Preparation and Characterization of Polydimethylsiloxane-based Paper Transparentizing Agent and its Application in Paper Coating

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A new type of polydimethylsiloxane-based paper transparentizing agent was prepared *via* a combined method. The performance of the transparentizing agent was investigated systematically by adding and dipping, with use of the surface sizing device of a paper machine. Optimum performance was found at 30% concentration of the transparentizing agent and 30 to 45 °C of dipping temperature. Under the optimal conditions, the transparentizing t agent achieved a rapid penetration in the base paper and filled in the pores of the paper, to be further effectively adsorbed and retained on the fiber surface. The transparency of test paper reached as high as 76±0.97 %, which was 37±1.4% higher than that of the control. However, the addition of transparentizing agent reduced the mechanical strength of paper slightly. The as-prepared transparentizing agent was found to exhibit excellent application stability and biodegradability when applied in the paper machine，When used as plastic film, the transparent paper would lose its strength completely after eight weeks. The resulting transparent paper can be used to develop paper-based film and other related plant based transparent/ translucent paper, which has great potential in replacing plastic products and eliminating white pollution.

Keywords: Transparentizing agent; Transparency; Transparent paper; Film; Biodegradation

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INTRODUCTION

Potential applications of transparent paper products can be found in many fields. For instance, the use of transparent paper instead of traditional plastic films can be considered (Zhu *et al.* 2014). Mulch cultivation technology, such as agricultural mulch film, has been used in various areas of agricultural production (Cirujeda *et al.* 2013). It plays an essential role in maintaining soil temperature and moisture content as well as supporting plant roots (Marí *et al.* 2019). Mulch cultivation can maintain soil moisture, improve mineralization, and save water consumption (Sun *et al*. 2019; Ibrahim *et al*. 2020). However, large amounts of plastic film have been utilized with mulch cultivation technology, and such practices would generate lots of plastic waste and can cause serious white pollution due to the non-degradable properties of plastics (Shen *et al.* 2011). Furthermore, if these used mulch films are buried directly into the soil without additional treatment or purification process, the nature of the soil would be changed and the root system of the next batch of crops would also be affected, thus resulting in infertility soil and environmental pollution (Cirujeda *et al.* 2012; Li *et al.* 2018). Transparent paper-based mulch film derived from natural plant fibers can be used as a kind of new type of paper-based products that can be environmentally friendly and easily degradable (Minoru and Yamauch 2001).

Light can significantly influence the seed germination and seedling growth, thereby ensuring that young plants are exposed to an optimal environment for photosynthesis. (Jang *et al*. 2008). However, the traditional paper-based products have a low transparency due to their high bulk property. The most common method to produce transparent or translucent paper is to increase the beating degree of cellulose fibers prepared for papers, and to thereby increase the density and reduce the internal pores of paper product with a low bulk (Mackey *et al*. 1994). However, this method requires strict selection of pulp raw materials, and it needs additional beating time and energy consumption (Chen *et al*. 2012). Pulp with a high beating degree is difficult to be dewatered during the paper-making process of transparent paper production, thus greatly limiting the production and application of transparent paper (Zhou *et al*. 2018a). Recently, nano-cellulose has been applied as a raw material for the preparation of nanopaper with high transparency (Su *et al*. 2018). However, because of the nano size and its strong retention ability of water, nano-cellulose requires a long time to dewater during the paper-making process, thus consuming more energy and causing high costs, which is not practical in the real production (Hassan *et al*. 2016).

Impregnating paper with a transparentizing agent to improve paper transparency is an effective method. Transparentizing agents have a similar refractive index to cellulose. When filled into the paper structure, transparentizing agents can help unify the overall refractive index of the paper, and by that means increase the paper transparency (Chen *et al.* 2012). Most commonly used transparentizing agents are resins, oils, synthetic substances, and paraffin wax (Li *et al.* 2019). The traditional preparation method that relies on a transparentizing agent from only one raw material cannot meet the strict industrial demands (Vitz *et al.* 2010). In this study, different materials (modified polydimethylsiloxane, varnish and paraffin, *etc.*) would be utilized with various properties to prepare a novel transparentizing agent emulsion with high efficiency (Abbasi *et al.* 2001). Transparent paper agent was applied by combining with conventional papermaking methods to produce degradable transparent paper with low production cost and high transparency (Tiwari and Mahanwar 2018). The transparency of test paper reached as high as 76 $\pm$ 0.97%, which was 37 $\pm$ 1.4% higher than that of the control. The transparent paper meets the strict requirements during the utilization as paper base films, instead of traditional plastic film, and can effectively solve the severe white pollution issue. The transparent paper's base weight lost 52.92 $\pm$ 7.35% after 6 weeks' treatment in an outdoor environment. The development and production of agricultural mulch paper will have important theoretical and practical significance.

EXPERIMENTAL

Materials

The main agents were modified polydimethylsiloxane (BYK-310, Kingchemical Co., Ltd, Shanghai, China), varnish (xsxd-002 Guangdong Shunde Bejuly New Material Co., Ltd, Guangdong, China), and paraffin (P104801, Tianjin Jiangtian Chemical Technology Co., Ltd, Tianjin, China).

Bleached softwood pulp was provided by the specialty paper research group in Tianjin University of Science and Technology, China. Deionized (DI) water was utilized in the experiment.

All other chemicals such as emulsifying agents (9041-29-6 Sanjiang sairuida Trading Co., Ltd, Tianjin, China), curing agents (MD-50, Laiqing new materials Co., Ltd, Tianjin, China), PAE (MD-50, Laiqing new materials Co., Ltd, Tianjin, China) and penetrating agents were of analytical grade and used without further purification (Pelton *et al.* 2019).

Methods

Preparation of transparentizing agent

Modified polydimethylsiloxane was applied to prepare the transparentizing agent by an integrated method. The paraffin mixture and varnish was heated to 60 °C in a water bath (in a 3:2 ratio), followed by the addition of emulsifier and modified polydimethylsiloxane into the above mixture (Tian *et al.* 2015). Deionized water was added slowly (pre-heated to 60 °C) under magnetic stirring. The mixture was emulsified for 10 min to ensure a stable emulsion (Taiki and Masami 2012). Afterwards, the curing agent and penetrating agent were subsequently added into the above mixture. At the end of reaction, the transparentizing agent was obtained by removing the heat and allowing the agent to cool down to room temperature (Guan *et al.* 2020).

Preparation of transparent paper

Bleach softwood pulp was beaten to 20 °SR. A base paper sheet with 40 g/m^2 dry basis weight was prepared with a sheet former. During the papermaking process, 1% PAE wet strength agent was added on one side of the paper. Drying temperature was set to 80 °C with a drying time of 20 min. The dipping method was used to prepare the transparent paper. The base paper was immersed in the transparentizing agent for a certain period (another group was impregnated with deionized water as control sample), the paper was lifted with a nylon drag, followed by removing the excess transparentizing agent from the paper sheet. The dipped paper sheet was dried with a drum dryer, and the dry transparent paper was obtained for further use (Syed *et al.* 2016).

Analysis of transparentizing agent

The viscosity of the transparentizing agent was determined by placing the transparentizing agent in the viscometer at 25 ℃, using the No. 1 rotor, adjusting the rotation speed to 120 rpm/min, and measuring the emulsion viscosity until 5 stable readings were taken (Each of the measurement in this paper was calculated from the average of three parallel runs).

The refractive index was determined after drying and curing of the transparentizing agent in the paper sheet. After the transparentizing agent was dried on a flat surface to form a film, it was cut into an appropriate size and measured with an Abbe refractometer (Xuancheng Instrument Co., Ltd., Shanghai, China) (Koroleva *et al* 2017).

Determination of paper transparency

The paper transparency can be measured by the 070E Brightness Meter (L&W Co., Ltd., Sweden). The average value of green light reflection factor of single layer specimen with white standard plate backing was designated as R_w, and the average value of green light reflection factor of single layer specimen with black standard cylinder backing was designated as R_o. The final transparency was calculated from the average of three parallel runs of measurements for error analysis. The transparency was given as follows:

$$transparency\ (\%) = \frac{R_w - R_o}{R_w} \times 100\% \quad (1)$$

Measurement of tensile strength of paper

The tensile strength of paper sample was measured according to TAPPI test method T456 om-87 (L&W Co., Ltd., Sweden). The tensile strength was calculated from the average of three parallel runs of measurements for error analysis.

Measurement of bursting strength and tear index

The bursting strength of paper was tested on a 13-60-00-0002 tester of bursting strength (TMI Co., Ltd., Sweden).

The tearing strength of paper was tested on a 009 tester of Paper and board-tearing tester (L&W Co., Ltd., Sweden), and the width of paper sample was 1.5 cm.

The final results were calculated from the average of three parallel runs of measurements for error analysis.

Analyses of scanning electron microscope (SEM)

For the SEM observation of transparent paper, pre-weighed samples were put on a desiccator overnight at room temperature. The samples then were mounted on a conductive carbon tape and coated with gold before the scanning process. The SEM pictures were captured using Oxford instrument with a 10 kV beam (Seta *et al.* 2020).

Biodegradability analyses of transparentizing agent

Soil burial tests were performed on a laboratory scale to examine the biodegradability of the transparent paper-based film. The soil burial test method described by Obasi *et al*. (2013) was used with some modification. The crushed and sieved soil blocks were taken outdoors and placed in 2000 mL beakers. The transparent paper was cut into discs with a diameter of 160 mm and buried at a depth of 3 cm from the soil surface to ensure that the samples were completely buried in the soil. The soil was kept moist and watered every three days (Al-Kayssi *et al*. 2013). The soil burial test lasted for 90 days. After the test, the samples were removed, washed with distilled water, dried in the oven at 70 °C for 24 h, then cooled to 20 °C in a desiccator.

RESULTS AND DISCUSSION

Effects of Transparentizing Agent Concentration on Its Application Effect

Effects of concentration of transparentizing agent on viscosity and transparency

The high viscosity of transparentizing agent improves its penetration speed in the base paper, which is of great significance for producing transparent paper at higher paper machine speeds (Chen *et al.* 2015). The viscosity and transparency of the prepared transparency at different concentrations are shown in Fig. 1. The immersion temperature was 20 °C, and the immersion time was 5 s.

Figure 1 shows that the increased concentration of transparentizing agent increased the viscosity to a high level. When the concentration was in the range of 10% to 30%, the viscosity increased slowly. At this time, the transparency of the finished paper was positively correlated with the viscosity, and it was assumed that the concentration of transparentizing agents has equal positive effect on the viscosity. When the concentration of transparentizing agent was more than 30%, the viscosity increased greatly, but the transparency showed a downward trend. When the concentration of the transparentizing agent was in a lower range (0 to 30%), the effects on the viscosity were not apparent. At this stage, the transparentizing agent always exhibited a low viscosity level. Thus, the penetration speed in the base paper was not affected significantly when the concentration of transparentizing agent did not exceed 30%, *i.e.*, the optimum concentration was 30% (Hämäläinen *et al.* 2011).

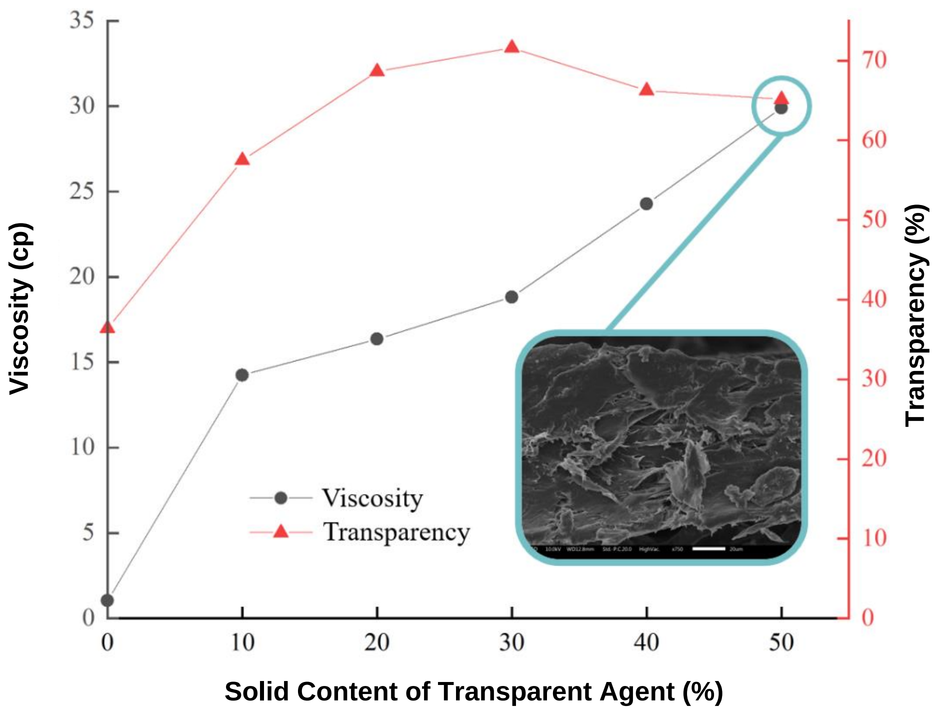

Fig. 1. Effects of concentration of transparentizing agent on the viscosity

Under the same immersion conditions, if the concentration of the transparentizing agent exceeded 30%, the transparency began to decrease. At this time, the higher viscosity would limit the dipping speed of the transparentizing agent. SEM image shows that the fluidity and permeability of the transparentizing agent were insufficient to permeate into paper and bridge effects even appeared on the surface of the paper when at a 50% transparentizing agent concentration. The transparentizing agent was stacked on the surface of paper sheet and could not penetrate into the paper sheet. Figure 1 also shows that the transparency of the paper also decreased. It should be noted that when the concentration was higher than 20%, the increasing trend of the paper transparency began to slow down. In the real production, the prepared transparentizing agent can precisely increase the transparency of paper sheets, thanks to the improved permeation ability of transparentizing agent into the paper sheet structure and fiber gaps (Zhenzhen *et al.* 2019).

Effects of transparentizing agent concentration on the paper performance

The base paper was dipped into transparentizing agent solutions with different concentrations under the same conditions (40 °C, immersion for 5 s, and the same liquid level) to investigate the effects of transparentizing agents of different concentrations on the performance of the paper. The experimental results are shown in Table 1.

Table 1. Effects of Transparentizing Agent Concentration on Paper Sheet Performance

Transparentizing Agent Concentration (%)	0	10	20	30	40	50
Basis weight (g/m^2)	41.60±2.75	51.30±2.13	58.40±4.06	60.10±3.37	52.00±3.06	49.70±6.66
Density (g/cm^3)	0.53±0.014	0.69±0.007	0.74±0.009	0.75±0.006	0.71±0.011	0.67±0.003
Burst index ($kPa{\cdot}m^2/g$)	4.37±0.24	3.10±0.23	3.03±0.19	2.57±0.07	2.60±0.21	2.52±0.17
Tensile Index (N·m/g)	52.83±1.36	37.20±1.14	32.41±0.85	30.53±1.58	30.12±0.61	29.87±0.57
Tear index ($mN{\cdot}m^2/g$)	8.31±0.26	5.78±0.57	4.81±0.19	4.67±0.25	5.37±0.51	5.91±0.27

The transparency of paper sheet increased with the augment in the dipping amount, but simultaneously, the strength performance of the paper sheet including burst tensile and tear indexes decreased, which shows that the strength loss occurred when the transparent material filled the fiber gaps (Table 1). Although it increased the uniform refractive index in the paper sheet, the hydrogen bonding and the bridging effects between fibers would be blocked and weakened. Therefore, a suitable transparentizing agent concentration should be determined to ensure that at this concentration, the active substances in the transparentizing agent can fill into the inter-fiber pores and maintain the strength properties of the paper sheet. It can be concluded that the optimum transparentizing agent concentration was 30%.

Effects of Dipping Time on the Transparency Property

Treatment of base paper to improve its transparency can be affected by various factors. The immersion time of paper sheet during the dipping process determines the retention rate of transparentizing agent in the voids of the paper sheet. It is understandable that extending the immersion time can facilitate a greater amount of transparentizing agent filling into the paper sheet. However, when the immersion time reaches a critical level, the transparentizing agent emulsion will fill into the internal pores of the paper. If the immersion time is overextended, the transparency of paper sheet won’t be improved, but there can be a serious loss of paper strength, and the speed of the paper machine will be significantly reduced (Fang *et al.* 2013). Therefore, the effect of impregnation time on paper was explored. The base paper was prepared by transparentizing agent impregnation at 20 °C with 30% concentration of transparentizing agent dosage.

Effects of dipping time on paper transparency

Figure 2 showed that the transparency of the paper sheet increased rapidly in a short period during the initial stage of dipping (the dipping temperature was 40 °C), reaching 70.6±0.41% at 5 s, which is higher compared to initial paper before dipping (34.2±0.67%). After 5 s, the pores in the paper were nearly saturated, the transparency of the paper sheet was unchanged and the transparency after immersion for 60 s was only increased by 2.6±0.83% with a dipping time of 5 s. The transparentizing agent developed in this paper fully penetrated the paper sheet quickly, thus improving the transparency, which is beneficial to the practical application on the paper machine.

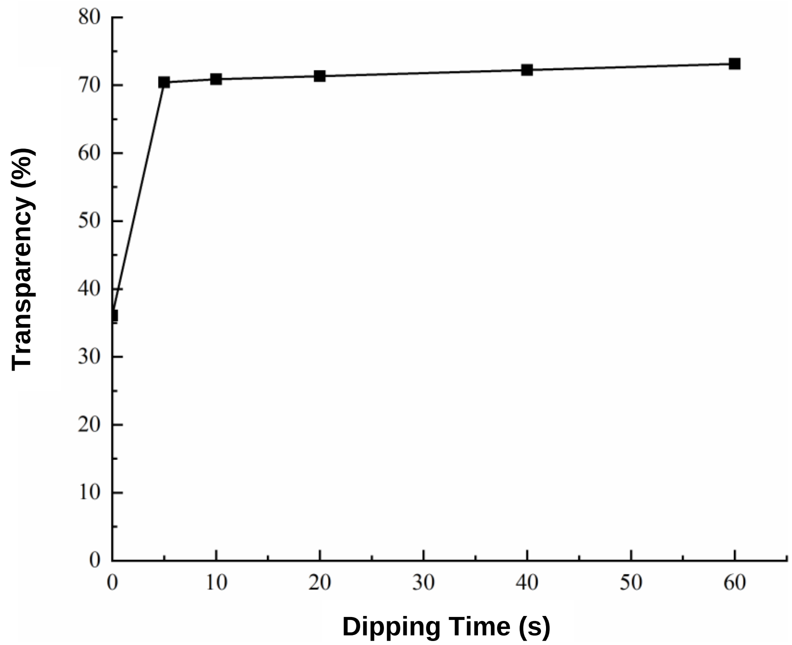

Fig. 2. Effects of dipping time on transparency of paper

Effects of dipping time on paper strength property

As shown in Fig. 3, with the increase of the immersion time in the agent, the strength properties, for example, tensile index of the paper sheet, were found to be decreased significantly, which can be ascribed to the breaking up/blocking effect of hydrogen bonding between fibers induced by the addition of transparentizing agent (Zhang *et al.* 2014). Besides, it was also found that the long-term immersion time can improve the moisture content of paper sheet, thus impeding the drying efficiency in the paper machine, which is undesirable in paper production (Zhou *et al.* 2018b). The optimal dipping time was 5 s, considering the results shown in Fig. 3.

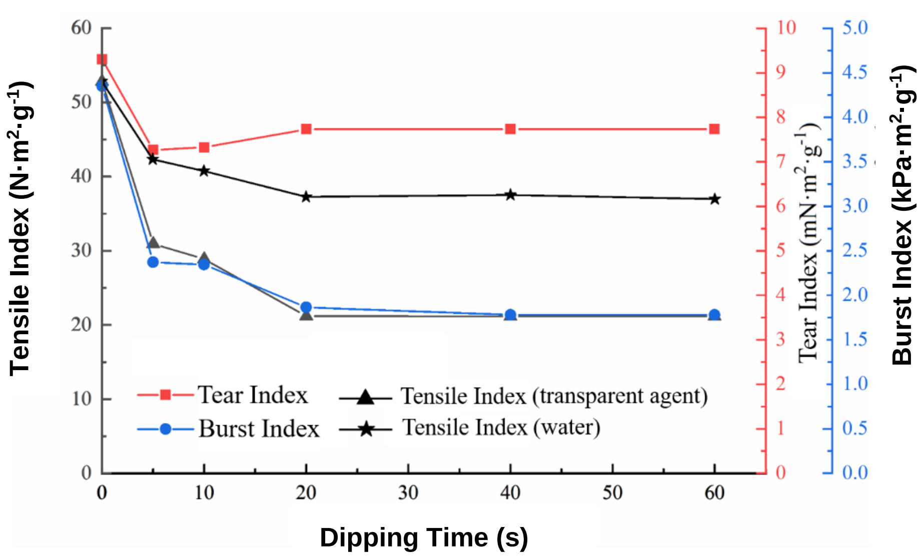

Fig. 3. Effects of dipping time on strength of paper

Effects of Dipping Temperature on the Transparency Performance of Paper Sheet

Effect of dipping temperature on paper transparency

Experimental results in Fig. 4 showed that the transparency of paper sheet did not change significantly with the increasing of the dipping temperature in a specific range (the dipping time was 5 s). The transparency of the paper sheet reached 76.04±1.44%, which was 37.38±0.56% higher than that of the base paper. When the temperature exceeded 45 °C, the transparency decreased. Theoretically, higher coating temperatures could reduce the viscosity of the transparentizing agent, but results indicate the opposite. Because the emulsion contains varnish, and it will form a thin film on the liquid surface at a high temperature, then the refractive index of agent was changed. Alternatively, increasing temperature will accelerate the transparentizing agent's liquid phase components' volatilization, causing the transparentizing agent's viscosity to increase rapidly, which would dramatically affect the penetration efficiency within a limited time (Tang and Yan 2017).

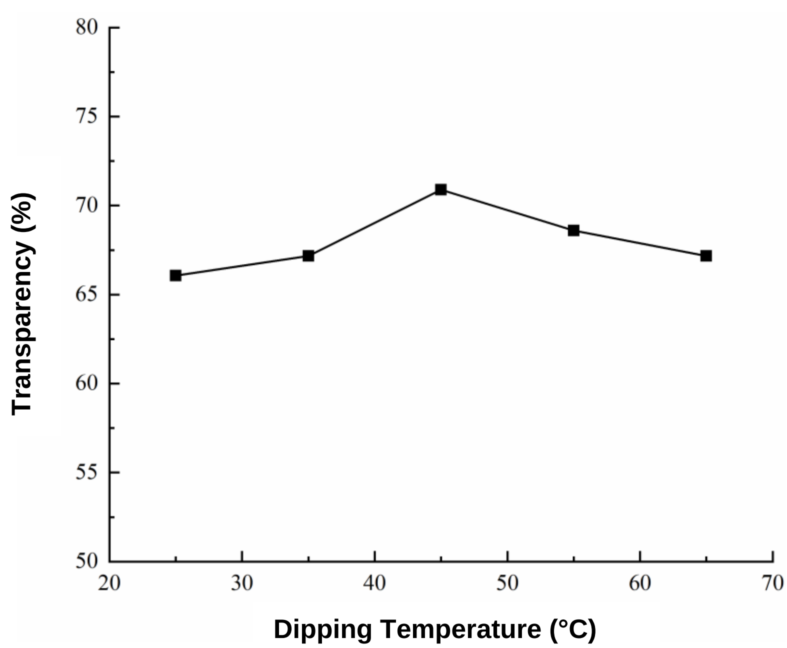

Fig. 4. Effect of dipping temperature on transparency of paper

Effects of dipping temperature on paper strength property

Figure 5 shows that the relatively low immersion temperature was beneficial to maintaining the strength of the paper. There was an increasing tensile index of the transparent paper at 25 to 35 °C of dipping temperature, allowing for maintaining the transparent paper at a higher level.

The increase of dipping temperature would deteriorate the transparency performance, due to the quick-drying rates at high temperature (Huang and Petermann 2015). This phenomenon could happen possibly because at high dipping temperature (>35 °C) the fibers of the paper started to be softened and then decreased the paper strength (Kouko and Retulainen 2014). Similar results can also be found in the tear and burst index.

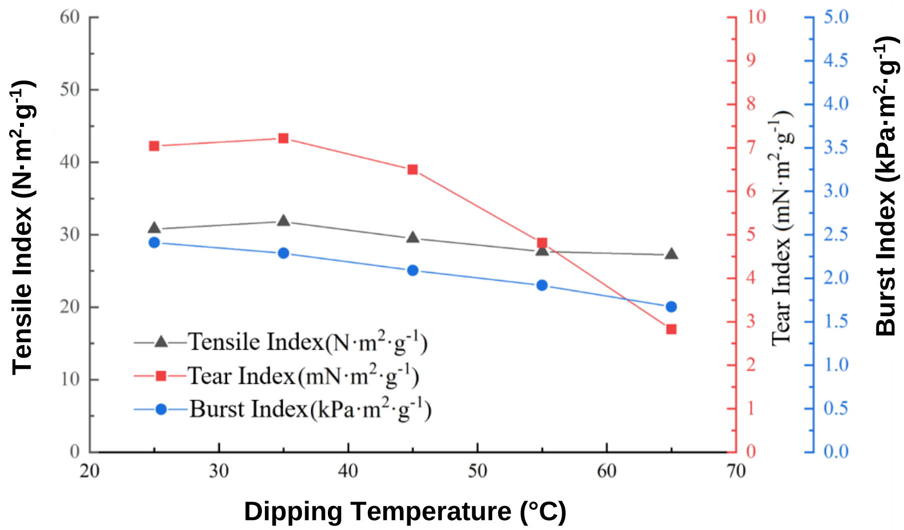

Fig. 5. Effects of dipping temperature on strength of paper

Distribution of Transparentizing Agent in Paper

The control paper sample and the finished transparent paper were observed in Fig. 6. Figures 6(a) and 6(b) show that the addition of transparentizing agent can improve the surface smoothness of paper, and fill into the voids/ pores between fibers in paper sheet, thus improving the transparency of paper sheet.

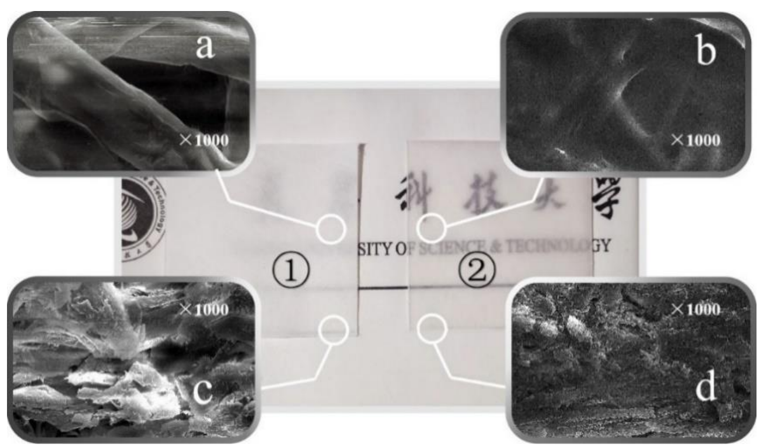

Fig. 6. Comparison of the effect of base paper and transparent paper: ① base paper, ② transparent paper, (a) SEM images of the surface of base paper, and (b) SEM images of the surface of transparent paper, (c) SEM images of the cross section of base paper, (d) SEM images of the cross section of base paper

Similar results can also be obtained from the cross section of the paper samples, as shown in Figs. 6(c) and 6(d). From the cross-section of the paper, the transparentizing agent had filled into the pores/ voids of paper sheets, there were no apparent boundaries between the fibers, only some small granular substances were loaded on the fiber surface (Bouramtane *et al.* 2019).

Stability Analysis of Transparentizing Agent

There were some key factors affecting the stability of the transparentizing agent emulsions. During the preparation and pumping stages, the transparentizing agent emulsion can be unstable under mechanical shearing treatment. In this case, the transparentizing agent emulsion was processed in a high-speed centrifuge (XiangYi centrifuge instrument Co., Ltd., Tianjin, China) at 3000 rpm for 15 min. It can be concluded that no delamination or precipitation was found in the transparentizing agent emulsions, indicating that the prepared transparentizing agent emulsion possessed excellent mechanical stability.

The transparentizing agent emulsion may be in a high-temperature environment during the storage process, transportation, and process. Therefore, the transparentizing agent was placed in a sealed glass vial and placed in a preheated oven at 60 °C for 5 days, to ensure that the emulsion will not break in that described above. There was no precipitation, delamination, or demulsification, indicating that the transparentizing agent emulsion can be stored for a long time in a low-temperature environment.

Another method to keep transparentizing agent is to dilute the transparentizing agent emulsion into different concentrations, the lowest to reach a concentration of 0.5%, and place them in test tubes with a liquid column height of 20 cm. The diluted transparentizing agent emulsion was stored at room temperature for more than 30 days without any delamination, precipitation, or demulsification. The test results showed that transparentizing agent emulsion had better dilution stability and storage stability.

Analysis of Biodegradability of Transparency Paper Sheet

The degradation performance of transparency paper in the soil was investigated, as shown in Fig. 7. This test simulated the laboratory's actual degradation process and analyzed the biodegradability of transparent mulch paper using the soil burial method. The results showed that the paper film's weight loss and strength loss co-occur during the degradation process. After four weeks of burial, the weight loss reached 30±6.30%, and the strength loss exceeded 50±3.12%. After being buried for eight weeks, the weight loss reached 52.11±7.81% and the strength loss reach almost 100%.

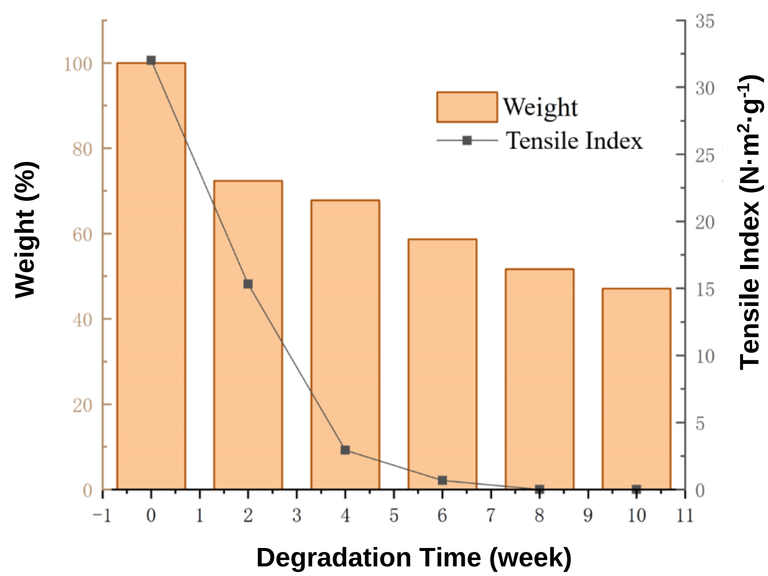

Fig. 7. Effects of degradation time on weight and tensile strength of paper

The actual degradation process of mulch paper is quite complicated (Ming and Chen 2020). In addition to photodegradation, thermal oxidation, weathering, and hydrolysis, soil also contains many microorganisms, including bacteria, molds, yeasts, and actinomycetes (Alvarez *et al.* 2006). Some types of bacteria and molds could also produce sufficient amounts of cellulase (Yaacob *et al.* 2015). The cellulose fibers within mulch paper would be degraded into various products under the above conditions. Those complex degradation products are interrelated to form the entire biodegradation process of transparent paper (Yang *et al.* 2015), which indicating an excellent biodegradation ability of the prepared mulch paper.

CONCLUSIONS

In this study, transparentizing agents for transparent paper products have been successfully prepared from natural fiber base paper via a feasible dipping method. The optimum conditions to prepare the transparency paper are: 30% of transparentizing agent concentration, dipping temperature of 30 to 45 °C, immersion time of 5 s. The prepared transparentizing agent can quickly penetrate the paper structure and the transparency of paper sample can reach as high as 76±0.97%, which was 37±1.4% higher than that of the control. The relative strength properties of the paper were reduced after the application of the transparency agent. The paper transparentizing agent prepared in this paper had a relatively low viscosity, which was beneficial to the dipping process treated via surface sizing device of a paper machine. The transparentizing agent had a better absorption and retention rate in the base paper, thus improving the filling effect in the pores of the base paper, and the paper transparency as well. Biodegradability tests confirmed that the prepared transparent paper possessed an excellent transparent stability and biodegradability. The proposed method for the preparation of transparentizing agent and transparent paper will play a great potential in the replacement of traditional plastic based products, aiming to eliminate the white pollution.

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