carbon fiber is an inorganic polymer fiber with a carbon content of more than 90% whereas graphite fiber has a carbon content higher than 99% [2].

At present, Indium Tin Oxide (ITO) thin film is most widely used in practice, and its preparation and film forming process is relatively mature. However, Indium Oxide (In_2O_3) which is the main component in ITO films is very costly. In_2O_3 is also highly toxic and can be harmful to human health if handled poorly during the preparation stages [3].

Fig. 1 – Enclosure structure agricultural greenhouse with flexible transparent conductive film of the study.

The transparent conductive film presented in this study applies CP as the substrate material and CF as the conductive material. The fabrication process can be summarized in a few steps as follows. First both CP and short cut CF materials are mixed for 2.5 h in order to produce uniformly blend-in CP-CF composition and wet copied to form a layer of raw the conductive film. Mercerization is then performed by soaking the film in Sodium Hydrate (NaOH) to uniformly space out clotted fiber within the conductive film. Finally, the film is soaked in acrylics acid resin (AAR) which transforms the film into a flexible and transparent film.

The developed CP-CF film is capable of radiating infrared when supplied with electrical energy. One suitable application of the developed film is in horticulture/greenhouse applications where the infrared radiation can be applied as a source of heating to enable plant growth under cold weather conditions. Currently in the world, modern horticulture facilities take up 600,000 square kilometers of land and are mainly distributed in Asia, whereas glass greenhouses take up about 40,000 square kilometers of land and are mainly distributed in Europe and the United States [4]. In particular, modern greenhouses are built with a wide range of automated systems for artificially controlling of ambient temperature, lighting, humidity, water, air composition and fertilizer to form a conducive environment that promotes the growth of crops. In developed countries such as the United States and Israel the greenhouse industry is aggressively developed [5].

In this study, the developed CP-CF transparent conductive film is flexible and yet sufficiently durable to be used as the retaining structure of agricultural greenhouses. The transparent property of the film allows natural sunlight to pass through for crop photosynthesis during the day while allowing for far-infrared radiation heating during the night to promote crop growth. Sample of the developed flexible transparent film is shown in Fig. 1.

The low cost production of CP and CF, along with a simplified manufacturing process, with far-infrared radiation element for heating purposes, the developed CP and CF film is suitable for mass production and application in agriculture greenhouses.

Edge-to-Edge Resistance, Film Resistivity, and the Light Transmittance will be used to measure the performance of the CP-CF film developed in this study. These performance indicators will be evaluated in relation to the CF contents, and the overall Basis-weight of the film.

Table 1 – The performance of the conducting film based on CP with CF.

Film No.	Thickness (TH, mm)	Basis-weigh (BW, g/m^2)	Carbon fiber content (% by weight)	Edge-to-Edge Resistance (EER, Ω)	Material Resistivity (ρ, Ω•mm)*	light transmittance (%) VL	IR	Full spectral transmittance VL + IR
Film-01	0.075	30	1.00	715.00	53.625	35.4	37.1	72.5
Film-02	0.087	35	1.50	503.95	43.843	30.1	40.60	70.7
Film-03	0.097	40	2.00	418.00	40.546	29.7	30.8	60.5

Note: Size in 200*200 mm, testing thickness by EVERTE® Digital display thickness gauge, Testing Basis-weight by RZ-A-1000 Electronic Balance, testing Edge-to Edge Resistance by TA8301 Digital Multi-meter, testing transmittance by LH-206 Optical Transmittance Meter. Edge-to Edge Resistance (EER) is a conductive film be installed copper carrying on both side, and then measured the between side resistance. ρ is Material Resistivity – commonly used unit Ω·m, in this experiment used unit ρ•mm,ρ (Ω•mm) = R x d = EER (Ω) x TH (mm). VL is Full spectral transmittance. IR is Full spectral transmittance. Average Edge-to-Edge Resistance is 545.65, Full spectral Light Transmittance is 67.9%.

2. Results and discussion

Table 1 shows the properties of the three samples of carbon fiber fabricated using CP as substrate. The samples have fixed size of 200x200mm.

Figure 2 (a) and (b) shows the effect of Basis-weight on Edge-to-Edge Resistance and Film Resistivity. It can be observed that as basis-weight increases, the edge-to-edge resistance and the film resistivity decreases. This finding is in line with the previous results showing the effects of CF content, as basis-weight is directly affected by CF content. As

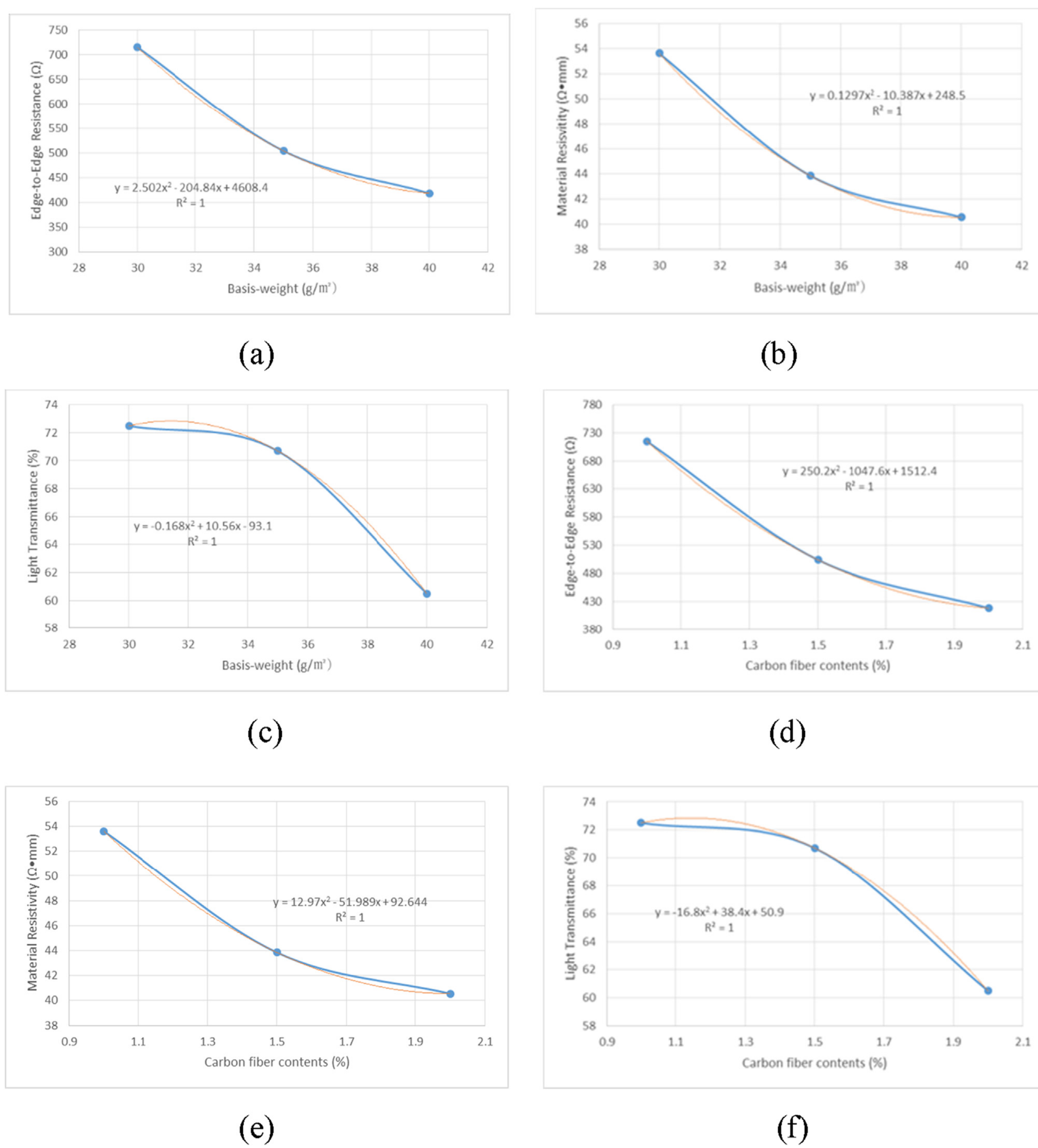


Fig. 2 — The relationship of Edge-to-Edge Resistance, Material Resistivity, Light Transmittance vs Basis-weight, Carbon fiber contents (a) The relationship of Edge-to-Edge Resistance vs Basis-weight with Equation, (b) The relationship of Material Resistivity vs Basis-weight with Equation, (c) The relationship of Light Transmittance vs Basis-weight with Equation, (d) The relationship of Edge-to-Edge Resistance vs Carbon fiber contents with Equation, (e) The relationship of Material Resistivity vs Carbon fiber contents with Equation, (f) The relationship of Light Transmittance vs Carbon fiber contents with Equation.

Table 2 – Summary for the Equation and calculate value.

The relation	Equation	Equation No.	Calculate value[1]	Calculate value[2]
EER vs BW	$EER = 2.502*BW^2 - 204.84*BW + 4608.4$ $R^2 = 1$	01	454.5580	715.0000
MR vs BW	$MR = 0.129*BW^2 - 10.38*BW + 248.5$ $R^2 = 1$	02	41.0410	53.2000
LT vs BW	$LT = -0.168*BW^2 + 10.56*BW - 93.1$ $R^2 = 1$	03	67.6280	72.5000
EER vs CFC	$EER = 250.2*CFC^2 - 1047.6*CFC + 1512.4$ $R^2 = 1$	04	437.3680	503.9500
MR vs CFC	$MR = 12.97*CFC^2 - 51.989*CFC + 92.644$ $R^2 = 1$	05	41.0866	43.8430
LT vs CFC	$LT = -16.8*CFC^2 + 38.4*CFC + 50.9$ $R^2 = 1$	06	65.5880	70.7000

Note: EER: Edge-to-Edge Resistance (Ω). MR: Material Resistivity (Ω•mm). LT: Light Transmittance (%). CFC: Carbon fiber contents (%). BW: Basis-weight (g/m^2). R^2: The value can reflect the degree of fitting between the estimated value of the trend line and the corresponding actual data. The higher the fitting degree, the higher the reliability of the trend line. The formula was obtained by quadratic regression.

CF increases, the basis-weight increases as well. Hence the Edge-to-Edge Resistance and Film Resistivity can be directly measured via the film's basis-weight without knowing the actual CF content. Similarly, Fig. 2 (c) shows the effect of basis-weight on Light Transmittance. As basis-weight increases, due to an increase in CF content, light transmittance drops.

The effect of CF contents on Edge-to-Edge Resistance and Film Resistivity is presented in Fig. 2 (d) and (e). The results show that as carbon fiber contents increases, the Edge-to-Edge Resistance and Material Resistivity will decrease, which indicates an increase in electrical conductivity. Figure 2 (f) shows the effect of Carbon fiber contents on the Light Transmittance of the film. As observed, as the content of carbon fiber particles increases, they will block light and decrease the light transmittance of the film.

Table 2 presents the derived equation representing the relationships between Edge-to-Edge Resistance, Film Resistivity and Light Transmittance with respect to Carbon Fiber content and Film Basis-Weight.

The derived equations in Table 2 allows the design of a transparent conductive film by setting the Basis-weight and CF contents to achieve the desired Edge-to-Edge Resistance, Light Transmittance, and Material Resistivity of the film. An example would be to set the Basis-weight as 37 g/m^2 and the CF contents as 1.8%. This results in edge-to-edge resistance of 454.55 to 437.36 Ω, light transmittance of 65.58%–67.63% and material resistivity of 41.04–41.08 Ω ·mm. Another example would be to set the Basis-weight as 30 g/m^2 and the CF contents as 1.5%, which results in edge-to-edge resistance of 715.00 to 503.95 Ω, light transmittance of 70.70%–72.50% and material resistivity of 53.20 to 43.84 Ω ·mm.

Table 3 shows the measured light transmitted through 40 points equidistant uniform distribution on the industrialize productions. The light transmittance value (in %) was calculated based on the LED light source of 6300 LUX. sample size is 1000 mm x 1500 mm (Fig. 3). The average light transmittance is 4280.70 LUX, and the average is 67.925%, the Edge-to-Edge Resistance is 500 Ω, within the limits of calculated value (see Fig. 4).

The average light transmittance is 4280.70 LUX, and the average is 67.925%, The average light transmittance is 4280.70 LUX, and the average is 67.925%, the Edge-to-Edge Resistance

Table 3 – Transmissivity and distribution of transparent conductive film for Industrial productions.

Measurement point No.	Pervious to light the amount (LUX)	Light transmissivity
01	4290.00	68.09
02	4188.00	66.47
03	4736.00	69.46
04	4380.00	69.52
05	4306.00	67.03
06	4213.00	66.87
07	4260.00	67.62
08	4280.00	67.94
09	4125.00	65.48
10	4256.00	67.56
11	4230.00	67.15
12	4285.00	68.02
13	4301.00	68.27
14	4260.00	67.62
15	4173.00	66.23
16	4263.00	67.67
17	4173.00	66.23
18	4623.00	67.67
19	4368.00	69.33
20	4239.00	69.08
21	4239.00	67.29
22	4199.00	67.29
23	4245.00	67.38
24	4290.00	68.09
25	4315.00	68.49
26	4322.00	68.60
27	4300.00	68.25
28	4322.00	67.17
29	4337.00	68.85
30	4227.00	67.89
31	4263.00	67,67
32	4325.00	68.65
33	4350.00	69.05
34	4290.00	68.10
35	4320.00	68.57
36	4276.00	67.87
37	4262.00	67.65
38	4355.00	69.13
39	4265.00	67.70
40	4262.00	67.65
Mean	4280.00	67.925

a) The average value of Edge-to-Edge Resistance is 500 Ω

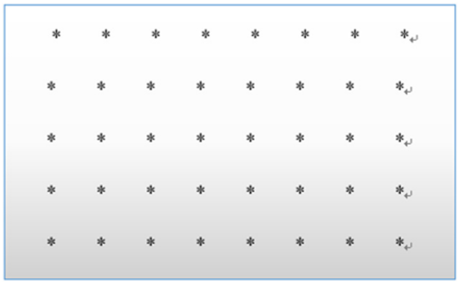


Fig. 3 – The distribute picture on transmittance point.

is 500 Ω, within the limits of calculated value (LT = 72.5–70.70%, EER = 715.00–503.95 Ω).

Table 4 shows the efficiency of the film in maintaining temperature in a miniature greenhouse located at Weifang city, Shandong Province. China, during a week test in the month of January. The Self-built experimental agricultural greenhouse is constructed in size of length 9000 mm, 4000 mm wide, and 6000 mm in height, installed with film coverage area of 25 m^2, it is rated power of the film are 5000W (The edge to edge resistance is 484 Ω/m^2, on supplied voltage 220V, the power dissipated is 100W/m^2), and heating 8 h in winter night (see Table 5).

According to Li et al. [30], Shandong Province is the main vegetable producing area in eastern China, the vegetable-plant area has reached about 130,000 acres in Shouguang, and the number of the agricultural greenhouse has reached more than 400,000. However, agricultural production is limited during the cold and hot seasons in Shandong Province, eastern China, where has ambient temperatures less than −10 °C during the cold season and in hot season approximately 40 °C [31]. Extreme weather conditions have significantly affected agricultural productivity in Shandong Province.

The climate characteristics of the region were summarised for many years in the previous study of Dong et al. [31] They reported that the annual average ambient temperature is

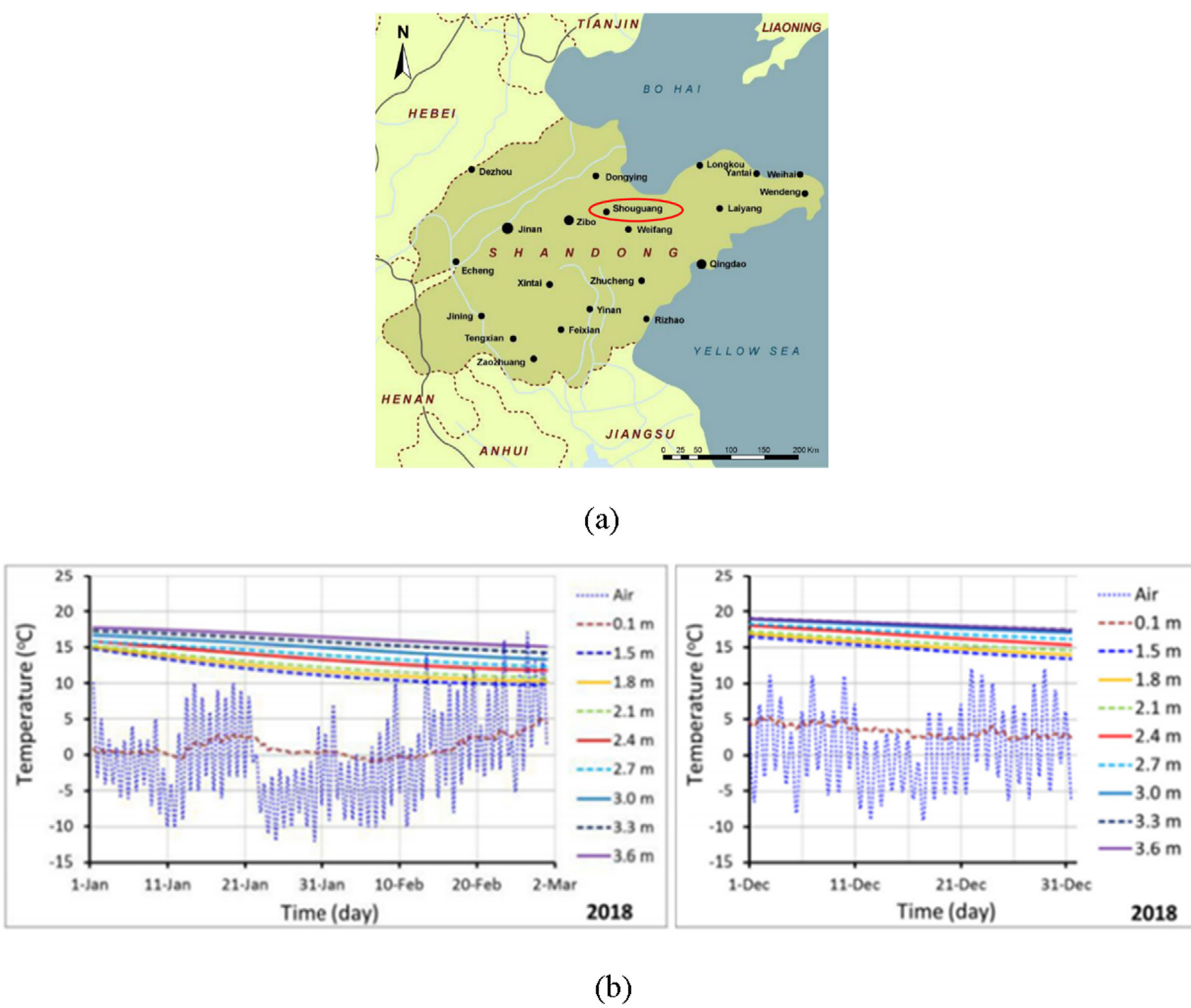


Fig. 4 – The farm area of Shouguang city, Shandong province, China and Temperature variation during the winter. (a) Location map of Shandong Province in China (the geographic coordinates of 114.48° 48′ E–122°42′ E longitude and 34°23′ N–38° 24′ N latitude). Source: https://www.chinadiscovery.com. (b) Obtain from: Measurement investigation on the feasibility of shallow geothermal energy for heating and cooling applied in agricultural greenhouses of Shouguang City: Ground temperature profiles and geothermal potential.

Table 4 – Average winter temperature in Weifang, Shandong province, China.

Month	Daily average maximum temperature (°C)	Daily average minimum temperature (°C)
Jan	4.00	−6.00
Feb	6.00	−3.00
Mar	15.00	3.00

Note: According to "Weifang City Heating management Measures" provisions: indoor heating temperature is 18 ± 2 °C, not lower than 16 °C.The heating period is from November 15 to March 15 of the following year.

Table 5 – The data on self-built greenhouse.

Size (m)			Area of volume		Installed Power (W)	Power density	
Length	Wide	Height	Area (m^2)	Volume (m^3)		Square (W/m^2)	Volume (W/m^3)
9.00	4.00	6.00	36.00	216.00	5000.00	138.88	23.15

12.9 °C. The total annual rainfall is 594 mm (which is mainly concentrated in June to August), the annual average sunshine duration is 2444.4 h, and the primary soil type is silty loamy alluvial soil (fluvo-aquic soils).

During the cold season, when the outdoor air temperature drops below 0 °C, the indoor air temperature drops below 10 °C at night time and early morning hours. The daily variation of the ambient air temperature in January and December ranged from −5 °C to −10 °C during the nighttime and from 5 °C to 10 °C or sub-zero temperatures during the daytime. In February, the daily air temperature variation was below 0 °C at the beginning of the month and above 5 °C at the end of the month.

Table 7 shows the measurement of Edge-to-Edge Resistance (EER) and Light Transmittance (LT) during a week of testing in actual greenhouse operation (see Table 6). The result aims to test the consistency of the EER and LT under different temperature, and relative humidity. The results shows that the properties are stable.

The ET temperature is the winter temperature of the actual measurement of the agricultural greenhouse, which is also the normal working environment temperature of the CP-CF membrane. In other words, using the CP-CF transparent conductive membrane to increase the temperature of the agricultural greenhouse, the temperature is raised to the normal growth requirement of the crops (8–18 °C).

The industrial transparent conductive film be used agricultural greenhouse, according to meet the crop growth that the greenhouse needs rated power 23.15 W/m^3, and power 138.88 W/m^2.

3. Methodology section

In this experiment CP was used as the substrate material and short cut CF as conductive material to mix them into paper pulp with pulping in 2.5 h by beating-machine, and was wet copied to form into the conductive film by paper-machine, then the conductive film is treated with NaOH using mercerizing process, and then the conductive film was soaked into the agent by Dipping-machine, so as to obtain the flexible transparent conductive film.

And the film is coated with electrical carrier by copper strips at both end of film in parallel, when supply with the copper strips on power, the CF in the film will radiate far-infrared that will heat up the agricultural greenhouse.

Mercerization process produces a random internal force that spreads out the clotted fibers, causing the fiber to rearrange uniformly space out to allow conducting power and dissipating far infrared radiation evenly.

The arrangement of fiber in the conductive membrane is shown in Fig. 5, which can be seen in the diagram, the fiber

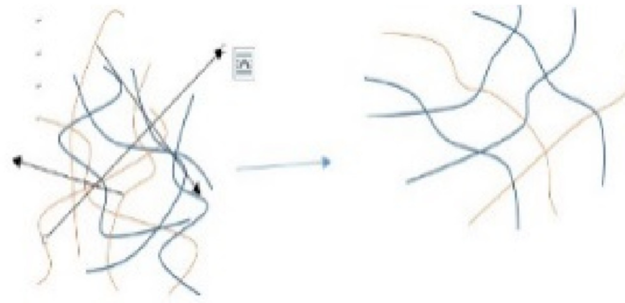
Table 6 – Agricultural greenhouse average temperature in self-built greenhouse.

Date	Outside temperature (°C)	Inside temperature (°C)	
		Am 10:00	Pm 4:00
10/Jan/2020	6 °C/-4 °C	8 °C	8 °C
11/Jan/2020	3 °C/-5 °C	7 °C	8 °C
12/Jan/2020	4 °C/-6 °C	10 °C	9 °C
13/Jan/2020	4 °C/-6 °C	13 °C	12 °C
14/Jan/2020	2 °C/-6 °C	15 °C	12 °C
15/Jan/2020	3 °C/-4 °C	15 °C	13 °C
16/Jan/2020	3 °C/-5 °C	18 °C	10 °C
17/Jan/2020	4 °C/-5 °C	18 °C	12 °C
18/Jan/2020	6 °C/-3 °C	20 °C	15 °C
19/Jan/2020	5 °C/-4 °C	20 °C	15 °C
20/Jan/2020	5 °C/-4 °C	20 °C	15 °C

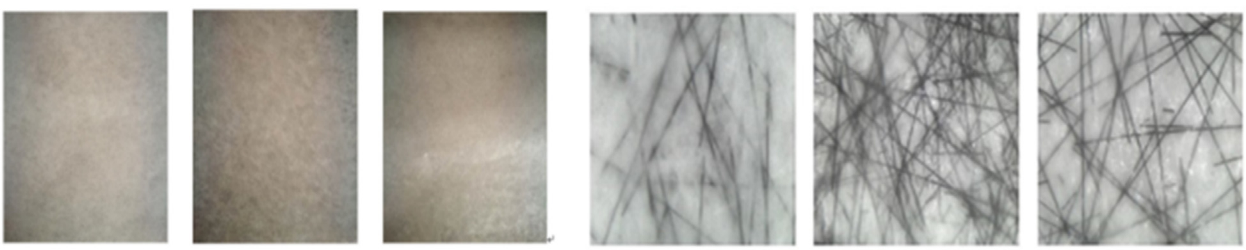
Table 7 – Stability of transparent conductive films in persistent light and high humidity environments.

Day	ET (°C)	RH (%)	EER (Ω)	LT (%)	Sun Light
MON	−4.5	90.00	161.00	68.15	Fine
TUE	−3.0	86.00	161.00	68.15	Fine
WED	−5.0	78.00	161.00	68.15	Fine
THU	−2.0	83.00	161.00	68.15	Fine
FRI	−3.5	95.00	161.00	68.15	Fine
SAT	−6.0	93.00	161.00	68.15	Fine
SUN	−9.0	88.00	161.00	68.15	Fine

Note: EER denotes edge-to-edge resistance, LT denotes light transmittance, ET denotes environment temperature, RH denotes relative humidity. The data is collected between January 11, 2021 to January 17, 2021. Prior to the test the EC is measured as 161.00Ω and LT is measured as 68.15%.



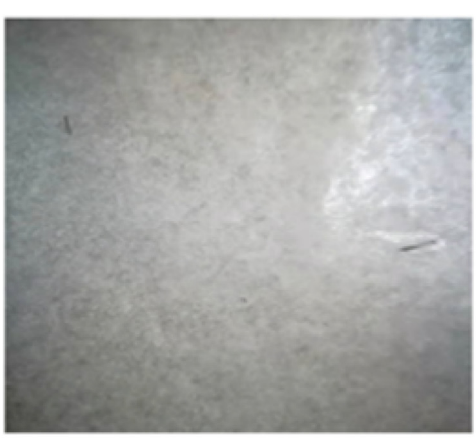
(a)



(b) (c) (d) (f) (g) (h)

Fig. 5 – The changing on film be treated by NaHo. (a) Be internal promotion mechanism was formed on conducting film, (b) Be an untreated conductive film. (c) Be a conductive film impregnated with a transparent agent of paper without mercerization. (e) Be a conductive film impregnated with a transparent agent of paper with mercerization. (f) Be a mercerized conductive film impregnated with transparent agent of paper. (g) Be an untreated conductive film without mercerization (Magnified 1000 times) (h) Be a conductive film impregnated with a transparent agent of paper with mercerization ** (Magnified 1000 times).

(a)



(b)

(c)

Fig. 6 – The transparent paper process steps and the non treated or treated by acrylic resin. (a) Transparent paper process step, (b) Not treated by acrylic resin, (c) treated by acrylic resin.

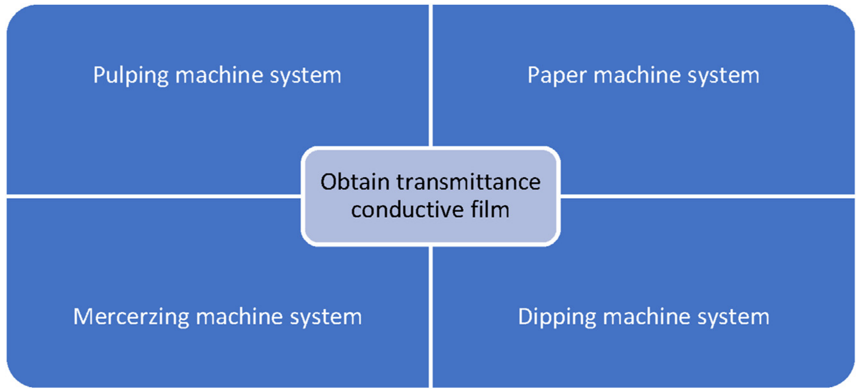


Fig. 7 – Four systems of industrial fabricating transmittance conductive film for CP-CF.

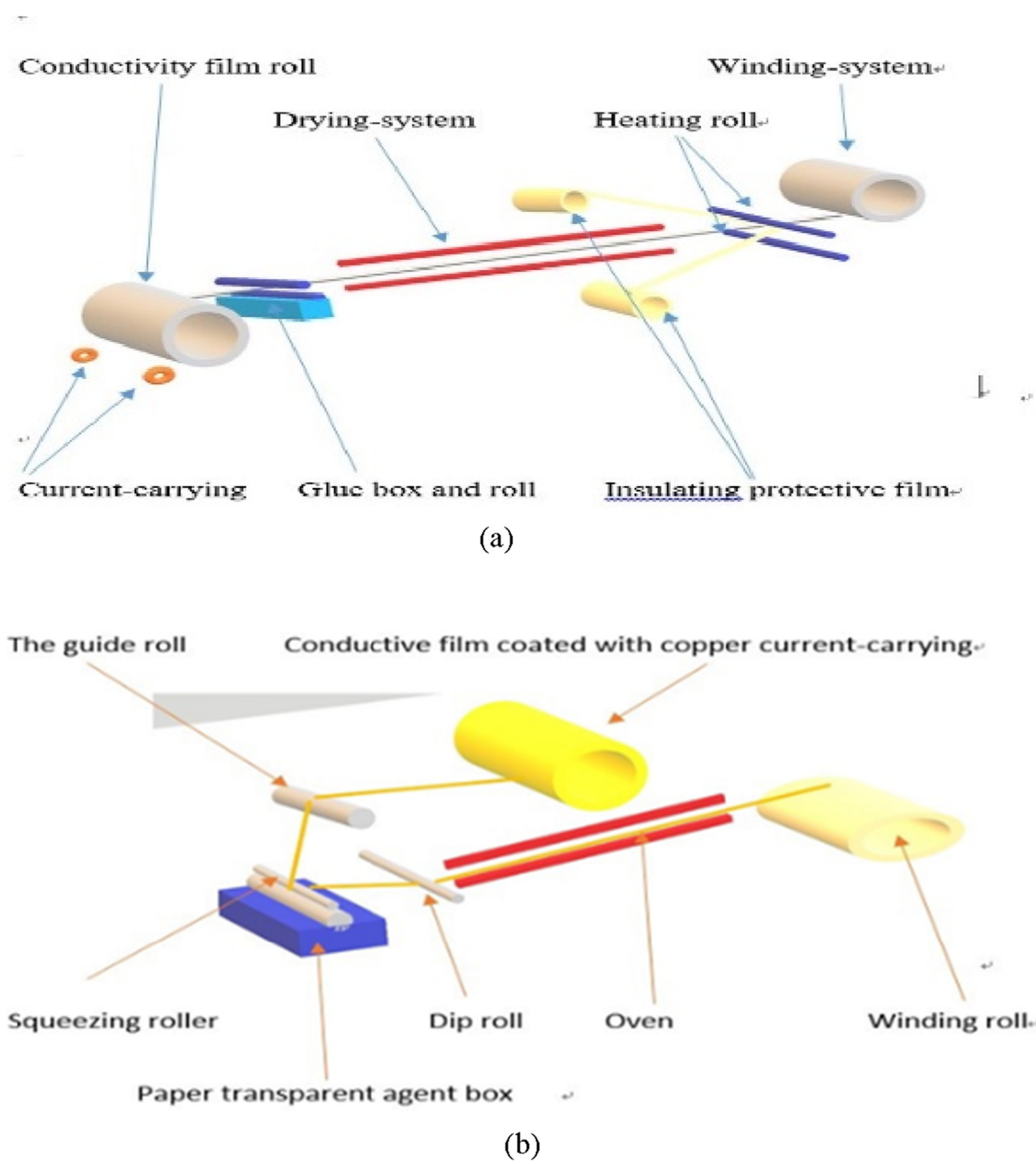


Fig. 8 – The transparent conductive process (a) CP-CF transparent conductive film process flow chart (b) Diagram of self-made transparent agent impregnation machine.

arrangement before and after the mercerization the carbon fiber grid is more uniform. It can be seen from the analysis that caustic soda has swelling effect on cotton fiber, while it has no effect on CF.

Transparent paper is made by conductive paper soaking acrylic resin the method of production of transparent paper can be divided into four types:

Fig. 6 show four steps production of transparent film, and the effect of with and without acrylic coating. The method of soaking with transparent agent (acrylic), the air in the paper is extruded, so that a lot of light transmission, can improve the transparency of the paper. Acrylic transparent agent can effectively remain in the CP and CF film bonds well with fiber and pulp, filling the space between the fibers, hence allowing light transmittance. Acrylic agent is a chemical of optical clarity material with outstanding bonding strength and stiffness.

Fig. 7 shows five steps process of CP-CF transparent conductive film. Industrial process general using Pulping machine system, Paper machine system to fabricate the conductive film, using Mercerizing machine system to well-space fiber, and using Dipping machine system to dip transmittance agent. Fig 8 and Fig 9.

1) Combine CP with chopped CF.
2) CP-CF conductive film by wet copying become in conductive paper or film.
3) Transparent aid for impregnation of CP-CF conductive film.
4) Be installed copper carrying in both side of the conductive film.
5) CP-CF transparent conductive film be installed on both sides face with plastic film, as insulating coating.

1) CP-CF transparent conductive film was prepared by mixing CP and short cut CF by wet form process. The two processes are similar to paper making technology and can be completed by paper making machinery.
2) Transparent aid for impregnation – (confusing; to rephrase) of CP-CF transparent conductive film, be installed copper carrying both two sides on the film, and be installed plastic film in both two side face on the film. The three processes have no similar process equipment and need to be developed and manufactured in the research process. The envisaged process route is shown in Fig. 8 (a)
3) It is an important step in the manufacturing process of flexible transparent conductive film to infiltrate the conductive film of CP matrix with transparent agent. The industrial production process is similar to the process of the dipping machine. However, through the investigation of the existing gumming technology has a common shortcoming, is the amount of gumming is not enough difficult to soak the conductive film of cotton pulp matrix. Therefore, the transmittance of the conductive film cannot meet the requirements. In this study, we can only manufacture our own equipment for the production of cotton pulp matrix flexible transparent conductive film. The envisaged process route is shown in Fig. 8 (b)
4) The manufacturing idea is: CP-CF conductive film be installed metal copper foil strip as electrical current carrier through a pressure roll (Fig. 9), and a rubber roller immersed in the transparent agent rubber tank, and then through a long heating drying channel, and then rewinding to obtain the finished product. The purpose of the metal copper are to conduct electricity for heating.

Fig. 9 – Be installed metal copper foil strip as electrical current carrier.

4. Conclusion

The transparent conductive film is fabricated by applying CF as a conductive material on a CP substrate. In generally, the film can be fabricated using conventional means, which involves the mixing and blending of cotton pulp with carbon fiber solution for 24 h, followed by wet coping to achieve a low basis-weight such that the resulting film is thin and transparent. This method is energy intensive and in order to achieve a very low basis-weight film requires costly machines. The process is feasible in theory, but not practical in actual production. In this study, we have streamlined the fabrication process by applying wet copying, soaking in NaOH for fiber spacing and then soaking in acrylic transparent agent to enable the transparent effect. This method is less complex and requires less than 3 h to complete. The resulting film has low basis-weight, is thin, and has good light transmittance.

In agricultural greenhouses, light transmittance is a critical criterion to evaluate the suitability of any film as a building material. In general, greenhouses constructed with plastic allowed for 50%~60% light transmittance, while glass greenhouse can achieve 60%~70%, and solar greenhouse reaching more than 70%. Crops generally require a temperature range of 32 °C during the day and 12 °C during the night. The newly developed CP-CF film has a light transmittance of 68.15% which comparable to glass, and delivered power dissipation rate that allows temperature to be maintained above 18 °C in subzero weather temperatures, making it a suitable material for agricultural greenhouses.

There are many types of transparent conductive films, the most common being made by depositing indium-tin-oxide (ITO) on ultra-thin glass substrate by physical or chemical methods. ITO films are good conductor of electricity, achieving low resistivity of 10–4 Ω cm which is similar to metal [7, 32], This however results in high power dissipation rate of 1500–6000W/m2 when connected to 220–240V, which results in high surface temperatures of 300–1000 °C. In this study, we have developed a transparent conductive film specifically for agriculture heating. By depositing conductive carbon fiber (CF) on cotton pump (CP) substrate (CP-CF film). For suitability in agricultural heating, the resistivity is designed to be around 40–50 Ω mm, which is much higher than typical ITO films.

Declaration of Competing Interest

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

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