The investigation of producing bacterial cellulose fibres through hand-spun

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Abstract. Nanocellulose fibres can be hand-spun from different intermediate states, such as nanocellulose paper and filter cake, which are made from the BC suspension as well as wet pellicle (WP) and dry pellicle (DP) from BC pellicles. In this study, it can be concluded that increasing the hanging weight can increase the Young’s modulus and the tensile strength of fibres. Nanofibres produced from BC pellicles as raw material have better performance than those made from BC suspension. The best properties obtained from the fibres produced from wet pellicles and suspended to a 100g hanging weight upon drying are Young’s modulus (33.8 GPa), tensile strength (610 MPa) and elongation at break (3.6%).

1 Introduction

Cellulose is an organic compound which can be widely found in wood and plant fibres [1]. It can also be formed by microorganisms [2]. The strength and stiffness of a single nanocellulose fibre is 1.6-3GPa and 100-160 GPa [3][4]. As a result, it can replace the glass fibres because of higher modulus and higher strength as well as greater availability. As highlighted by the increasing research papers, attention has been paid to the development of nanocellulose as reinforcement for green composites [5]. Nowadays, nanocellulose fibres [6] attract a lot of research interest as an alternative to nanopapers. Compared with nanopapers, the alignment of nanofibres along the fibre axis during the manufacturing process is better and then it will lead to the manufacture of unidirectional composites [7]. In this study, a novel approach has been introduced to manufacture the continuous nanocellulose fibres without the use of chemicals: hand spinning. The produced fibres are supposed to exceed the best properties of nanofibres found in the literature.

2 Material and methods

Bacterial cellulose in the form of nata de coco was purified in deionized water (150g of nata de coco in 3.5L of deionized water) with 0.1 M of NaOH (VWR, 98.5% purity) at 80°C for 2 hours. Then the purified bacterial cellulose was cooled at ambient temperature for 1 day. The obtained BC pieces were retrieved in a sieve and rinsed for several times with deionised water until the PH of filter liquor is less than 10 so that the excess NaOH can be removed. The resulting BC pieces were mixed with water and put into an 800W blender (Breville VBL065) to obtain a homogeneous suspension. A centrifuge (Sigma 4-16S, UK) was used to separate BC from the high PH water to reach neutral PH (7-8). Then deionised water was added to the aqueous suspension to make the weight percentage of BC 1.5wt-2wt%.

Two different BC states were used as raw materials: BC pellicle and BC aqueous suspension. Then the intermediate states: filter cakes and dry papers were produced from the BC aqueous suspension while dry pellicle and wet pellicle were obtained from BC pellicle. A certain amount of aqueous suspension was added in an 800W blend with deionised water and blended until the dispersion of nanocellulose becomes homogeneous. Then the obtained solution was vacuum filtered on a filter paper to form a filter cake. The filter cake was first dried by the filter papers and then put in a hot-press at 120°C to reduce the residual water. These intermediate states were cut into narrow strips, clamped at each end by small clips, and then spun manually to keep them under tension. The number of twists depends on the length of fibres: 10 twists/cm (one twist means a 360° rotation of one clip). After twisting, a mass was connected to each formed fibres. The hanging weight (weight of the mass) are 2, 50, 100g. Water was sprayed on these fibres to make a possible elongation. These hanged fibres were left hanging for 1 day at ambient temperature to remove the moisture inside.

3 Result and discussion

3.1 SEM analysis

Scanning electron microscopy (SEM) was used to characterise the morphology of the produced fibres. Figure 1 shows the SEM figures of fibres made from different intermediate states (filter cakes, dry papers, dry pellicle and wet pellicle) stretched with a 100g mass. It...
can be observed that the surface of these fibres depends a lot on their origin material. To be more specific, fibres produced from dry paper and filter cakes have a rough surface while those produced from dry pellicle and wet pellicle have a smoother surface. In terms of the fibres produced from dry paper and filter cake, the cross section diameter for fibres varies a lot along the fibre axis. It is due to the fact that it is difficult to hand spin these fibre and spread the twist along the fibre length correctly. However, the diameter of the fibres produced from dry pellicle and wet pellicle is more constant which means that the formed fibres are more homogeneous.

As shown in figure 2, the fracture surface of the fibre produced from these four intermediate states is shown in these SEM figures. The effect of twisting can be clearly found. The pores inside the fibres indicated by the black area in these figures are caused by the failure process. The layer structure of fibres produced from the filter cake is different from those from dry paper. This is because hydrogen bonding occurs between these two layers during the desiccation. Moreover, the cross sections of fibres produced from dry pellicle and wet pellicle are flatter and denser than those produced from the dry paper and filter cake.

**Fig.1** Scanning Electron Microscopy of the hand-spun fibres (Dry paper (top left), filter cake (top right), dry pellicle (bottom left) and wet pellicle (bottom right)

**Fig.2** Scanning Electron Microscopy of the fracture surfaces of hand-spun fibres

### 3.2 Tensile tests

These 12 kinds of fibres undertook the tensile tests according to the standard ASTM C1557-14. The tensile tests were performed by the means of a 200 N loadcell microtest (DEBEN UK Ltd, Woolpit, UK) at a crosshead speed of 0.5 mm/min. An Imetrum optical camera (Imetrum, Bristol, UK) was used to measure the strain, and therefore avoid imprecision due to the microtest compliance. The strain at failure was corrected when necessary according to ASTM D638-10. Every tensile results presented in this report are an average of 5 samples. The results are shown in figure 3 (tensile strength and Young’s modulus) and figure 4 (strain at failure).

It can be found that fibres produced by wet pellicle have the highest strength and modulus. The strength and modulus of the rest fibres: Dry pellicle > filter cake > dry paper which indicates that fibres produced from BC pellicles have better performance than those produced from BC suspension. This is because the nanocellulose network in the pellicle is stronger than that one in the BC paper since the mechanical and chemical treatments like blending will damage the nanofibres’ network in the BC suspension. The obtained maximum strength and modulus are 33.8 GPa and 610 MPa. The reason for the better performance of fibres produced from wet pellicle than those produced from dry pellicle is that the nanofibres in the wet pellicle is more mobile than that in the dry pellicle, which makes the nanofibre orientation caused by stretching during drying much easier for the fibres.

In addition, it can also be found that the heavier the hanging weight, the greater the tensile properties. In terms of the fibres produced from wet pellicle, with increasing hanging weight from 2g to 100g, the modulus and strength increases from 7.9 GPa to 100g, the modulus and strength increases from 7.9 GPa to 33.8 GPa and 220 MPa to 610 MPa, respectively. The improvement of mechanical properties is more significant when comparing the results of fibres produced from paper and dry pellicle. This may be caused by the fact that nanofibres orientation in the macrofibre axis direction upon desiccation is supposed to be enhanced when the hanging weight is increasing.

**Fig.3** Tensile test results of the fibres. (DP means dry pellicle, WP means wet pellicle, FC means filter cake and P means dry paper)

Furthermore, it can be found on figure 4 that the strain at failure of fibres decreases with increasing hanging weight. This is because these fibres have already been pre-stressed by the mass and then it will lead to the pre-strain on the fibres. The more hanging weight, the more pre-strain and less apparent strain at failure are.
Fig. 4: Strain at failure of the hand-spun fibres (DP means dry pellicle, WP means wet pellicle, FC means filter cake and P means dry paper)

4 Conclusion

Hand spun bacterial cellulose fibres were obtained through different intermediate states: filter cakes and dry papers made from a BC suspension as well as wet pellicles and dry pellicles. The effects of the BC intermediate state and the hanging weight during drying were investigated. The mechanical properties increase with increasing hanging weight during drying because of the nanofibre orientation effect. A regular circular cross section for the fibres produced by wet pellicles is confirmed through SEM. The best mechanical properties obtained in our study are using wet pellicles as intermediate state. The measured Young’s modulus (33.8 GPa) and tensile strength (610 MPa) are superior to any continuous fibre made of nanocellulose in the literature. Thus, it can be concluded that the hand spun bacterial cellulose fibres are very promising. In the future, more works should be done to observe the orientation phenomenon at the nanoscale and then the hierarchical structure of fibres can be determined. In addition, further studies on BC nanocellulose fibres can also help the development of BC production to a greater scale.

References

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