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Ultra-fast and low-cost fabrication of transparent paper

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Transparent paper is a kind of promising and environmentally friendly material. In this study, we show that transparent paper can be fabricated in an ultra-fast and low-cost way. This low-cost top-down method only takes three steps of cell separation, lignin removal, and cold pressing to obtain a high-quality transparent paper. The fabrication time is further reduced, and the resulted transparent paper shows high transparency up to 90.3%. The application as a substrate material for transparent and flexible electronic devices is demonstrated by emulating the printed circuit on the prepared transparent paper. This top-down method will greatly promote the market-oriented applications of transparent paper as an environment friendly material.

Keywords: transparent paper; transparent film; biodegradation; plastic replacement; sustainable biomaterial.

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1. Introduction

Plastic is used in a wide range of fields for its transparency and flexibility including portable personal electronics, packaging, biomedical materials, etc. However, due to scattered using sites, poor emission management, high recovery costs, and difficulties in recovery sorting, less than 10% of the world's plastic is recycled and reused. Most of the remaining plastic waste enters the ocean. As a result, 50%–80% of marine debris is plastic, and presently nearly 8 million tons of plastic garbage is dumped into the ocean every year[1–7]. These plastic wastes are difficult to degrade, and many fragment into microplastics with less than 2 mm in size. They finally enter the food chain along with the water cycle, which seriously harms the environment and human survival. Therefore, biodegradable materials are urgently needed to replace plastic. Thus, naturally derived biomaterials, such as silk, cellulose, chitin, and other materials have attracted the attention of researchers[8].

Recently, transparent paper prepared from natural cellulose fibers is considered as a potential substitute for plastic film replacement due to its high transparency, high mechanical strength, flexibility, and biodegradability[9–23]. It is based on the finding that plants are hierarchical fiber materials with unit cellulose fibers of only several nanometers in diameter. The principle of transparency for transparent paper is that the light scattering of nanocellulose fibers is extremely weak. As a new environmentally friendly material, its application areas are widely discovered and studied, such as displays[15], transistors[16,17], batteries[18], sensors[18], solar cells[19,20], and many other functional devices[9,10,19,21].

However, the extremely high preparation cost is the main obstacle to the market application of transparent paper. Although plant raw materials are cheap enough, the extraction of their nanofibers is very time-consuming and costly, including chemical methods[22] (2,2,6,6-tetramethyl-1-piperidinyloxy), mechanical methods[23–25] (homogenization, microfluidic, ultrasonication, extrusion, and steam explosion), biological methods[26] (enzymatic hydrolysis), etc. The methods using nanocellulose fibers to prepare transparent paper are bottom-up methods, which are difficult to achieve mass production. The rinsing, filtration, and dewatering processes for nanofibers in the bottom-up methods are always time-consuming and tedious[21]. For example, using the commonly used TEMPO method, the transparent paper preparation time is at least 48 h[23,27–29]. Recently, we proposed a very efficient top-down method for fabricating transparent paper that does not involve the extraction of cellulose nanofibers. Only two steps are needed, lignin removal and cold pressing[29,30]. Though it is more efficient for this method (about 5 h), the resulted transparent paper is still very expensive because of the use of wood slices as raw materials. Here, we demonstrate the ultra-fast and low-cost transparent paper fabrication in only 42 min and with greatly expanded raw materials. This method uses cheap raw materials and further shortens the preparation time by an order of magnitude, thereby greatly lowering the price of transparent paper and promoting its market applications.

2. Experimental Section

2.1. Materials

Basswood, straw, wheat straw, moso bamboo, laurel, and vines were used in this study just for demonstration. Basswood was purchased from Oriental Aolong Wooden Crafts Co., Ltd., Wuhan, Hubei. Straw and wheat straw are planted in Xuzhou, Jiangsu. Moso bamboo was purchased from Credit Department Store Co., Ltd., Suqian, Jiangsu. The laurel branches and vine were all gained from landscape plant of Nanjing University. Sodium hypochlorite [NaClO, 10% (mass fraction), Shanghai Linfeng Chemical Reagent Co., Ltd.] was used to remove the lignin content. The solvents used were deionized (DI) water and ethanol (98%), which was purchased from Yasheng Chemical Co., Ltd., Wuxi, Jiangsu.

2.2. Transparent paper fabrication

Different plants (wood, bamboo, straw, wheat, laurel, vine, etc.) could be made into transparent paper through the same preparation processes. Here, bamboo is used as an example plant material to demonstrate the typical preparation process. Firstly, bamboo was crushed into chips with a size about 3 mm × 3 mm, and, then, the chips were immersed with 10% NaClO solution at 50°C for 15–20 min (the mass ratio of bamboo to NaClO is 1 : 60). After the chips turned to white, they were washed by the water and ethanol mixture (the mass ratio is 1 : 1) three times to remove excess chemicals. Finally, a transparent paper was prepared by removing excess water through suction filtration and cold pressing. In the process of cold pressing, a polymethyl methacrylate (PMMA) or polycarbonate (PC) board, the sample, a microporous filter membrane, and filter paper are sequentially placed in the press machine, where the applied pressure is about 3 MPa and the holding time is about 10 min.

2.3. Screen printed circuit

Circuits were screen printed on transparent paper using a stainless steel mask with a mesh count of 325 and a wire diameter of 15 μm[31]. In this study, the optimized printing speed is 110 mm · s^{-1}, and the screen-printed silver electrode thickness is 6 μm. After printing, the printed anodes were annealed at 120°C in air for 15 min.

3. Results and Discussion

The commonality of plant microstructures greatly broadens the selection range of raw materials for preparing transparent paper (Fig. 1). Generally, plants are made up of cells, and the cell walls of most plants have similar composition and cellular

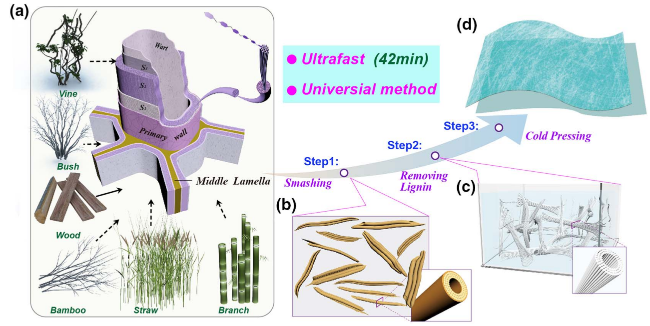

Fig. 1. Schematic of the rapid, universal fabrication process of transparent paper using different plants by lignin removal and cold pressing. (a) Taking the wood cell profile model as an example, plant cell walls have similar microstructures. Plant walls such as vine, bush, wood, bamboo, straw, branch, wheat, and other common plants have similar microstructures. The plant is (b) separated into small pieces and (c) delignified by NaClO subsequently. (d) Transparent paper achieved by cold pressing finally.

hierarchical microstructure[32,33]. Plants are mainly composed of cellulose, hemicellulose, and lignin. As shown in Fig. 1(a), the cell walls of plants, such as vine, bush, wood, bamboo, straw, and branch, have similar hierarchical multifiber structures, and lignin is filled in the spaces between the cellulose fibrils. Taking the wood cell structure as an example [Fig. 1(a)], the cell wall is usually divided into a primary wall (P), a secondary wall (S), and a middle lamella that exists between cells. The cellulose fibrils in the P are arranged like a network. S_1–S_3 constitute over 70%–90% of the wood cell wall, and the cellulose fibrils in S_2 are almost parallel to the cell axis. Therefore, based on the commonality of the microscopic structure of these plants, a universal method can be developed to prepare transparent paper simply and quickly. It only needs three steps. The first step is to isolate the plants into small pieces [Fig. 1(b)], which facilitates the full removal of lignin and shortens the reaction time. Then, the colored lignin is removed, and micron size nanofibril aggregates are obtained [Fig. 1(c)]. Finally, a transparent paper is acquired after cold pressing in the last step [Fig. 1(d)]. This method mainly operates on plant cells with the scale of several hundreds of microns to obtain transparent paper composed of nanofibers, thereby belonging to the top-down method. Also, it throws away the necessary time-consuming nanofibers extraction processes in traditional methods, and hence the production efficiency is greatly increased.

In this method, high-quality transparent paper can be obtained in only 42 min. Taking the wood branch as an example to fabricate a transparent paper, the process can be divided into three steps: first, wood branches [Fig. 2(a)] are mechanically disintegrated into wood flour or wood chips [Fig. 2(b)], which takes about 2 min, and then the colored lignin is removed from the wood powder to obtain white nanofibril aggregates solution [Fig. 2(c)], which takes only 20 min. After the lignin removal, the cellulose fibers can move freely and spread out with little applied force. Finally, the micron nanofibril aggregates are washed in the ordinary way of filtration for about 10 min, and excess water is removed by cold pressing for 10 min to obtain a transparent paper [Fig. 2(d)]. Compared with the lengthy preparation process of nanofibers, nanofibril aggregates shorten the time of processing, cleaning, and separation by orders of magnitude. The scanning electron microscope (SEM) image of the sample corresponding to each step shows that there is no nanofiber operation in the process of changing the wood branch to transparent paper [Figs. 2(e)–2(h)]. The surface of the finally obtained transparent paper is flat and smooth, and the traces of the original micron fibers are no longer visible [Fig. 2(h)]. Moreover, from the SEM image and atomic force microscopy (AFM) photos [Fig. 2(j) and 2(k)], the surface of the transparent paper is made to the nanometer level by cold pressing, and the nanofibers in the transparent paper can be observed directly, which are randomly arranged to show the isotropic nature of the paper. The X-ray scattering image of transparent paper in Fig. S1 of Supplementary Materials is a circle at high-intensity diffraction, which indicates the random distribution of cellulose molecular chains and the isotropy of the transparent paper. Other types of plants, such as bamboo, straw, branch, bush, and vine with similar structure and composition, have also been practically shown to produce high-quality transparent paper composed of cellulose nanofibers quickly and efficiently using this method (Figs. S2–S13 in Supplementary Materials). The X-ray diffraction (XRD) patterns of wood chips and wood transparent paper show that peaks corresponding to cellulose crystals change little [Fig. 2(i) and Fig. S14 in Supplementary Materials]. This indicates that the ordered structure of the crystalline region in transparent paper is not disrupted by the delignification and pressing fabrication processes.

In view of the urgent needs of future applications, a film with high light transmittance under visible light is needed[34]. Transparent paper prepared from various plants has high transparency and high haze in the visible light range. The paper is isotropic, and it exhibits homogenous optical properties. Figure 3(a) shows the transparency and haze of paper prepared from bamboo. In the visible light range of most wavebands, the total transmittance is close to 90%, and the haze is in the range of 45–65. We can clearly see the school badge through the transparent paper. Further, the transparency and haze of the paper are quite uniform. As shown in Fig. 3(b), nine points were selected from the same piece of wood transparent paper for the test of transparency and haze. The values of transmittance are basically maintained at about 90.30%, and the values of haze range from 45% to 57.47%. Due to the isotropy of the microstructure of transparent paper, its light scattering is also isotropic. Figure S15 in Supplementary Materials and Fig. 3(c) show the transmitted light scattering intensity as a function of angle obtained by a collimated laser on a transparent paper prepared from basswood, and the thickness is about 120 μm. The incoming laser diffuses due to the scattering in paper and exhibits an isotropic scattering effect in the light propagation cross-section plane for transparent paper [Fig. S15(c) in Supplementary Materials]. The scattered light intensity distributions in both the x and y directions [Figs. S15(a) and S15(d) in Supplementary Materials] in the 2D plane perpendicular to the light propagation direction (z direction) show the light scattering angle can be up to 57 deg. The random orientation fiber arrangement in transparent paper leads to its intense isotropic optical properties. The high transparency of transparent paper is mainly due to the following two aspects. Firstly, the lignin

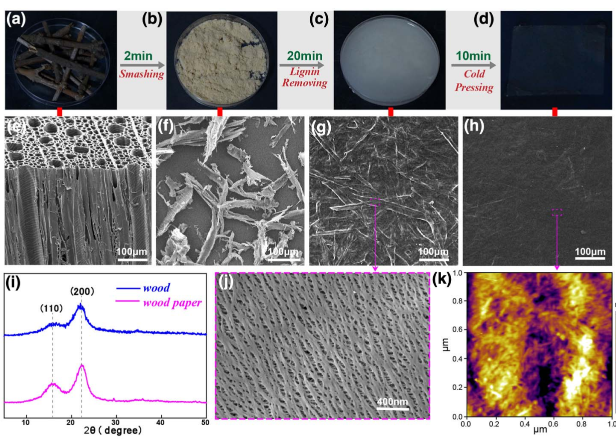

Fig. 2. Take wood as an example to show the preparation process of transparent paper and the microstructure changes of corresponding materials. (a)–(d) Photos of the main processes of typical transparent paper fabrication and (e)–(h) the corresponding microstructure changes. (i) XRD profiles of wood chips and wood transparent paper, showing its main component of cellulose I, where 2θ of 16.5° and 22.5° correspond to the (110) and (200) planes of the cellulose crystal, respectively. (j) Nanofibrils with a diameter of about 30 nm in the wood cell. (k) AFM photo of transparent paper and the nanofibers can be observed.

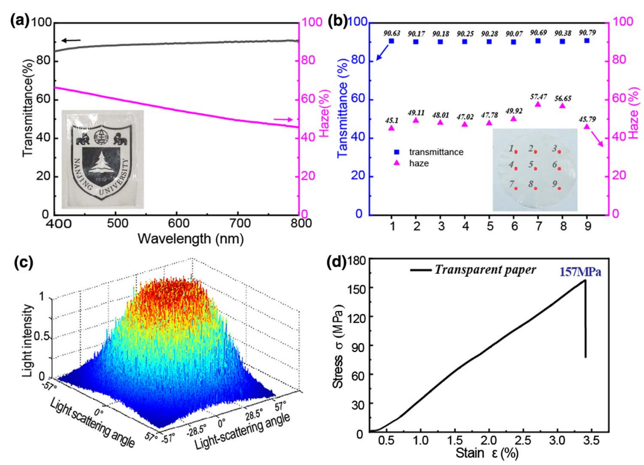

Fig. 3. Excellent homogeneous optical and mechanical properties of transparent paper. (a) Transmittance and haze of the transparent paper with a thickness of about 50 μm. The inset shows that the school badge underneath the transparent paper made of bamboo is clearly seen. (b) Transmittance and haze of different nine points on a piece of wood transparent paper. (c) Graph of the 3D scattered light intensity, showing light is scattered in an isotropic manner. (d) Tensile stress-strain curve for bamboo transparent paper.

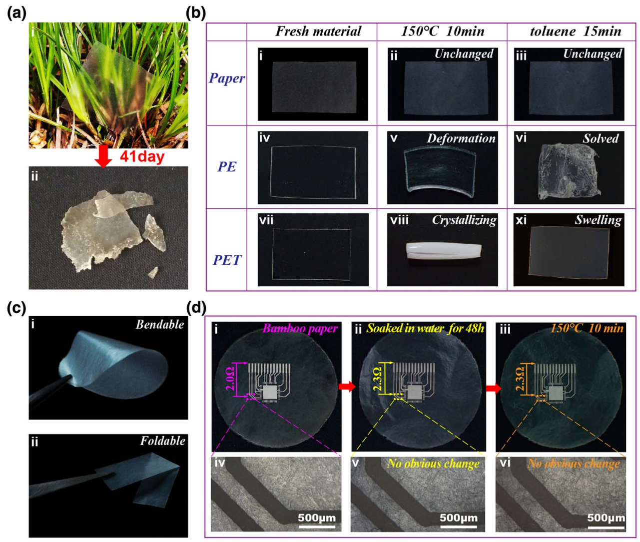

Fig. 4. Demonstration of transparent paper with good biodegradability, high temperature, and water stability as the substrate for electronic device. (a) Photos of transparent paper: (i) photo in grass soil and (ii) photo of it after 41 days. (b) (i)–(iii) Photos of original transparent paper and paper after treatment of high temperature and immersing chemical solvent, respectively. The paper was unchanged, after continuous treatment above. (b) (iv)–(vi) In contrast, PE deformed and solved after treatment of high temperature and chemical treatment, respectively, and (b) (vii)–(xi) PET crystallized and swelled after treatment of high temperature and chemical treatment, respectively. (c) (i), (ii) Transparent paper also can be bendable and foldable. (d) The bamboo transparent paper as printed circuit substrate. (d) (i)–(iii) Photos of original transparent paper with printed circuit and paper after treatment of high temperature and immersing chemical solvent, respectively. The circuit showed good conductivity. (d) (iv)–(vi) The microscope photos, correspondingly, after water and high-temperature treatment.

content, which has strong absorption in the visible light band, is removed to get rid of the light absorption loss. Secondly, the pores and space inside the paper are removed to suppress most light scattering loss by the pressing process. As a result, the total light energy loss in the transparent paper is greatly reduced, and its transparency is greatly increased. In addition to homogenous isotropic optical properties, the transparent paper has excellent mechanical strength as well, and its tensile strength is up to 157 MPa [Fig. 3(d)].

Transparent paper from different kinds of plants can be used as a substrate material for biodegradable flexible optoelectronic devices due to its excellent properties such as flexibility, favorable water stability, and thermal stability (Fig. 4). As a demonstration of its biodegradability, transparent paper made from straw was placed in grass soil and exposed to the elements in nature [panel (i), Fig. 4(a)]. After 41 days, it was degraded into pieces by the microorganisms [panel (ii), Fig. 4(a)]. This indicates that transparent paper can be biodegraded in a short period of time and achieve the goal of coming from nature and returning to nature after service. Compared with commonly used plastic such as polyethylene (PE) and poly(ethylene terephthalate) (PET), transparent paper exhibits excellent performance in terms of thermal stability and solvent resistance. It can be seen from Fig. 4(b) that the paper still retained the original morphological characteristics, while PE and PET have softened and crystallized at 150°C for 10 min. Therefore, it proves that the transparent paper can be applied under higher temperatures. Transparent paper can also maintain its intact morphology and structure in commonly used chemical solvents. There was no visible change on the paper, while PE has been dissolved and deformed, and PET has also been swollen when placed in acetone for 15 min. Besides, as shown in Fig. 4(c), the paper can be bended and folded, which enables it be used as a flexible substrate.

As a demonstration, we have shown a printed circuit on a transparent paper made of bamboo. After silk screen printing, it was annealed at 120°C for 10 min. The thickness of the silver electrode is about 6 μm, and the resistance value of the silver circuit is about 2.0 Ω [panel (i), Fig. 4(d)]. The bonding strength between the silver electrode and the transparent paper is strong enough. We soaked the sample in water for 48 h and then dried it. As shown in panel (ii), Fig. 4(d), there was no fracture on the circuit, the printing circuit was still in the path, the binding mode of the circuit with the substrate was not changed, and the wire resistance changed slightly to 2.3 Ω. From the micrograph before and after immersion in water, the fibers did not move or change. The water environment did no harm to the function of the printed circuit. After that, the same sample was placed in an oven at a temperature of 150°C for 10 min [panel (iii), Fig. 4(d)]. The printing circuit based on the transparent paper remained intact, the wires remained in the path, the resistance did not change significantly, and the shape and color of the paper did not change apparently. Obviously, heat treatment does not affect the printing circuit. From the microscope photos, it can be seen that the wire and fiber substrate still bind together tightly, and there was no obvious change on fiber morphology.

4. Conclusion

In summary, we demonstrate ultra-fast and low-cost fabrication of transparent paper based on the commonality of plant microstructures. Only 42 min is needed in the three fabrication steps of plant cell separation, lignin removal, and cold pressing. Cheap raw materials can be used, such as vine, bush, wood, bamboo, straw, branch, wheat, and other common plants. The obtained transparent paper shows high quality with a transparency up to 90.3%. It is a qualified electronic substrate material demonstrated by the circuit on it and has a good working ability after high-temperature and high-humidity treatment. Our method will greatly lower the cost of transparent paper and promote its market applications.

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