

Article

A Biodegradable Ramie Fiber-Based Nonwoven Film Used for Increasing Oxygen Supply to Cultivated Soil

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Featured Application: Short-term plant cultivation within containers where anoxia often occurs.

Abstract: Plastic agricultural nonwoven films are traditionally used as covering materials, and are prone to cause various ecological problems due to their poor biodegradability. In this paper, a ramie fiber/starch nonwoven film was prepared, and was used as bedding material, that was covered by cultivated soil as opposed to covering it. The biodegradability and porosity characteristics of the film were analyzed, and its effect on oxygen supply to soil was investigated. Results showed that the prepared film had good biodegradability (65.6% after 72 days), and had a loose and porous structure, with the main pore size being in the range of 250–300 μm . After the soil moisture content was reduced to about 44%, the oxygen concentration in the soil that was in close contact with the film, which padded the bottom surface of the plate, rose sharply and then kept stable at 20.1%, whereas soil directly in contact with the plate remained extremely anoxic (0.2%). It was concluded that use of the prepared film increased the oxygen supply to the soil in contact with it, which sufficiently compensated for the oxygen consumption caused by soil microbial activities. Thus, the prepared film is very suitable in short-term plant cultivation within containers where anoxia often occurs.

Keywords: agricultural film; nonwoven; ramie; soil; oxygen

1. Introduction

Since it was first introduced to agriculture in the 1950s, plastic film mulching has become a globally applied agricultural practice because of its instant economic benefits, such as higher yields, earlier harvests, improved fruit quality, and increased water-use efficiency [1–4].

Plastic films are generally compact and low-permeable or impermeable, whereas nonwoven films, which are produced through bonding randomly-oriented micron-sized fibers together physically or chemically, are usually loose and porous. These structural differences give nonwoven films some unique physical properties (e.g., air permeability) that can be advantageous, compared to plastic film when used as covering materials. For example, the temperature changes under nonwoven film are shown to be more stable, therefore avoiding the extremely high temperatures at noon that often occur under plastic film [5–7], and the occurrence of disease is described as being reduced due to the decreased air humidity under nonwoven film [8–10]. Because of these advantages, nonwoven film is increasingly used for crop cultivation as a substitute for common plastic film in modern agriculture [11].

Traditionally, agricultural nonwoven films are mainly made of fossil fuel-based plastics (e.g., polyester, polypropylene, vinylon) due to their good mechanical properties and low cost [8]. However, it is relatively expensive, and thus unattractive, to remove and recycle used films from the field. Consequently, these agrotexiles are often intentionally or unintentionally left on the land, where they accumulate gradually in the soil and persist for years due to poor biodegradability, and ultimately cause various ecological problems [12–15].

A solution to overcome these issues is to employ biodegradable polymers such as polybutylene adipate terephthalate (PBAT), polycaprolactone (PCL), polybutylene succinate (PBS), PBS-co-adipate (PBSA), polylactic acid (PLA), and polyhydroxyalkanoate (PHA) as raw materials. PLA is most often selected as the major feedstock of biodegradable nonwoven films because of its relatively low cost, abundance, high mechanical strength, and frequent use [16]. Many studies have demonstrated that nonwoven films made of PLA perform satisfactorily in the field [6,17–19]. However, considering the cost and richness of the raw materials, biodegradable natural plant fibers such as cotton, flax, hemp, and ramie, which have excellent mechanical properties, could be more suitable for use in agricultural nonwoven films. Some plant fiber-based nonwoven films have been reported on, such as cotton-based [20], flax-based [21], and jute-based [22,23].

A ramie fiber/starch nonwoven film was developed in our previous study. Waste fiber from the ramie spinning industry was used as the main feedstock due to its huge supply in China and its distinctive characteristics (e.g., excellent air permeability and hygroscopicity). A continuous process, mainly consisting of air laying web formation, bonding, and drying, was used to prepare the film. Unlike the traditional agricultural nonwoven films that are usually used as mulching materials to cover soil [24,25], the ramie fiber/starch nonwoven film was used as bedding material that was to be covered by cultivated soil as opposed to covering it. In a typical application of our previous work, the film was applied so that it padded the bottom surface of the plates that were to be used to raise machine-transplanted rice seedlings, and was then covered by seedling soil (Figure 1). Previous studies showed that the growth of rice seedling roots was significantly promoted by the use of this film, as it helped to form a strong and not easily broken seedling block, which meant the efficiency of the machine transplanting was improved—a process that was delayed by the presence of broken seedling blocks where this film was not employed [26–28]. Rice seedlings raised in this way showed many characteristics similar to rice seedlings raised under aerobic cultivation [29,30], suggesting that the function of the ramie fiber/starch nonwoven film was related to oxygen supply in seedling soil. In this paper, the biodegradability and porosity characteristics of the ramie fiber/starch nonwoven film were analyzed in detail, and its effect on oxygen supply to soil was investigated.



Figure 1. (left) The ramie fiber/starch nonwoven film; and (right) Its application. A: Empty plate for raising rice seedlings for machine-transplanting, B: The film is padded on the bottom surface of the plate, C: The film is covered with cultivated soil and rice seeds are sown on it.

2. Materials and Methods

2.1. Materials and Reagents

The ramie fiber/starch nonwoven film was prepared in our cooperative enterprise (Haerbin Jingzhu Agricultural Science and Technology Co. Ltd., Haerbin, China) by a continuous process that included web formation by air-laid web-forming machine, bonding with 3–4% (w/v) modified corn starch aqueous solution, and desiccation in a drying chamber. It had an approximate thickness of 0.25 mm and weight of 40 g/m², and contained approximately 14% modified corn starch and 86% waste fibers from the textile industry (ramie and cotton fibers with a mass ratio of 4:1). The properties of the ramie fiber were as follows: approximately 2–5 cm in length, 30 µm in diameter, 1.49 g/cm³ in density, and 6.5% in moisture content. The properties of the cotton fiber were as follows: approximately 13 mm in length, 20 µm in diameter, 1.58 g/cm³ in density, and 7.2% in moisture content. The modified 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%.

2.2. Evaluation of Biodegradability

The biodegradability of the film was evaluated according to the China standard GB/T 19277.1-2011 [31]. In this evaluation, cellulose powder (particle size <20 µm) was employed as a positive control reference material, the mixture of film fragments (or cellulose powder) and activated vermiculite (inoculated with microbial flora) was placed under a controlled environment (58 ± 2 °C, water content ≈ 50%, sufficient oxygen supply) to undergo a strong aerobic composting, and the degree of biodegradation (Db) was assessed by measuring the amount of carbon dioxide emission during the composting process and calculated according to the following formula: $Db = \frac{AcCO_2}{ThCO_2} \times 100\%$, where AcCO₂ stands for actual release amount of carbon dioxide produced by experimental materials during the composting process and ThCO₂ stands for theoretical release amount of carbon dioxide of experimental materials.

2.3. Analysis Related to Porosity Characteristics

The microstructure of the film was analyzed through scanning electron microscopy (SEM, QUANTA FEG450, FEI, Hillsboro, OR, USA), and SEM images were obtained with an accelerating voltage of 15 kV and a working distance of 13.5 mm (for 200× magnification) and 13.6 mm (for 800× magnification). A capillary flow porometer (3H-2000PB, Beishide Instrument Technology (Beijing) Co., Ltd., Beijing, China) was used to measure pore size distribution of the film with anhydrous ethanol as infiltrating fluid. The air permeability of the film was assessed by an air permeability tester (TQD-G1, Labthink Instruments Co., Ltd., Jinan, China) at the pressure of 10 MPa, and the water vapor permeability of the film was assessed by a water vapor permeability tester (W3/031, Labthink Instruments Co., Ltd., Jinan, China) at the temperature of 38 °C and relative humidity of 90%.

2.4. Actual Detection of the Effect on Oxygen Supply to Soil

A seedling cultivation plate with small holes (φ = 3 mm) at the bottom was used in this detection. The treatment and control were arranged in the same plate to ensure consistency of moisture and other soil conditions during the determination. Half of the plate was covered by the film (treatment) while the other half was not (control), and two fixed needle-type oxygen mini-sensors (OXF500PT, PyroScience GmbH, Aachen, Germany) were placed in the plate, with their ends in close contact with the film and the bottom surface of plate, respectively (Figure 2). The seedling plate was then filled with crushed seedling soil (loam from paddy field) and the soil was watered well. The oxygen concentration was measured and recorded continuously and automatically with an optical oxygen meter (FSO₂-4, PyroScience GmbH, Aachen, Germany) every 4 min. In the process of determination, the soil would be watered well again once it dried out. The above detection was carried out in an artificial climate box (MGC-400H, Bluepard Instruments Co., Ltd., Shanghai, China). The temperature of the climate box

was set at 25 °C and the relative humidity at 60%. A 3-day continuous measurement was conducted, during which the soil was thoroughly watered twice, and some soil samples were taken from the plate to determine moisture content using the drying method.

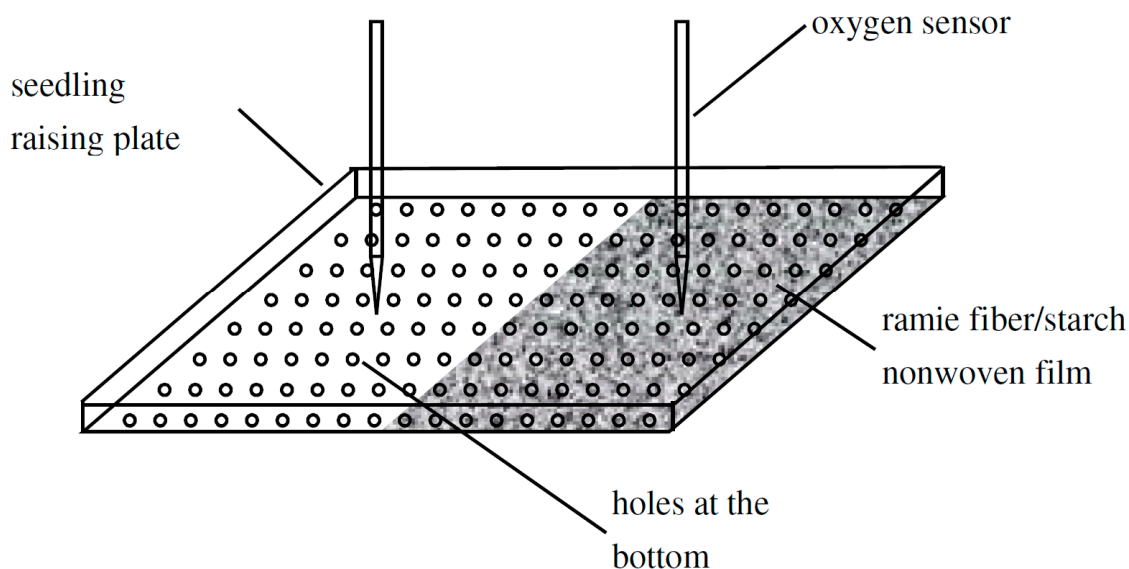


Figure 2. Diagram of the experimental device (not yet covered by seedling soil).

2.5. Simulation Demonstration of the Effect on Oxygen Supply

The presence of oxygen was detected by an oxygen indicator which contained agar (1%, w/v), methylene blue ($C_{16}H_{18}ClN_3S$, 13 mg/L), and sodium dithionite ($Na_2S_2O_4$, 130 mg/L). It is originally colorless, but once it comes into contact with oxygen it turns blue [32]. The film was first deoxidized by soaking it in aqueous solution that contained methylene blue (13 mg/L) and sodium dithionite (260 mg/L). Two kinds of petri dishes (Petri dish A and Petri dish B) were used in the experiment. The difference between them was that the bottom of the Petri dish B had a hole ($\varphi = 3$ mm, sealed beforehand with tape). In an anaerobic operating box, the heated oxygen indicator (in melted state) was first poured into Petri dish A. In the meantime, the deoxidized film was laid on the bottom of Petri dish B. After the oxygen indicator cooled to become an agar block, it was transferred into Petri dish B so that it was in close contact with the film. Petri dish B was then covered with a lid and sealed with a sealing membrane to insulate it from the outside air. During this process, the agar block and film were always kept isolated from oxygen, and therefore colorless. The operation of the control was the same as above except that there was no deoxidized film laid on the bottom of Petri dish B. Finally, all the prepared Petri dish Bs were placed in air and the tape on the holes were torn off, and the color changes of the agar blocks were observed and recorded.

3. Results and Discussion

3.1. Biodegradability

As presented in Figure 3, the biodegradation of the ramie fiber/starch nonwoven film started with a rapid degradation period of 4 days, in which time the diurnal degradation rate (4.08%/day on average) was very close to that of the cellulose (reference material) (4.73%/day on average). This was followed by a much slower, but roughly constant, (0.86%/day on average) degradation period, which continued until day 60 (at day 60 the degradation degree was 64.5%). In a slightly different way, the cellulose degraded at a roughly constant rate (3.29%/day on average) until day 20 (at day 20 the degradation degree was 65.9%). After that, the degradation degree tended to be constant. At the end of the determination period (day 72), the degradation degree of the cellulose reference and the film were 77.1% and 65.6%, respectively. Because the degradation degree of the film in the first

4-days (16.3%) was slightly greater than the mass ratio of corn starch in the film (14%), we inferred that the rapid degradation of the film during the first 4-days was mainly due to the degradation of the modified corn starch, as it degraded more easily than ramie fiber. In other words, when the film is embedded in the soil, the adhesive material will biodegrade rapidly, but the fiber skeleton will remain and work for a relatively longer time.

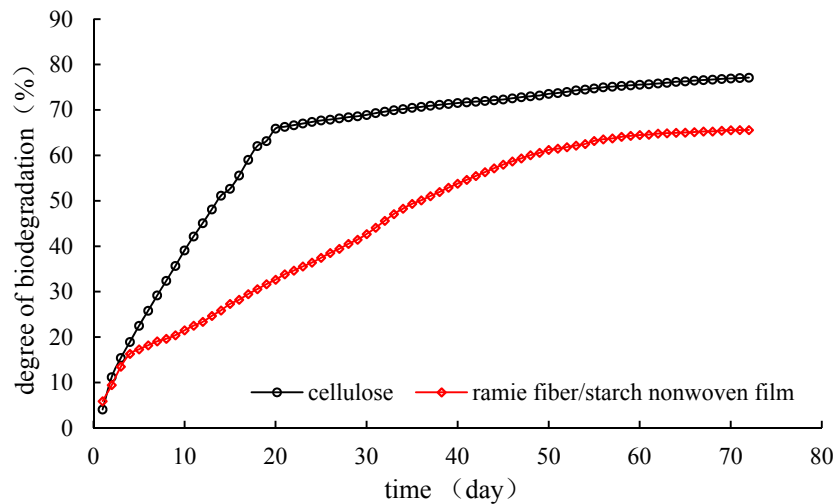


Figure 3. Biodegradation of the ramie fiber/starch nonwoven film.

3.2. Porosity Characteristics

Figure 4a,b show the microstructure of ramie fiber/starch nonwoven film that was investigated by SEM analysis at 200× and 800× magnification, respectively. It can be clearly seen that ramie fibers crisscrossed in the film, and gaps between them were incompletely filled with corn starch, forming a loose and porous structure. As shown in Figure 5a, the film's pore diameter was mainly distributed in the range 250–300 μm, accounting for 46.9% of the total, followed by 0–50 μm, 50–100 μm, and 100–150 μm, accounting for 20.0%, 18.3%, and 13.1%, respectively. The air permeability and water vapor permeability of the film were 2.71 L/(cm²·min) and 107.1 g/(m²·h), respectively (Figure 5b), indicating that the porous structure provided the film with good permeability.

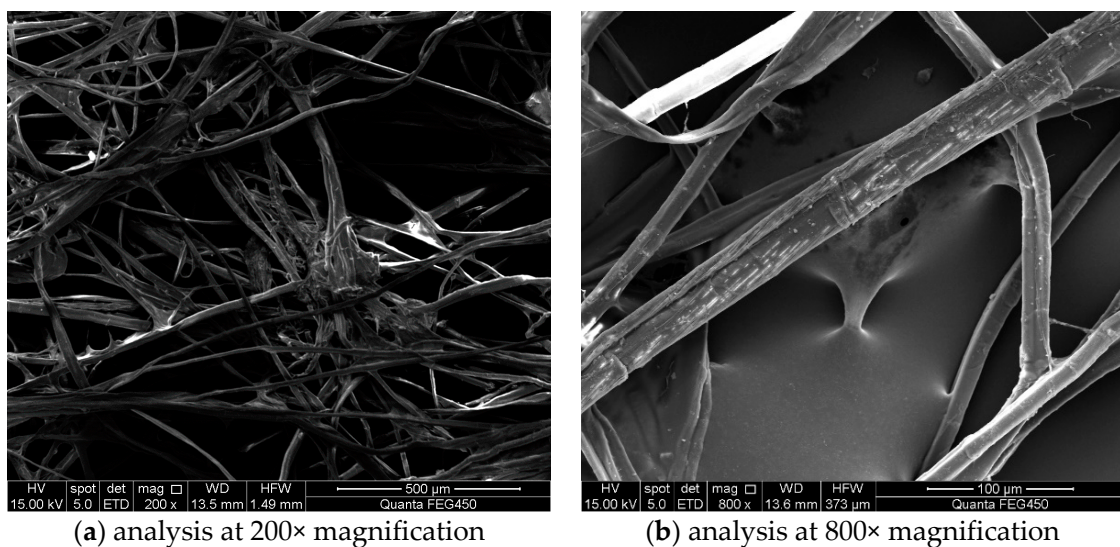


Figure 4. Scanning electron microscopy (SEM) pictures of the ramie fiber/starch nonwoven film.

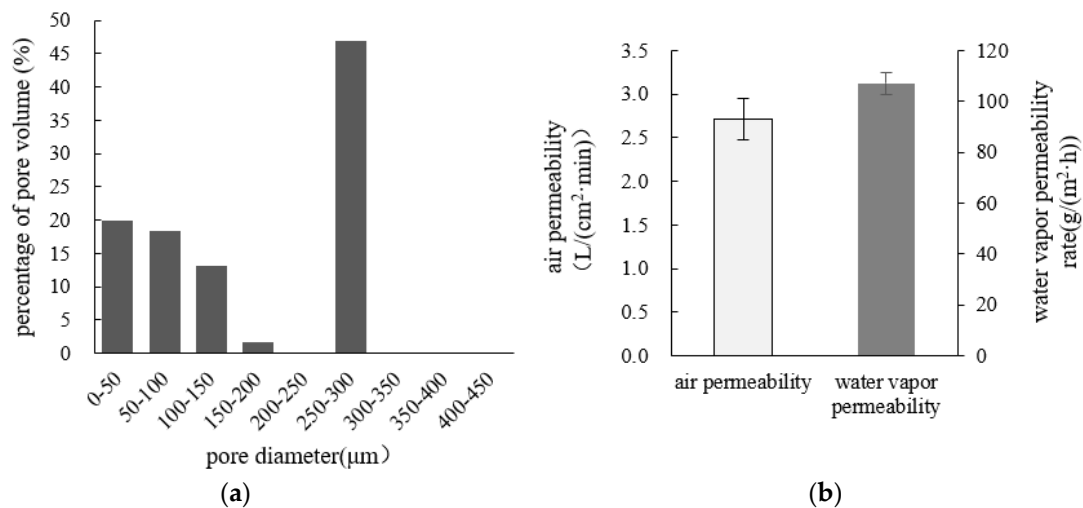


Figure 5. Pore size distribution (a) and permeability (b) of the ramie fiber/starch nonwoven film.

3.3. Effect on Oxygen Supply to Soil

As can be seen in Figure 6, soil moisture content decreased linearly with time. The two roughly parallel fitting lines (dotted straight lines) indicate that it decreased at approximately the same rate for the two water cycles. In water-saturated soil (about 48% moisture content), whether the bottom of the plate was covered by the film or not, soil oxygen concentration decreased rapidly from 20.1% to 0.2–0.5% in about 7 h, and then remained steady. However, when the soil moisture content decreased to about 44% (estimated results based on linear fitting equation) 19 h later, the oxygen concentration in the soil that was in close contact with the film (i.e., oxygen concentration at the film surface), padded on the bottom surface of the plate, increased sharply from 0.5% to 20.1% in less than 1 h, and then remained steady. The same changes in oxygen concentration at the film surface (i.e., rapid decline—steady anoxic state—sharp rise—steady oxygen saturation state) occurred again in the second water cycle. The oxygen concentration in soil that was directly in contact with the plate bottom (i.e., oxygen concentration at the plate bottom surface) that was not covered with the film, however, always remained at 0.2% for both water cycles, regardless of the decline in soil moisture content.

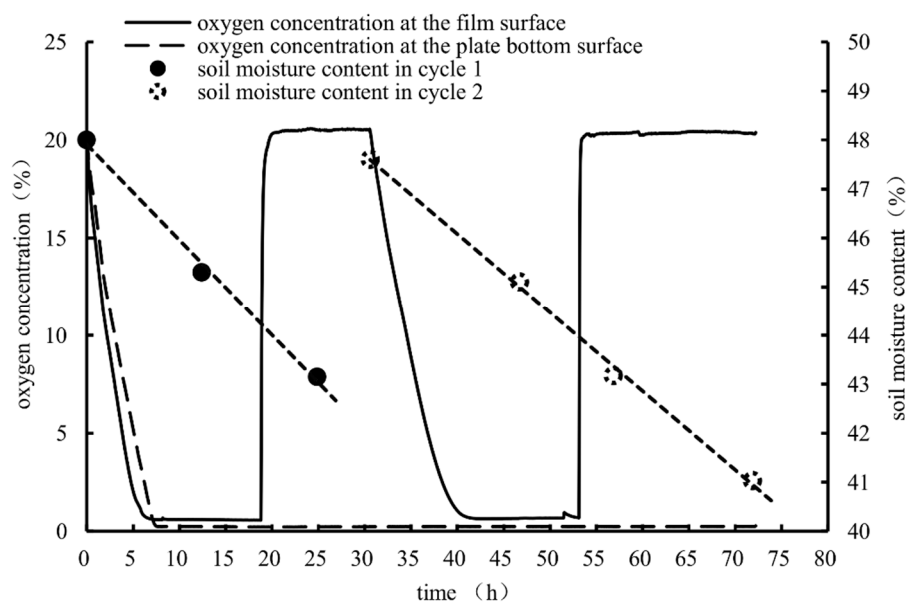


Figure 6. Changes in oxygen concentration and soil moisture content with time.

The rapid decline in oxygen concentration in water-saturated soil in this study fully accorded with the reported fact that water-saturated soil becomes anoxic as soon as there is persistent depletion of oxygen by soil aerobic microorganisms. This is because the diffusion of oxygen into water-saturated soil is extremely slow (the effective diffusion coefficient is on the order of 10^{-9} to 10^{-8} m²/s), meaning that an adequate compensation of oxygen supply cannot be obtained [33]. Considering that the oxygen depletion by soil aerobic microorganisms continued when the soil water content reduced, the persistent high oxygen concentration at the film surface indicates that there was a continuous adequate oxygen supply. When considering the contemporaneous constant anoxia at the bottom surface of the plate, it can be safely concluded that it was the film that increased the supply of oxygen to the bottom soil.

3.4. Process of Oxygen Into the Soil

As shown in Figure 7a,b, when the holes were sealed, whether the bottom of the petri dish was covered by the film or not, the agar blocks within them remained colorless, indicating that no oxygen entered the petri dish. However, after tearing off the tape, color change occurred in the agar block in the petri dish covered by the film. A blue round plaque formed rapidly at the hole in the bottom of the dish, and gradually expanded and deepened in color, indicating that radial oxygen diffusion occurred in the bottom of the agar block. At the same time, no obvious color change occurred in the other agar block, where the petri dish was not covered by the film, except for a small blue plaque at the central hole, indicating oxygen diffusion was dependent on the film.

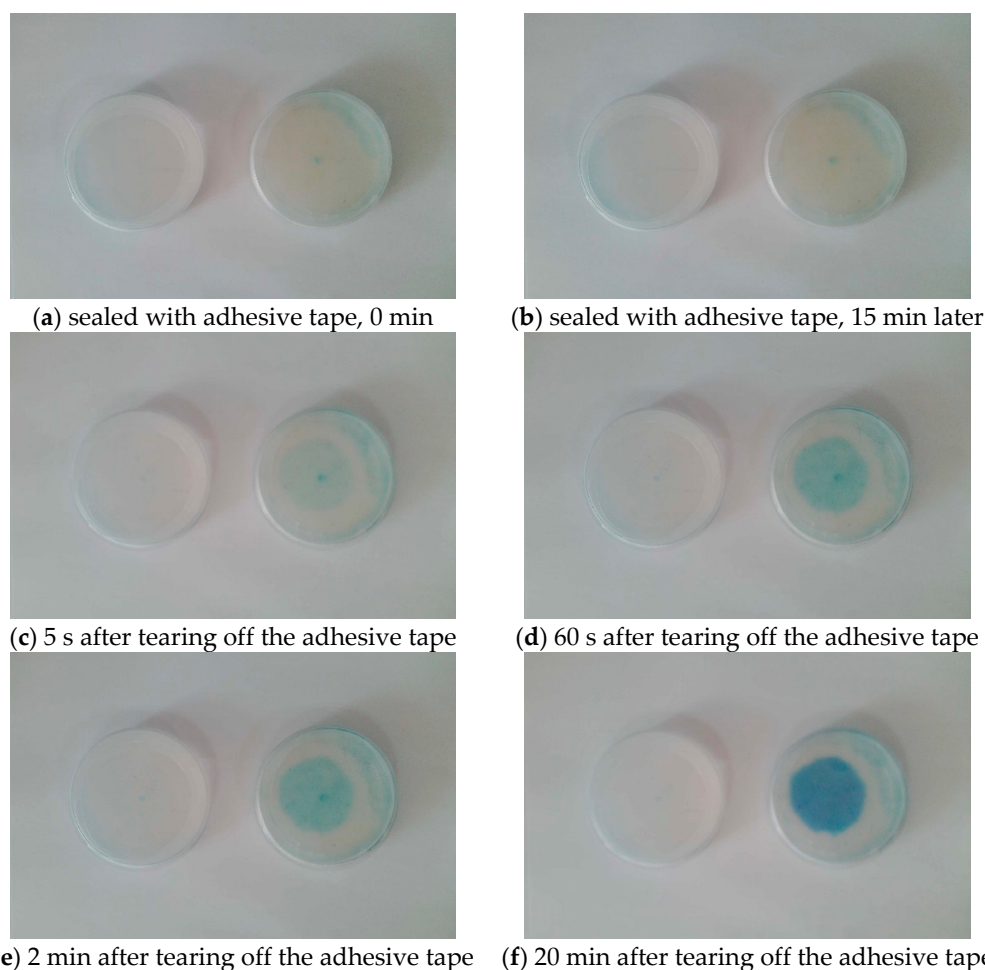


Figure 7. Color change of agar block. The bottom surface of the petri dish on the right was covered by the ramie fiber/starch nonwoven film whereas the left was not, the diameter of the petri dish was 9 cm.

In order to detect the actual effect of the film on the oxygen supply to soil, loam was used to cover the film. It is well known that this kind of soil has an enormous number of capillary pores, with an equivalent pore size range of 2–20 μm , and thus has good water holding capacity [34]. However, the main pore size range of the ramie fiber/starch nonwoven film was 250–300 μm . That was non-capillary porosity, which means that water in the pores of the film were inclined to be absorbed by the soil when in close contact. After the loss of moisture, the film returned to its initial porous state and air diffusion occurred. As the effective diffusion coefficient in the film was of the same in magnitude as that in air (10^{-5} to 10^{-4} m^2/s), the oxygen transfer rate in the porous film was much higher than that in water-saturated soil. As shown in Figure 8, the outside oxygen entered the film from the hole, spread over the film rapidly, then the diffusion of oxygen into the soil occurred across the entire film surface, rather than only at the hole (which occurred in the plate that was not covered by the film). Consequently, more oxygen diffused into the bottom soil and compensated for the oxygen consumption there.

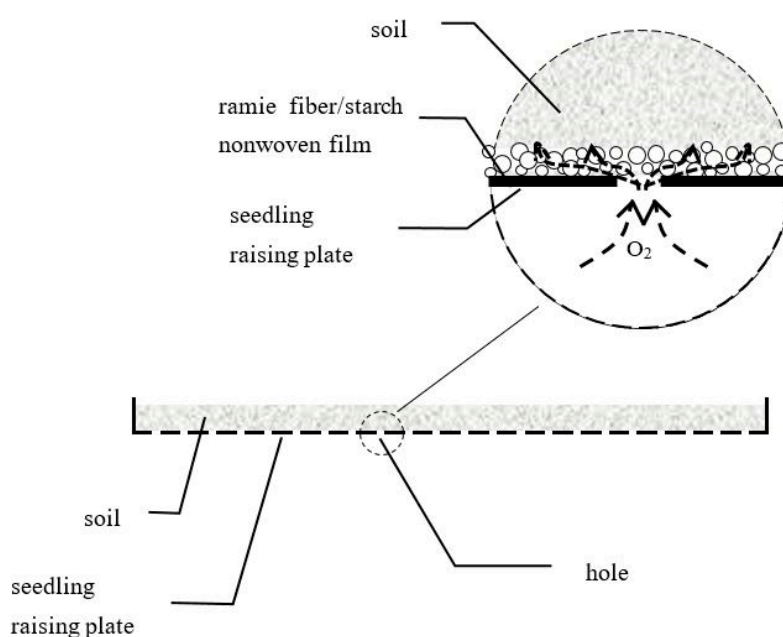


Figure 8. Process of oxygen into soil through diffusion in the ramie fiber/starch nonwoven film.

4. Conclusions

This study confirmed the good biodegradability of the ramie fiber/starch nonwoven film. Thus, it can be safely used for some short-term crop production without worrying about its long-term residual pollution in the soil. Moreover, due to its unique structure (i.e., a large number of non-capillary pores), when covered by soil, the water in the film tends to be absorbed by the soil. This makes the film permeable so that oxygen can spread quickly within the film and diffuse into the soil from the entire film surface, therefore increasing the supply of oxygen to the bottom soil and compensating for the oxygen consumption there. It would also be very beneficial to the growth and development of rice seedlings raised using plates. Rice seedling soil is often too wet to remain permeable in the early stage of cultivating rice seedlings, which means anoxia tends to occur in the soil. Thus, it would be particularly useful to apply the ramie fiber/starch nonwoven film during this stage. Finally, we deduce that the film would also increase the oxygen supply to the soil next to the inner surface of any other cultivation container where anoxia often occurs, if padded with the film, and accordingly benefit the growth and development of any plants in the container, as their roots could get more oxygen.

Author Contributions: W.Z., Y.N. and C.W. conceived and designed the experiments; W.Z., Y.N., Y.Y. (Yuanru Yang), Y.Y. (Yongjian Yi), W.Y. and H.W. performed the experiments; W.Z. and Z.T. analyzed the data; W.Z. wrote and revised the paper.

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

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