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Preparation and Properties of Natural Cellulose Fibres from *Broussonetia papyrifera* (L.) Vent. Bast

Abstract
Natural cellulose fibres from Broussonetia papyrifera (L.) Vent. (BP) bast were systematically investigated in this paper. To begin with, BP fibres were successively extracted from BP bast by four different degumming methods, among which the microwave-assisted method exhibited high efficiency. It was found that non-cellulose substances were sufficiently removed or reduced after the degumming process, but the cellulose I structure had not changed from bast to fibres based on the XRD and FTIR results. Meanwhile the BP fibres exhibited high crystallinity (75 ~ 77%), high breaking strength (2.19 ~ 2.39 cN/dtex) and a remarkable moisture region (6.3 ~ 8.7%), but low breaking elongation (1.0 ~ 2.1%). All those results indicated that the BP fibres had properties resembling those of traditional natural cellulose fibres (e.g. cotton and flax); therefore they could be viewed as a promising alternative source for natural cellulose bundle fibres.

Key words: *Broussonetia papyrifera (L.) Vent. (BP), natural cellulose fibre, fibre extraction, microwave-assisted.*

Introduction

Broussonetia papyrifera (L.) Vent. (BP) or paper mulberry, a fast growing tree of the Moraceae family, is widely distributed in East Asia and the Pacific Islands [1]. This plant exhibits a range of attractive features, such as wide adaptability, a strong germinating ability and an excellent regeneration capability [2, 3]. Furthermore it could be easily grown in some severe conditions, e.g. in an upland area with moist alluvial soil, such as in stream banks and valley floors.

Furthermore the *Broussonetia papyrifera (L.)* Vent. was termed the "money-making tree" because its bast and stem core are prime raw materials for high quality paper and food packaging paper [4, 5]. Consequently previous researchers have mainly focused on the application of BP for papermaking and pulping purposes, and to our knowledge few investigations have been made to explore other applications [6, 7].

In fact, as supplementary sources for natural cellulose fibres, some common bast plants have already been investigated , including jute [8, 9], hemp [10, 11] and flax [12]. Recently a few other novel natural cellulose fibres were extracted with acceptable textile properties, from e.g. hop stems [13], corn stalks [14], velvet leaves [15], wheat and rice straw [16], switchgrass [17], and soybean straw [18], as well as some regenerated fibres (pineapple leaf, banana) [19].

Generally, natural cellulose bundle fibres, such as flax and hemp, should possess certain residual pectin and other binding materials after degumming to bind individual short single fibres together into bundles [12]. Hence the residual gum content was a key parameter to evaluate fibre quality. Also the residual lignin content significantly influenced the quality of fibres. For instance some researchers[20] indicated that fibres with little residual lignin appeared glossy and turned a white color with a lignin content up to 0.8%. However, if the lignin content was too high, the fibres became yellowish and difficult for further processes. Besides the residual gum and lignin contents, the fibre breaking tenacity was another important parameter to assess the degumming process.

In this study we attempted to extract textile fibres from *Broussonetia papyrifera (L.)* Vent. (BP) bast. Four different methods were successively applied to extract BP fibre so as to compare their feasibility and effectiveness. Then the BP bast and fibres obtained were characterised by XRD, FTIR and SEM. Furthermore some important properties of the fibres obtained were measured and compared with those of traditional cellulose fibres including cotton and flax.

Experiments

Material

The BP trees used in our study were planted in the upland area of Shandong Province, China. The BP stem was cut and its bast was ripped, followed by the fibre extraction procedure.

Fibre extraction

Four methods for BP fibre extraction were successively employed for both pretreatments and degumming processes, illustrated in **Figure 1**. During the pretreatment or degumming process, traditional heating (water bath) was replaced by microwave heating in *method three* and *four*. We named the fibres extracted by the four methods as Fibre B, Fibre C, Fibre MA and Fibre MP, respectively. In fact, the alkali-H_2O_2 one bath degumming approach was firstly developed by our lab [21], and microwave utilisation has been reported in hemp extraction [22]. Among all the methods, a series of experiments were arranged to determine the final optimum conditions for fibre extraction.

The parameters in each process step were finally determined as:

Acid pretreatment: H_2SO_4 solution (1 ml/l), temperature 50 °C, liquor ratio 1:15, water bath heating for 60 min.

Enzyme pretreatment: alkaline pectinase as (activity 4000 U/g) the enzyme and its solution (4 g/l), temperature 50 - 55 °C, liquor ratio 1:15, water bath heating for 240 min.

Microwave assisted acid pretreatment: H_2SO_4 solution (1 ml/l), temperature 45 °C, liquor ratio 1:15, microwave reactor (Apex, Shanghai EU Microwave Chemistry Technology, China) for 15 min at a power of 600 W.

Alkali-H_2O_2 one bath degumming: NaOH solution (5 g/l), $MgSO_4 \cdot 7H_2O$ solution (0.1 g/l), H_2O_2 solution (4 g/l), ATMP (Amino Trimethylene Phosphonic Acid) and magnesium

Qu L, Tian M, Guo X, Pan N, Zhang X, Zhu S. Preparation and Properties of Natural Cellulose Fibres from *Broussonetia papyrifera* (L.) Vent. Bast.
FIBRES & TEXTILES in Eastern Europe 2014; 22, 4(106): 24-28.

chloride ($MgCl_2$) as the H_2O_2 stabiliser (1.2 g/l), temperature 99 °C, liquor ratio 1:15, water bath heating for 150 min.

Microwave assisted alkali-H_2O_2 one bath degumming: NaOH solution (5 g/l), $MgSO_4 \cdot 7H_2O$ solution (0.1 g/l), H_2O_2 solution (4 g/l), H_2O_2 stabiliser ($MgCl_2$) (1.2 g/l), temperature 99 °C, liquor ratio 1:15, microwave reactor (Apex, Shanghai EU Microwave Chemistry Technology, China) for 50 min at a power of 600 W. All the chemical reagents used were of analytical purity.

Fibre composition

Three major compositions of BP bast (cellulose, lignin and ash) were determined. The cellulose content was determined as Acid Detergent Fibre (ADF) by the AOAC method 973.18 [23]. Lignin in the fibres was determined as Klason lignin based on ASTM method D1106-96, and the ash content was obtained according to ASTM method E1755-01. Five replications of BP bast were simultaneously measured and their average values defined as standard results.

Initial performance assessment of BP fibres

Three parameters: the residual gum content, residual lignin content and tenacity, were employed to evaluate the primary quality of the BP fibres obtained, where the residual gum and residual lignin contents were determined using the process described in [23], while the fibre tenacity was measured according to Standard ASTM D 1445-75.

X-ray diffraction (XRD)

The crystal structure of BP bast and all four fibres (Fibre-B, Fibre-C, Fibre-MA and Fibre-MP) were measured by powder X-ray diffraction. Powders of the BP bast and fibre samples were obtained by firstly grounding the materials in a Wiley mill and then screening by 60 meshes prior to pressing into testing specimens. Then their X-ray diagrams were successively recorded on a vacuum X-ray camera mounted on a Philips X-ray generator (Philips PC18kW, the Netherlands) operated at 20 mA and 30 kV.

The fibre crystallinity C_r (%) was calculated by ***Equation 1*** [24].

$$C_r = \frac{S_e}{S_e + S_n} \times 100, \text{ in } \% \qquad (1)$$

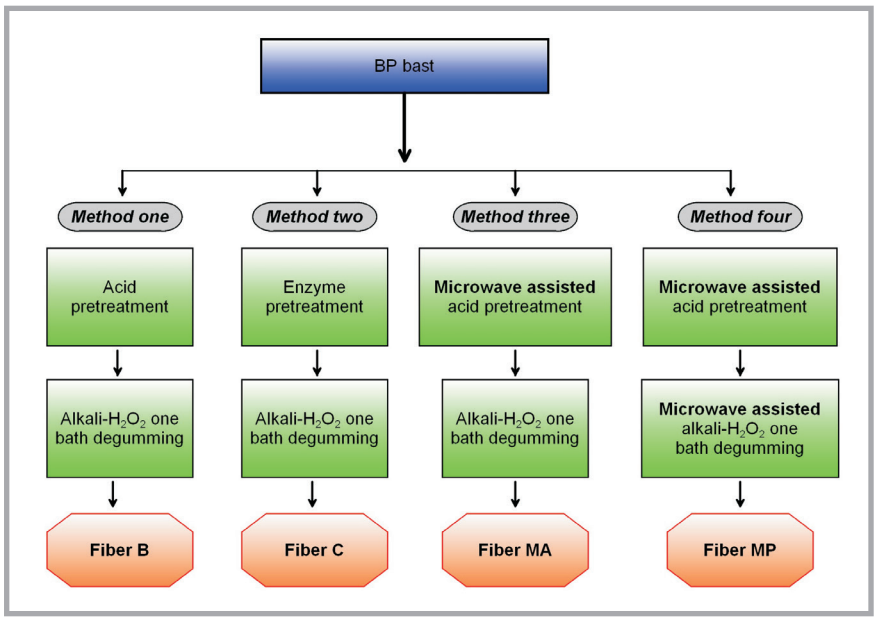

Figure 1. *Schematic of methods used to extract fibres from BP bast.*

where: S_e is the area under the crystalline peaks and S_n the area of amorphous peaks, respectively.

FTIR spectroscopy (FTIR)

The structural properties of BP bast as well as all four fibres were analysed by a Fourier transform infrared (FTIR) spectrometer (NEXUS-670, Nicolet Company, USA). The samples were all milled to powder, mixed with analytical grade KBr and then pressed into a disk for the FTIR measurements.

Scanning electron microscopy (SEM)

Fibre MP, with the best degumming results among the fibres obtained, was observed compared with the BP bast. Fresh BP bast samples were cut into small cubes, fixed with absolute ethyl alcohol, and then dried under critical point conditions in a Polaron Critical Point Dryer operated with liquid CO_2. To obtain a cross-sectional view, Fibre MP was embedded in epoxy resin and then sectioned using a microtome. Then the samples were mounted on conductive adhesive tape and sputter coated with gold palladium before being observed under a SEM-6390LV (Jeol., Japan).

Physical properties of BP fibres

Fibre MP was selected as the best fibre to investigate a series of physical properties i.e. fibre length, fineness, moisture regain and breaking tenacity. Before testing, the samples were conditioned in a standard atmosphere of 20 °C and relative humidity of 65% for at least 12 hours.

Fibre length: 100 bundle fibres (Fibre-MP) were chosen and measured by a comb-type fibre length sorter (Y131, Ningbo Textile Instrument Company, China), and then the values were averaged as the mean fibre length.

Fineness and moisture regain: the fibre fineness was tested by the Middle Weighting Method from ref. [25], and its moisture regain was determined at standard conditions as the average of three measurements.

Tensile properties: tensile tests were performed using an YG001 Miriam Tensiometer (Shanghai, China) with a pretension of 0.2 cN, gauge length of 10 mm and crosshead speed of 10 mm/min. The average tensile strength of 50 single fibres was calculated.

Results and discussion

Composition of BP bast

The composition of BP bast is listed in ***Table 1***, in which data for cotton and flax were from literature [26]. The BP bast had a high cellulose content similar to that of flax but lower than that of cotton,

Table 1. *Composition of BP bast compared with cotton and flax.*

Composition (% on dry weight)	BP bast	Cotton	Flax
Cellulose	56 - 75	85 - 90	60 - 81
Lignin	2 - 4	0.7 - 1.6	2 - 5
Ash	2 - 4	0.8 - 2.0	1.0 - 1.5

Table 2. Quality parameter of BP fibers obtained by different methods.

Quality parameter	Fibre-B	Fibre-C	Fibre-MA	Fibre-MP
Residual gum content, %	7.25 - 7.71	6.95 - 7.34	6.5 - 6.89	5.77 - 6.45
Residual lignin content, %	1.01 - 0.80	0.82 - 0.62	0.75 - 0.60	0.68 - 0.56
Tenacity, cN/dtex	1.24 - 1.66	2.11 - 2.25	2.13 - 2.29	2.19 - 2.39

as shown in ***Table 1***. On the other hand the lignin and ash content of BP bast were both a little bit higher than that of cotton and flax. Cotton was single cell fibre and did not need lignin and other binding materials, whereas the common bast fibres, including flax, needed some gum to hold several single cells together and form bundle fibres [13]. All this evidence indicated that BP bast was a reasonable potential source for extracting natural cellulose fibre.

Comparison between fibre extraction methods

Three characteristic parameters (residual gum content, residual lignin content and tenacity) of the BP fibres from the four extraction methods were tested and listed in ***Table 2***. Notably the rankings of both residual gum and lignin contents for the four fibres were: Fibre B > Fibre C > Fibre MA > Fibre MP, whereas their tenacity ranking was in the reverse sequence: Fibre MP > Fibre MA> Fibre C > Fibre B. Therefore Fibre MP, extracted via *method four* with the microwave-assisted procedure in both the pretreatment and degumming steps, possessed most desirable qualities i.e. low residual gum and residual lignin contents, attractive appearance and excellent mechanical tenacity. Furthermore it required the shortest time in both steps: pretreatment (15 min) and degumming (50 min), compared with the other methods. In other words, the microwave-assisted procedure was a more rapid and effective approach, with high efficiency and low energy consumption in natural cellulose fibre extraction.

XRD analysis

X-ray diffraction spectra of the BP bast and four BP fibre samples are shown in ***Figure 2***. It was seen that all of them showed diffraction peaks of 2θ angles of 14.9, 16.4, 22.7 & 34.5°, assigned to the (101), (101), (002) and (040) planes, indicating that they were of a cellulose I structure [27, 28]. It was thus demonstrated that the degumming process had little influence on the crystalline locations and interplanar crystal spacing in the BP bast.

In addition, crystallinity C_r values were calculated by ***Equation 1*** and the C_r values of Fibre-C, Fibre-B, Fibre-MA and Fibre-MP were 74.82%, 76.71%, 77.07% & 77.25% respectively, being all greater than that of the BP bast (C_r = 71.03%). Furthermore these BP fibres exhibited higher crystallinity than that of cotton (C_r = 60%) and flax fibre (C_r = 65 - 70%) [13, 29]. The high crystallinity means more crystalline regions in the cellulose fibres, and more regular arrangement with fewer pores of the cellulose molecules compared with a fibre of low crystallinity [30]; therefore BP fibres may possess reasonable mechanical properties.

FTIR analysis

FTIR spectra of the BP bast and fibres (Fibre B, Fibre C, Fibre MA, Fibre MP) are shown in ***Figure 3***. The absorption peaks at 3420 cm^{-1} for the OH group and those at 1432, 1164 & 1058 cm^{-1} in the fingerprint area, attributed to the cellulose structure, appeared very similar for all fibre samples and the bast. However, the FTIR spectrum of the bast showed certain differences for the fibres obtained. First the vibration peak at 1735 cm^{-1}, attributed to the CO stretching of methylester and carboxylic acid in the pectin or the acetyl group in hemicelluloses, disappeared after degumming [31]. In other words, the pectin and hemicelluloses were largely removed by each of the degumming processes. Secondly the absorption band at 2900 cm^{-1} , ascribed to CH and CH_2 stretching vibrations in the BP bast, slightly split into two separate peaks corresponding to CH_2 asymmetric and symmetric vibrations at 2918 and 2852 cm^{-1}, indicating that the hydrogen bonds between these two peaks were unlocked during the degumming process. Thirdly the absorption band at 1626 cm^{-1}, attributed to antisymmetric COO- in the bast curve, was drastically weakened in the spectra of the fibres, being an indication that the degumming methods removed the lignin effectively. In other words, after the fibre extraction process, non-cellulose substances, such as hemicelluloses, wax, pectin and lignin, in the bast were all eliminated or reduced, while the cellulose I structure remained nearly unchanged.

Therefore the short time of both pretreatment and degumming microwave assisted *method four* was advantageous and the resulting Fibre-MP possessed high

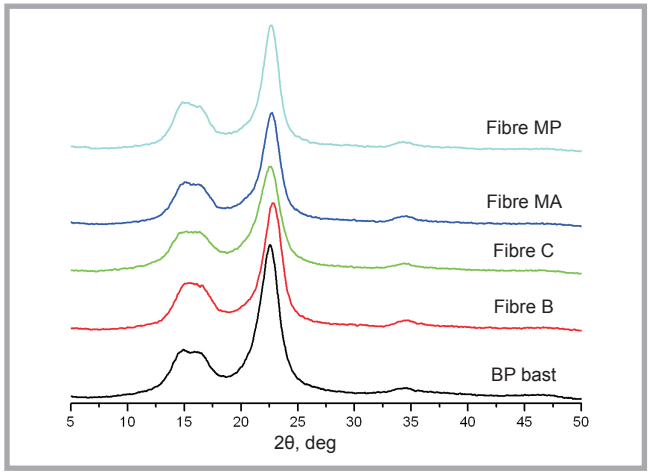

Figure 2. *XRD of BP bast and fibres obtained by the four extraction methods.*

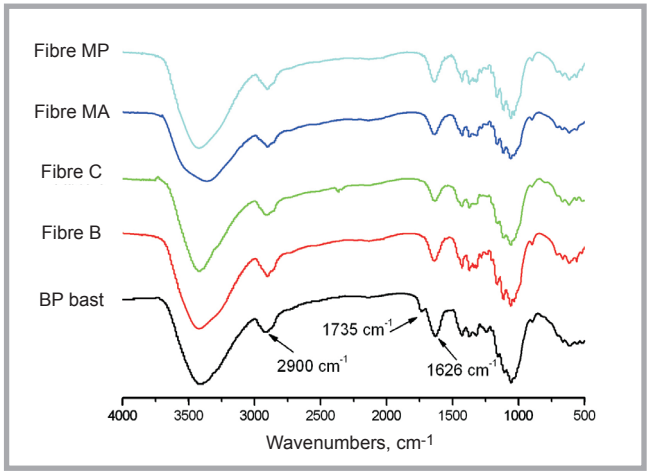

Figure 3. *FTIR spectra of BP bast and fibres from the four extraction methods.*

qualities overall and was thus selected as standard BP fibre and compared with other fibres in this study.

Appearances

Photo images of BP bast (***Figure 4.A***) and Fibre-MP (***Figure 4.B***) from the microwave-assisted extraction method (*method four*) were made for visual inspection and evaluation. In ***Figure 4.A***, after being stretched, the BP bast transformed into a fibrous network with pectin and other binding materials gumming the fibres to make them stable. From our results above the cellulose content of BP bast appears to be very high, and fibres are clearly shown in straw yellow with a certain luster. On the other hand, the Fibre-MP samples in ***Figure 4.B*** revealed high quality fibres sufficiently separated, with a glossy and soft white color, and were readily processible.

Morphological study

Longitudinal and transversal views of the BP bast and Fibre-MP are shown in ***Figure 5.A - 5.D***, respectively. From the longitudinal view of the BP bast (***Figure 5.A***), the inner parallel fibres were substantially gummed and fully covered with a layer of hemicellulose, pectin, lignin and wax substances, which could not only help to bind single fibres together but also form a certain shield against damage. After the degumming process, as shown in ***Figure 5.B***, most of the non-cellulosic substances in Fibre MP were removed efficiently and very little residual pectin was left on the Fibre MP's surface. Moreover the surface of the fibre seemed to be rugged, which might affect its mechanical properties [32]. From its transversal views (***Figure 5.C***), like a "honeycombed" structure, the inner single fibres were fixed and confined in a certain position by the surrounding layers. In addition, the cross-section of single fibres were circular or roughly circular with a central kidney-shaped lumen as in the cotton fibre. From the fibre obtained in ***Figure 5.D*** the shape of single fibres was rarely deformed and preserved similar statues to that of the bast. Therefore the microwave-assisted degumming process could not only degum the binding materials efficiently but also retain the original properties of its internal fibres.

Fibre properties

The fibre length, fineness, moisture regain and tensile properties of Fibre MP were tested and compared with those of both flax and cotton fibres in ***Table 3***; data for cotton and flax were from literature [26].

In terms of fibre size, BP fibre owned a similar length and fineness to those of flax fibre; therefore the desirable standard of spinning could be achieved. Also when tested under the same conditions, its moisture regain were comparable with that of cotton. As for the tensile properties, the breaking strength of Fibre MP was higher than that of cotton but lower than that of flax, as further elaborated in ref. [13]. However, the elongation at

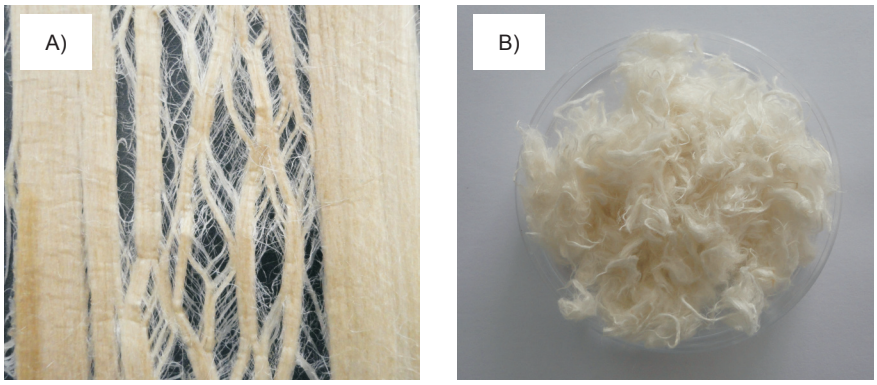

Figure 4. *Photo images: (A) BP bast and (B) BP fiber (Fiber-MP).*

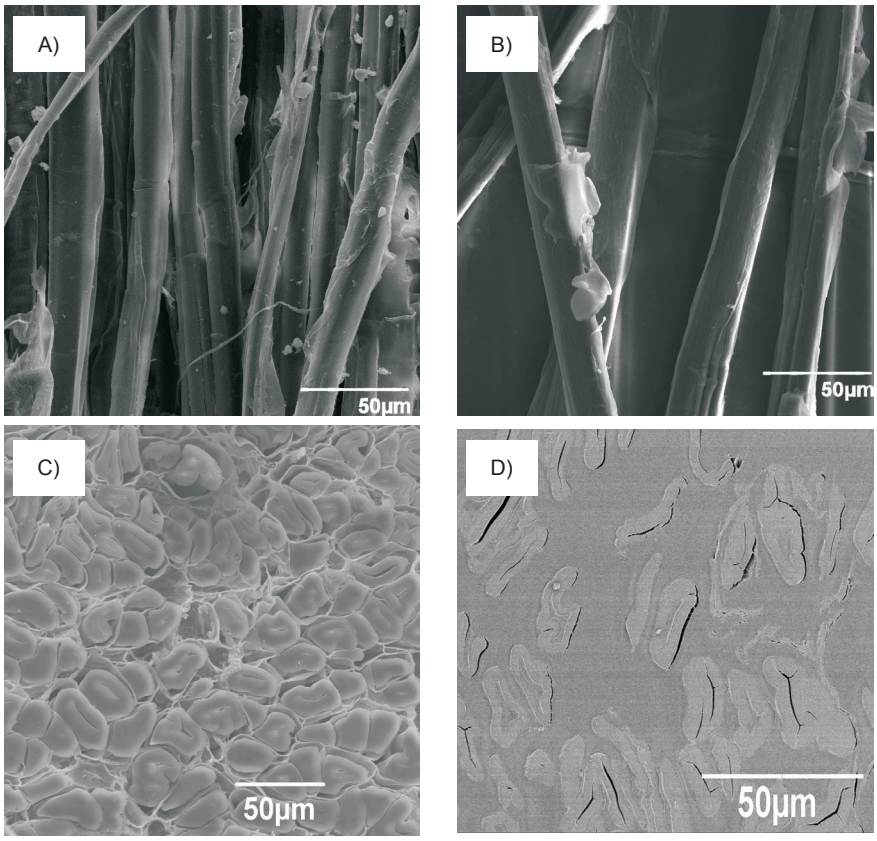

Figure 5. *Longitudinal SEM images: (A) BP bast and (B) BP fibre (Fibre-MP).*

Table 3. *Properties of BP fibres (Fibre-MP) compared with flax and cotton.*

Fiber properties	BP fibre (Fibre-MP)	Flax	Cotton
Strength, cN/dtex	2.19 - 2.39	4.1 - 5.5	1.9 - 3.5
Elongation, %	1.0 - 2.1	1.6 - 3.3	6.0 - 9.0
Length, mm	12.5 - 45.3	17 - 25	15 - 56
Fineness, dtex	1.7 - 3.2	1.7 - 3.3	1.5 - 2.0
Moisture regain, %	6.3 - 8.7	12 - 14	6.9 - 8.2

break of Fibre MP was the lowest of the three, likely caused by its high crystallinity - 77.25%. In terms of deformation stiffness or tensile modulus, Fibre MP was more similar to flax. In conclusion, BP fibres, especially Fibre MP, could be considered as an alternative source of natural cellulose fibres with acceptable properties.

Conclusions

Natural cellulose BP fibres were investigated in this paper for their suitability for textile applications. Fibres were extracted from BP bast by chemical and microwave assisted methods, and then the morphology of the fibres obtained and the BP bast were all characterised. In terms of the extraction method, the microwave-assisted procedure was recommended as a highly efficienct and low cost approach, and the non-cellulose contents in the BP bast were effectively removed after the degumming process. The BP fibres were comparable to cotton and flax in crystallinity and moisture absorption, with reasonably high tenacity and elongation at break. Also, since the BP plants were relatively easy to grow, they could be considered as an alternative source for natural cellulose fibres.

Acknowledgements

This work was supported by the Natural Science Foundation of China via grant no. 51273097 & 51306095 and Taishan scholars construction engineering of Shandong province. We would also like to thank the teachers who provided the test conditions and Dr. Ying Pan for her helpful advice.

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Received 03.07.2013 Reviewed 12.11.2013