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A novel measurement of contact angle on cylinder-shaped lignocellulosic fiber for surface wettability evaluation

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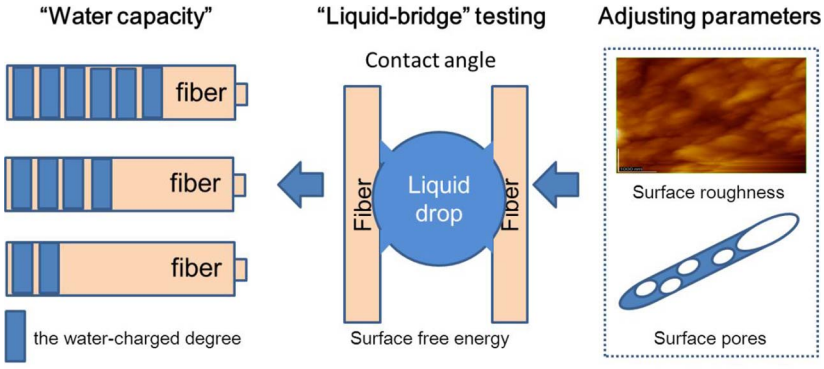
GRAPHICAL ABSTRACT



The charged degree evaluation with surface free energy and contact angle based on the "liquid-bridge" testing (by adjusting surface roughness and pores).

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ABSTRACT

Lignocellulosic fiber, composed of hydrophilic component (carbohydrates, including cellulose and hemicellulose) and hydrophobic component (lignin), is widely used in bio-composites, paper and paperboard products, and other fiber-based bio-products. The surface wettability of cylinder-shaped fibers, focusing on the liquid spreading ability on fiber surface, essentially has an important effect on many kinds of fiber properties. A novel measurement of contact angle ("liquid-bridge" testing) was utilized for the calculation of surface free energy, expressed as the evaluation of fiber surface wettability. The results showed that the fiber contact angle determined by the "liquid-bridge" testing was quite similar to that measured by the traditional sessile drop technique. The fiber surface free energy increased dramatically from 46.63 mJ m^{-2} to 54.45 mJ m^{-2}, calculated by the fiber contact angle with water (74.30 ° to 43.24 °) and glycerol (64.50 ° to 49.41 °), when the PFI mechanical treatment was strengthened up to 15,000 rev. This novel method has also been applied to test the positive variation and sensitivity of surface wettability with the increase of fiber surface roughness and surface pore size.

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1. Introduction

Lignocellulosic fiber, which is mainly consisted of hydrophilic component (carbohydrates, including cellulose and hemicellulose) and hydrophobic component (lignin), is widely used in daily life for its reusability and biodegradability [1–3]. Fiber surface properties, including surface composites, surface charge, surface wettability and so on, play a critical role in fiber applications by influencing the interfacial properties between fibers and other materials directly. Typically, the fiber surface wettability, the ability for a certain liquid (especially water) to spread onto the fiber surface, is so important a characteristic affecting the fiber properties, such as fiber swelling and softness. In textile materials, fiber with a good surface wettability will be easy to be dyed and well glued with adhesives. Similarly, the fiber surface wettability has a strong influence on the interfacial compatibility and mechanical properties of fiber-based bio-composites [4].

As a new point of view to evaluate the fiber surface wettability, the fiber surface free energy has attracted attentions in some kinds of fiber applications recently, such as sisal fiber-based materials [5] and synthetic fibers in textile materials [6]. For lignocellulosic fiber-based products, the free energy has been emphasized as an indicator to analyze the surface coating and surface wettability [7,8]. However, limited literature could be obtained regarding measuring the surface free energy of lignocellulosic fiber raw materials.

The contact angle is mostly used for the calculation of surface free energy [9,10]. Wilhelmy force method [11] and sessile drop techninque [12] are two major determinations for contact angle. The Wilhelmy force method is accomplished by determining the wetting force on fiber when it is immersed into water, which is largely dependent on the fiber length put into water. Both of the wetting perimeter and the buoyancy force should be quantifiable based on the fiber dimension in this determination. It's reported that the surface contact angle of some long fibers could be tested by this method [13]. However, the Wilhelmy force method doesn't work well for natural lignocellulosic fibers with an irregular dimension.

The sessile drop technique is manufactured by analyzing the profile of the liquid drop onto the substrate with optical instruments, which has also been used to measure the contact angle of some long fibers (natural cellulose fiber and polyester fiber) [14]. The fiber surface free energy determined by the sessile drop technique is influenced by raw materials, the liquid used, the temperature and the humidity. Above all, a horizontal plane of the substrate is usually needed in the sessile drop technique. However, the lignocellulosic fiber is a cylinder-like material, whose surface is not a horizontal plane. It's hard to get a stable water drop onto the fiber surface, whose roughness also leads to the inhomogeneity of the angle between the fiber and the water drop [15].

In our work, the surface contact angle of lignin-rich lignocellulosic fibers will be determined with a novel method called "liquid-bridge" testing developed from the conventional sessile drop technique. The fiber contact angle determined by both techniques will be compared with each other to verify the accuracy of the "liquid-bridge" testing. The surface free energy would be calculated according to the contact angle to have a better understanding of the fiber surface wettability. The influence of the fiber surface roughness and the pore characteristics on the fiber surface wettability will also be investigated.

2. Materials and methods

2.1. Materials

An unbleached pine thermo-mechanical pulp (TMP) fiber was classified by Bauer-McNett fiber classifier (TMI 8901-5, USA) after a good latency pretreatment (slow stirring was done in a water bath at about 95 °C for 30 min), and the fibers of 2.4–2.6 mm long and 40.1–40.5 μm wide was used as raw material in this study. The fiber was then mechanically treated with PFI refiner (Frank-PTI 71-03-04-0002, Germany), and subject to pretty less fibrillation. The fiber consistency was 30% and different revolutions (0 r, 5000 r, 10,000 r, 13,000 r, and 15,000 r) were made.

2.2. Contact angle determination

About 0.05 g of the treated fibers was then suspended in water with a consistency of 0.1%. The suspension was drained onto a 200-mesh filter cloth by using a TAPPI standard handsheet former (Lab Tech, Canada). The fibers on the filter cloth were then air-dried at a room temperature (25 °C) for 24 h in preparation of the contact angle determination.

The surface contact angle determination of fibers was manufactured at a temperature of 25 °C and the moisture content was also kept the same. Two fibers were picked up from the filter cloth and put onto a specially made slide in a parallel way as shown in Fig. 1. The distance between two parallel fibers was kept from 0.2 mm to 2 mm. The small distance made the liquid drop possible to stay between two fibers so that a better graph could be obtained from the microscope (C-FLED2, Japan) for the contact angle calculation. The contact angle was measured by the graph with the help of the software accompanied with the microscope. Dozens of fibers were tested for each sample. Two kinds of liquids used in this project were the deionized water and the glycerol.

Some of the fiber sample was then made into the plane for the measurement by the traditional sessile drop technique with the KRUSS contact angle tester (DSA 100, Germany). A fiber was pasted onto the sample state with double faced adhesive tape and a water drop of 10 μL was dropped onto the fiber. The charge coupled device (CCD) was used to record the whole process. The final contact angle was calculated using the software accompanied with the instrument using ellipse method [16].

2.3. "Water capacity" analysis

The water retention value (WRV) was used as the expression of "water capacity", which was measured based on the TAPPI method um-256 procedure. The centrifugal force used was (3000 ± 50) g (g is the acceleration of gravity, 9.81 m s^{-2}). About 0.15 g of fiber sample was

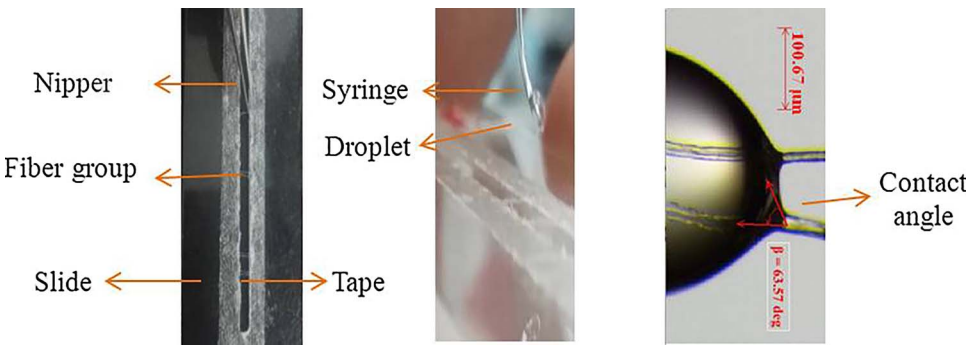

Fig. 1. The determination of lignocellulosic fiber surface contact angle by the "liquid-bridge" testing.