

Article

# Physical and Degradable Properties of Mulching Films Prepared from Natural Fibers and Biodegradable Polymers

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**Abstract:** The use of plastic film in agriculture has the serious drawback of producing vast quantities of waste. In this work, films were prepared from natural fibers and biodegradable polymers as potential substitutes for the conventional non-biodegradable plastic film used as mulching material in agricultural production. The physical properties (e.g., mechanical properties, heat preservation, water permeability, and photopermeability) and degradation characteristics (evaluated by micro-organic culture testing and soil burial testing) of the films were studied in both laboratory and field tests. The experimental results indicated that these fiber/polymer films exhibited favorable physical properties that were sufficient for use in mulching film applications. Moreover, the degradation degree of the three tested films decreased in the following order: fiber/starch (ST) film > fiber/poly(vinyl alcohol) (PVA) film > fiber/polyacrylate (PA) film. The fiber/starch and fiber/PVA films were made from completely biodegradable materials and demonstrated the potential to substitute non-biodegradable films.

**Keywords:** mulching film; biodegradation; natural fiber; biodegradable polymer

## 1. Introduction

The practice of using mulching film to improve the growth and yield of annual and perennial crops has long been recognized [1]. Mulching film preserves heat and moisture, reduces pressure from weeds and pathogens, and conserves water and fertilizer [2–4]. In these ways, mulching film contributes to sustainable agricultural production.

Plastic films (e.g., polyethylene (PE), poly(vinyl chloride), polybutylene, copolymers of ethylene with vinyl acetate) are the most widely used materials because of their excellent mechanical properties and low cost. However, the major problem associated with the use of non-biodegradable plastic films is that they can pollute soil when they are buried in landfills; in addition, the removal and disposal of these plastic residues from the field either before or after harvest is costly and time-consuming [1,5]. For these reasons, farmers usually incorporate used films into the soil; occasionally, they burn the used plastic films, causing harmful pollution [6]. In order to increase the sustainability of agricultural practices and to overcome the disposal problems associated with conventional plastic films, the development and use of mulching films based on biodegradable materials is highly desirable.

Until now, various films capable of being degraded by microorganisms in the soil have been developed, including oxo-degradable plastic film [7], paper film [8], and biodegradable film [9–11]. Oxo-degradable plastic is low-cost and easy to install, but the parts of the film that are buried in the soil do not break down and must be exposed to light and air for degradation [12]. Paper film has been

considered as an alternative to plastic film; however, paper film suffers from very rapid degradation and is easily broken down after exposure to soil, rain, and wind [13]. Furthermore, in order to cause no undesirable effects to the performance of agricultural soil, no harmful substances resulting from the degradation of biodegradable materials used as mulching film should accumulate in the soil [14].

The fibrous materials derived from renewable crops or from their by-products or agro-industrial wastes are good candidates for use in blends and composites [15]. Natural fibers have good mechanical properties and are biodegradable, which makes them highly suitable for use in films in combination with biodegradable polymers. Some natural fibers, such as ramie, flax, hemp, and cotton, are abundant in China and are mainly used for textiles. However, the waste fiber has not been suitably recycled after industrial production. Some biodegradable polymers such as starch, poly(vinyl alcohol) (PVA), poly(butylene adipate-co-terephthalate) (PBAT), poly(butylene succinate-co-adipate) (PBSA), and poly(lactic acid) (PLA) have been used to prepare biodegradable agricultural films [13,14,16–20]. Some films based on natural fibers and polymers were also prepared, such as an orange fiber residue/PVA film [21] and an Osage orange wood/PLA film [10]. These films were prepared using the mixture of grinded fibers and a polymer aqueous solution. The aim of this study was to prepare films based on natural fibers and biodegradable polymers as potential substitutes for plastic films. In this work, three mulching films were severally developed from natural fibers (a blend of waste ramie and cotton fiber) and polymers (PA, ST, and PVA) using the method of air laying in nonwoven fabric. The biodegradability of the films was studied in detail.

## 2. Materials and Methods

### 2.1. Materials and Reagents

Three mulching films (fiber/PVA, fiber/PA, and fiber/ST, originally white) were prepared in our cooperative enterprise (YuanJiang Jingzhu Science and Technology Co. Ltd, Yiyang, China). The fiber/PA, fiber/ST, and fiber/PVA films all had an approximate thickness of 0.35 mm and weight of 40 g/m<sup>2</sup>, which contained approximately 16% polymer (polyacrylate, starch, or poly(vinyl alcohol), respectively) and 84% waste fibers in the textile industry (ramie and cotton fibers with a mass ratio of 4:1). The properties of ramie fiber are as follows: approximately 2–5 cm in length, 30 μm in diameter, a density of 1.49 g/cm<sup>3</sup>, and 6.5% moisture content. The properties of cotton fiber are as follows: approximately 13 mm in length, 20 μm in diameter, a density of 1.58 g/cm<sup>3</sup>, and 7.2% moisture content. All the polymers were of technical grade. Polyacrylate (30% emulsion) was mixed copolymers of methyl acrylate, ethyl acrylate, and butyl acrylate, with an average molecular weight of 1,2000 and purchased from Zhuzhou Chemical Factory, Zhuzhou, China; corn starch was purchased from Suzhou Shuanghuan Chemical Technology Co., Ltd., Suzhou, China, with an average molecular weight of 15,000 and purity of ≥ 98%; PVA (model 100-40H) was purchased from Shanxi Sanwei Group Co., Ltd., Linfen, China, with an average molecular weight of 14,000 and alcoholysis degree ≥ 99%. In addition, a commonly used PE film (white and black) was purchased from the market with a thickness of 0.04 mm.

### 2.2. Physical Properties of Mulching Films

#### 2.2.1. Mechanical Properties

The tensile strength and percentage elongation of each film were measured using a Tinius Olsen testing machine (Model H5KL, Tinius Olsen Testing Machine Company, Horsham, PA, USA) according to ASTM D4964-64 (Test method for tension and elongation of elastic fabrics). The test was carried out before the trial in the field, and each sample was determined in triplicate.

#### 2.2.2. Heat Preservation

Heat preservation was investigated by performing a field test that involved the cultivation of Chinese cabbage in the garden of Wangcheng District, Changsha, China (latitude: 28°18' N, longitude:

112°51' E, altitude: 50 m). The soil temperature was recorded by a soil temperature recorder (model TZS-6W-G, Zhejiang Top Instrument Co., Ltd., Hangzhou, China). The heat preservation behavior of the three fiber/polymer films was similar; therefore, only the results from the fiber/PVA film have been reported. The films were laid on the field, and the temperature at the position of 0 cm, 10 cm, and 20 cm under the soil surface was recorded with no film as a control using the reported method by Moreno [22]. The test was carried out over a period of 39 days from November 8, 2014 to December 17, 2014.

### 2.2.3. Water Permeability

The films' water permeability was assessed using 6-cm-diameter Fisher Payne permeability cups that contained 5 mL of water and were sealed by the films. No film was set as control [21]. The experiment was performed in an air-conditioned room (25 °C temperature and 60% relative humidity (RH)) and variations in the water weight over time were recorded.

### 2.2.4. Photopermeability

The percentage of light transmittance was measured at different wavelengths (450, 650, and 850 nm) using an ultraviolet-visible (UV-Vis) spectrophotometer (UV-2100, Unico, San Diego, CA, USA), with the sample placed in the light path using the reported method by Kijchavengkul [23].

### 2.2.5. Thermal Stability

Thermal gravimetric analysis (TGA) was performed to analyze the thermal stability of the film samples using a TGA/DSC 1 SF/1382 analyzer (METTLER TOLEDO, Shanghai, China). A sample of 10 mg was placed in an aluminum pan and heated from 25 to 600 °C at a heating rate of 10 °C/min under a nitrogen atmosphere.

## 2.3. Biodegradability Tests

### 2.3.1. Micro-Organic Culture Test

Microbial measurements were based on the dilution method of plate counting. Suspensions of soil collected from the garden of Wangcheng District, Changsha, China, which had been covered with the fiber/PVA and PE films for the cultivation of Chinese cabbage, were prepared. The properties for the soil were as follows: The soil pH was 6.05, the soil carbon was 24.52 g/kg, total nitrogen was 1.28 g/kg, total phosphorus was 0.85 g/kg, and total potassium was 17.37 g/kg. The test was carried out over a period of 49 days from November 1, 2014 to December 19, 2014. The fiber/PVA film was used for the micro-organic culture test by laying the film at the bottom of the culture dish. Beef-protein agar medium was used to culture bacteria at 37 °C for 48 h, Martin substratum was used to culture fungi at 28 °C for 72 h, and Gause's No. 1 synthetic medium was used to culture Actinomycetes at 28 °C for 72 h. The measurement of microorganism was performed in triplicate.

### 2.3.2. Soil Burial Test

Soil burial biodegradation tests were performed in a flowerpot with a capacity of approximately 1000 mL. Soil samples were obtained from the test garden in Wangcheng District, Changsha, China. All of the film samples (50 mm × 50 mm) were buried in the pot under 10 cm of soil with 65% moisture. The temperature was 10–25 °C, and the air humidity was 25%–45%. The test was carried out over a period of 45 days in the laboratory from November 4, 2013 to December 18, 2013. At different time points, samples were collected from the pot, gently cleaned by rinsing with water and brushing, dried to a constant weight at 105 °C, and weighed. The percentage weight loss of the samples was calculated according to the following equation:

$$\text{Weight loss} = [(M_0 - M_1)/M_0] \times 100\%, \quad (1)$$

where  $M_0$  is the initial mass of the films before the test, and  $M_1$  is the residual mass of the films after the test.

Fourier transform-infrared spectroscopy (FT-IR) was used to analyze the samples before and after degradation by the standard KBr pellet method. The IR spectra were obtained using a Thermo Nicolet 380 Fourier transform infrared spectrometer (Thermo Nicolet Corporation, Madison, WI, USA) in the range of 4000–600  $\text{cm}^{-1}$ . The films were tested before and after degradation using the transmission method. Scanning electron microscopy (SEM) was performed to observe surface changes on the films before and after degradation. The film samples were coated with gold, and the surface was observed using a Hitachi S-4800 scanning electron microscope (Hitachi, Tokyo, Japan) with a stated resolution of 1.4 nm at 1 kV acceleration voltage.

### 3. Results and Discussion

#### 3.1. Mechanical Properties

The results from the tensile strength test and the percentage elongation of the four tested films are shown in Table 1; these results indicated that the tensile strength and elongation of the fiber/polymer films were poorer than that of commonly used PE film. The fiber/polymer films of this tensile strength were sufficiently strong for use as a mulching film in our field test.

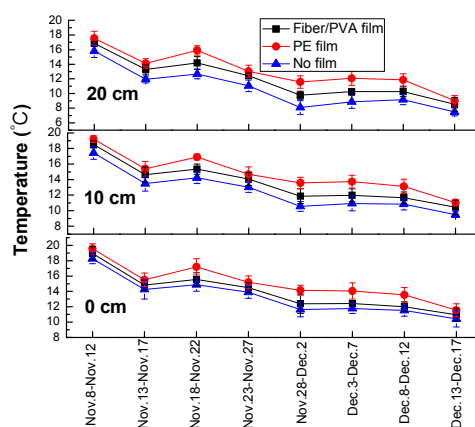
**Table 1.** Tensile strength at break of the different mulching films. ST: Starch; PVA: Poly(vinyl alcohol); PA: Polyacrylate; PE: Polyethylene <sup>a</sup>.

Type of Film	Machine (average, MPa)	Percentage Elongation (%)	Cross-Machine (Average, MPa)	Percentage Elongation (%)
Fiber/ST film	2.92b	24.77b	3.52a	9.06b
Fiber/PVA film	3.58a,b	23.71b	2.62b	10.92b
Fiber/PA film	3.69a	10.92b	3.74a	18.31b
PE film	12.96c	132.3a	12.96c	132.3a

<sup>a</sup> The different lowercases indicate significant difference at  $P < 0.05$  level in the same column.

#### 3.2. Heat Preservation

The fiber/PVA and PE films were used to culture Chinese cabbage; the control used no film. The results in Figure 1 show that the ability of the films to preserve temperature followed the following order: PE film > fiber/PVA film > no film. The ability of the fiber/polymer film to preserve heat was poorer than that of the PE film; however, our field test demonstrated that the fiber/polymer film could be beneficial under certain conditions, such as during the summer season or in high-temperature regions.



**Figure 1.** Temperatures measured under soil (at depths of 0, 10, and 20 cm) covered by different mulching films (fiber/PVA and PE) and no film.

### 3.3. Water Permeability

The water permeability of the three fiber/polymer films was measured under constant conditions of 25 °C and 60% RH. The results shown in Figure 2 reveal that the water permeability of different treatments decreased in the following order: no film > fiber/ST > fiber/PVA > fiber/PA. It can be inferred that this arrangement is in line with the hydroscopicity of the films and, indirectly, is in accordance with the hydrophilic ability of polymers. Both fiber/polymer films allowed water to easily permeate into the soil and maintain the moisture content of the soil, which had a better water holding capacity than PE film.

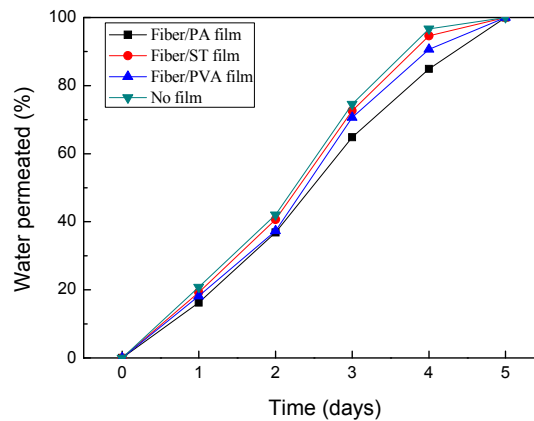


Figure 2. Water permeability of the three fiber/polymer films.

### 3.4. Photopermeability

The determination of photopermeability can provide the data of the extent to which the white fiber/polymer films can prohibit the light. Figure 3 shows that the light transmittance of the three white fiber/polymer films is below 25%, much lower than that of the PE film; the black PE film had alight transmittance below 3%. Therefore, the fiber/polymer films were able to prohibit the growth of weeds by reducing the transmitted light to some extent. Some natural dyes, such as carbon black, tannin, and humic substances, can be blended with the films to produce different colors.

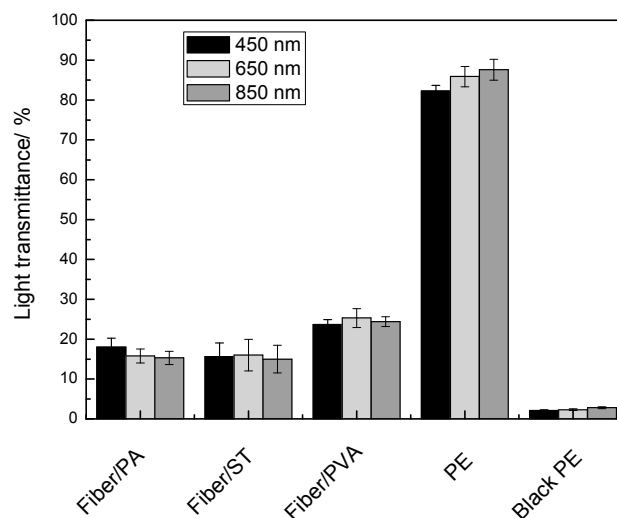


Figure 3. Photopermeability of the four tested films.

### 3.5. Thermal Stability

The TGA curves in Figure 4 show that weight loss began to occur for the three fiber/polymer films at approximately 300 °C, indicating that their thermal stability was poorer than that of the PE film; however, they were sufficiently stable for use as a mulching film at temperatures occurring in the natural environment.

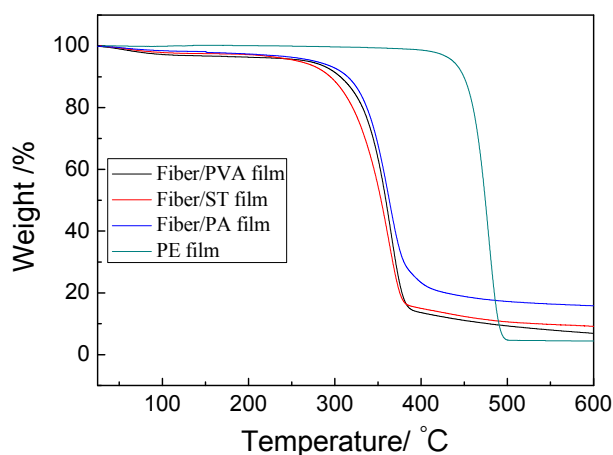


Figure 4. Thermal gravimetric analysis (TGA) curves for the four tested films.

### 3.6. Biodegradability

#### 3.6.1. Micro-Organic Culture Test

The three fiber/polymer films behaved similarly in the micro-organic culture test through our primary experiments; therefore, only the results from the fiber/PVA film had been reported. The soil microbial quantities are presented in Figure 5 and show that the amount of microbes increased over time. The bacteria and actinomycetes were richest using fiber/polymer films, and the fungi was richest using the PE film. The covering of films can produce more microbial, which can be caused by the degradation of the film or heat generated beneath the films; thus, the increase in bacteria and actinomycetes might be beneficial for the degradation of this fiber/polymer film.

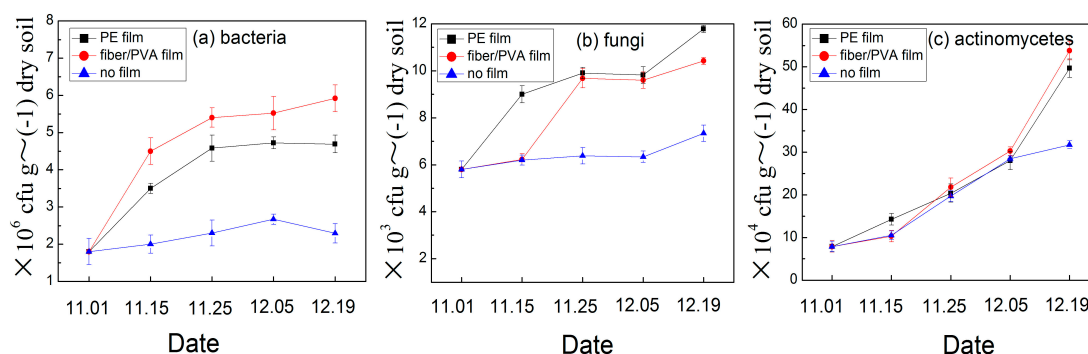


Figure 5. The amount of microorganisms in soil covered by different films between November 1, 2014 and December 19, 2014.

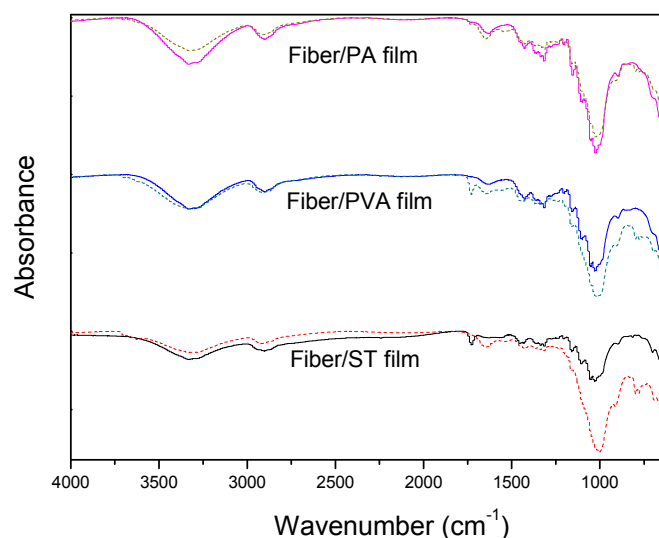
#### 3.6.2. Soil Burial Test

The weight loss of the samples in the soil burial test is shown in Table 2; the results indicated that the degradation degree of the three fiber/polymer films decreased in the following order: fiber/ST film > fiber/PVA film > fiber/PA film.

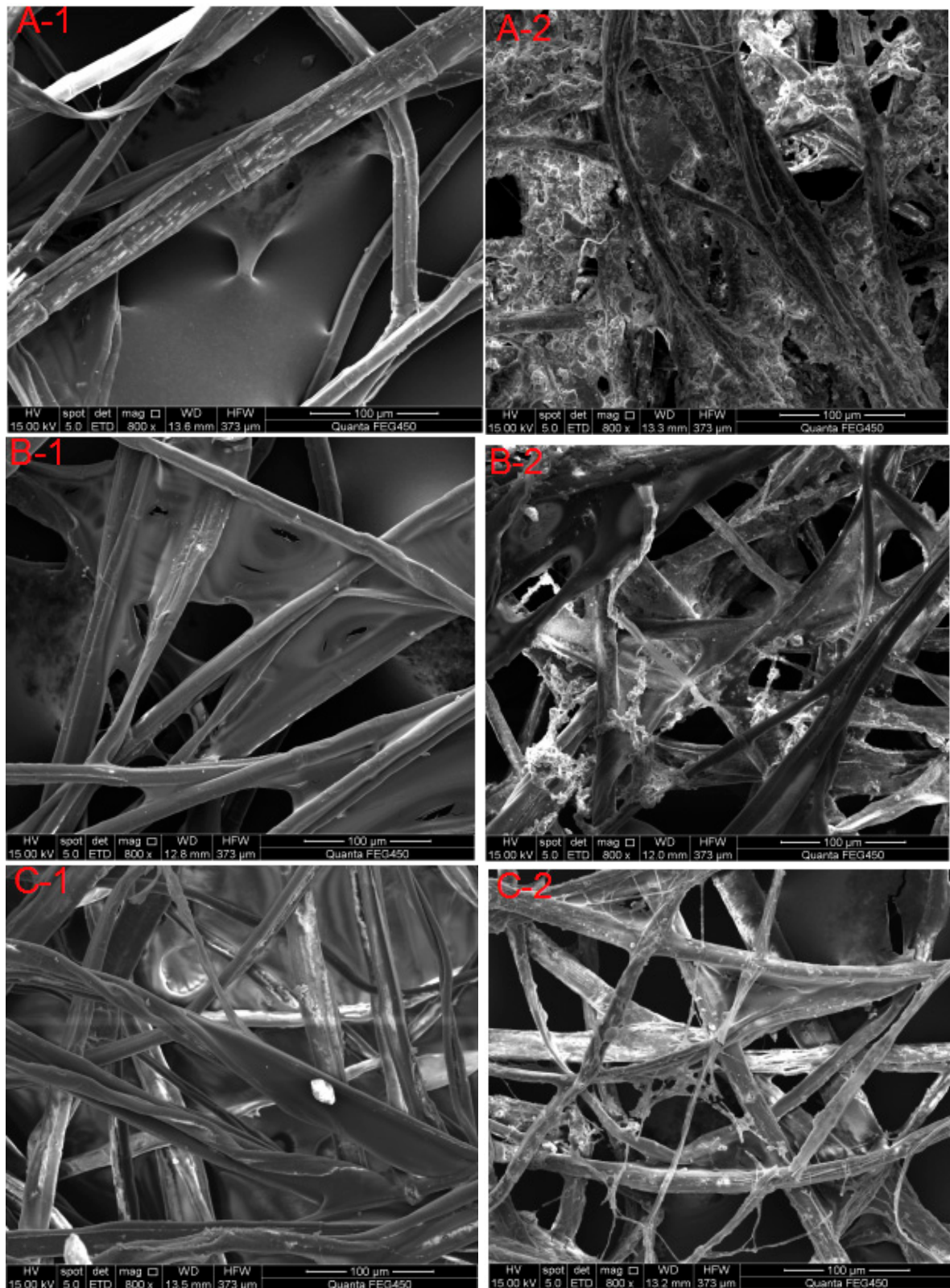
**Table 2.** The weight loss of fiber/polymer films after different degradation time by soil burial test.

Type of Film	Start Time/Initial Weight (g)	End Time/Weight (g)	Weight Loss (%)	Time, Months
Fiber/PVA	Nov. 4/0.2578	Nov. 18/0.2065	19.9%	0.5 month
	Nov. 4/0.2893	Dec. 4/0.1896	34.5%	1 month
	Nov. 4/0.2697	Dec. 18/0.056	79.2%	1.5 month
Fiber/PA	Nov. 4/0.3448	Dec. 4/0.2895	16.0%	1 month
Fiber/ST	Nov. 4/0.3527	Dec. 4/0.1634	53.7%	1 month
PE film	Nov. 4/0.1050	Dec. 4/0.1030	0%	1 month

In the soil burial test, the three films were degraded for four weeks. The samples were characterized by FT-IR before and after degradation to identify the changes in spectra intensities in the films' functional groups [23]. The results are shown in Figure 6. Before degradation, the fiber/PVA film showed characteristic absorption bands at  $3331\text{ cm}^{-1}$  (O–H stretching),  $2902\text{ cm}^{-1}$  (C–H stretching),  $1628\text{ cm}^{-1}$  (C–O stretching), and  $1316\text{ cm}^{-1}$  ( $-\text{CH}_2$  bending). In addition, the absorption bands at  $1159\text{ cm}^{-1}$  (anti-symmetric stretching of the C–O–C bridge) and  $1028\text{ cm}^{-1}$  (skeletal vibrations involving C–O stretching) were characteristic of a saccharide structure. The IR spectra for the fiber/ST film and the fiber/PA film were similar to that of the fiber/PVA film, although the fiber/PA film had a characteristic absorption band at  $1727\text{ cm}^{-1}$  (C=O stretching). Figure 6 shows major differences of absorption intensity in spectra for both the fiber/PVA and fiber/starch films before and after degradation, whereas the fiber/PA film spectra show little variance before and after degradation. The IR spectra indicates that the contents of the samples changed with the absorption intensity, also changing some characteristic peaks. These findings also indicate that the fiber/PVA and fiber/starch films degraded rapidly during this period, while the fiber/PA film degraded more slowly by contrast.

**Figure 6.** Fourier transform-infrared spectroscopy (FT-IR) spectra for the three fiber/polymer films before (solid line) and after (dashed line) degradation.

SEM micrographs of films taken before and after the soil burial test are presented in Figure 7. The samples had many ripples and holes after 1 month of degradation, indicating the partial biodegradation of the polymer and fiber; however, degradation of the fiber requires more time. The SEM micrographs showed that the degradation degree of the fiber/PVA and fiber/ST films was greater than that of the fiber/PA film.



**Figure 7.** Scanning electron microscopy (SEM) images of film samples before and after degradation. (A-1) original fiber/starch film; (A-2) fiber/starch film after 4 weeks of degradation. (B-1) original fiber/PVA film; (B-2) fiber/PVA film after 4 weeks of degradation. (C-1) original fiber/polyacrylate film; (C-2) fiber/polyacrylate film after 4 weeks of degradation.



Both the field and laboratory tests indicate the potential usefulness of the fiber/starch film; however, because of its rapid degradation in soil (approximately 1–2 months in different seasons), it can only be used as a mulching film for crops with short growth periods. The fiber/PVA and fiber/PA films can be completely degraded in 2–3 months and 3–4 months, respectively, during different seasons according to the findings of our field test. PVA and starch were reported as biodegradable polymers; however, it reported that polyacrylate was not a biodegradable polymer [24–27]. Therefore, the fiber/PA film will not be suitable for large-scale applications. On the whole, the fiber/polymer films used in this study have a much shorter degradation time, which can only be used for crops with a short growth periodicity. Therefore, much work should be done by us to prolong the degradation time of these fiber/polymer films in our further studies.

#### 4. Conclusions

The use of biodegradable films in agriculture can promote sustainability, reduce soil contamination, and increase the use of renewable raw materials obtained from agro-industrial waste. The wastes of some fibers such as ramie and cotton can be recycled, and PVA and starch are biodegradable polymers that can be used to prepare mulching film. The tested fiber/polymer films showed good physical properties. The water permeability and heat preservation tests demonstrated that the films had good water permeability and heat preservation capability. The micro-organic culture and soil burial tests demonstrated the good biodegradability of the films. The soil burial test and the FT-IR and SEM analyses indicated that the degradation degree of the three films decreased in the following order: fiber/ST film > fiber/PVA film > fiber/PA film. In the view of the biodegradability, fiber/ST and fiber/PVA films are more suitable than non-degradable films for use as mulching film for crops, but much work should be done to prolong the degradation time of these fiber/polymer films.

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**Author Contributions:** Zhijian Tan and Chaoyun Wang conceived and designed the experiments; Yongjian Yi, Hongying Wang, and Wanlai Zhou performed the experiments; Zhijian Tan and Yuanru Yang analyzed the data; Zhijian Tan wrote and revised the paper.

**Conflicts of Interest:** The authors declare no conflict of interest.

#### Abbreviations

The following abbreviations are used in this manuscript:

PVA	poly(vinyl alcohol)
PA	polyacrylate
ST	starch
PE	polyethylene
PBAT	poly(butylene adipate-co-terephthalate)
PBSA	poly(butylene succinate-co-adipate)
PLA	poly(lactic acid)
RH	relative humidity
TGA	thermal gravimetric analysis
FT-IR	Fourier transform-infrared spectroscopy
SEM	scanning electron microscopy

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