A method for separating decorticated bast skin into individual fibres, comprising maintaining decorticated bast skin in a closed container in the presence of at least one enzymatic and/or chemical retting agent for a period sufficient for at least a portion of the fibre bundles to separate into individual fibres.
Figure 1: A sketch of the experimental procedures

1. Decorticated dry skin
   - Soaking in enzyme solution
   - Draining
   - Bagging and sealing
   - Incubating
   - Dunking in hot water
   - Pressure roller squeezing
   - Further dunking and squeezing
   - Oiling and drying
   - Coarse opening
   - Fine opening
   - Sample conditioning
   - Sample testing using OFDA

2. Decorticated green skin
   - Enzyme spraying (optional)
DEGUMMING OF BAST FIBRES

TECHNICAL FIELD

[0001] This invention concerns a method for producing bast fibres, particularly hemp fibres. The invention also extends to bast fibres produced by the method of the present invention and to yams, fabrics and materials manufactured from these fibres. The invention further provides a method for the evaluation of the quality of degummed bast fibres.

BACKGROUND

[0002] Traditionally, plant bast fibres have been degummed by retting. A number of retting techniques have been developed - dew retting, pool retting, stream retting, tank retting, chemical retting, enzymatic retting, and/or a combination of these techniques.

[0003] Dew retting is a process in which the fibres (eg flax) are treated by exposure in the field to the action of dew and sunlight. It is labour intensive, takes several weeks and the retted fibres are usually inconsistent in quality.

[0004] Pool retting is the most rapid of the existing natural retting techniques because of the excess bacteria in stagnant water which speeds up fermentation. This technique takes about a week.

[0005] Stream retting is similar to pool retting, except the bast fibres (eg flax) are placed in a flowing stream that does not have as much of the bacteria that is required for good retting. Clearly, this technique requires that a stream be located nearby.

[0006] Tank retting is considered to be the best and the quickest method of retting, taking only about 3 days. Finer fibre results from this method.

[0007] Chemical retting relies on the action of chemicals such as soda ash, caustic soda and oxalic acid and the action can be violent and uncontrollable, if the retting process is not carried out properly. While chemical retting has been carried out on an industrial scale, further improvement in the retting process is a subject of on-going research.

[0008] Enzymatic retting of bast fibres has been reported before, using various procedures that give fibres of different quality attributes.

[0009] The quest for new and improved ways of degumming is ongoing. We have discovered that degumming of fibre bundles in decorticated bast fibre can be achieved by enzymatic and/or chemical retting of the decorticated fibre in a closed, substantially gas impermeable container, for example, a plastic bag. An advantage of carrying out retting in a closed container is that the fibres are protected from other environmental factors that may lead to unwanted degradation of the fibre. A further advantage in the case of enzymatic retting is that it allows the action of the enzyme to be carried under anaerobic conditions. In addition, this technique has the potential of significantly reducing water consumption compared to traditional retting methods.

SUMMARY OF THE INVENTION

[0010] Accordingly, in a first aspect, the present invention provides a method for separating bast skin into individual fibres, comprising maintaining bast skin in a closed container in the presence of at least one enzymatic and/or chemical retting agent for a period sufficient for at least a portion of the skin to separate into individual fibres.

[0011] Preferably the bast skin is decorticated bast skin.

[0012] Preferably the bast skin is in the form of fibre bundles.

DETAILED DESCRIPTION OF THE INVENTION

[0013] The method of the first aspect may be achieved by taking advantage of the presence of enzyme-producing microorganisms that occur naturally in green bast fibre. In particular, we have found that this natural retting of the decorticated hemp fibre can be achieved by maintaining green decorticated hemp fibre under anaerobic condition for a predetermined period of time.

[0014] Accordingly, in a second aspect, the present invention provides a method for separating bast skin into individual fibres, comprising maintaining fresh decorticated green bast skin in a closed container under anaerobic conditions for a period sufficient for at least a portion of the skin to separate into individual fibres.

[0015] Preferably the bast skin is decorticated bast skin comprising fibre bundles.

[0016] The required anaerobic conditions may be established as a result of production of gas(es) by the freshly decorticated green skin. Alternatively, or in addition, a non-oxygen gas may be introduced into the container (eg nitrogen).

[0017] In the method of the second step, the natural enzymes already present in the green bast fibre may be augmented by one or more other enzymes and/or chemical retting agent. They may be applied to the green bast skins in any suitable manner, for example by soaking the skins in the a composition containing the agent or by spraying a composition onto the skins.

[0018] Alternatively, the method of the first aspect may be carried out by contacting dry decorticated bast skin with one or more enzymatic and/or chemical retting agent(s).

[0019] Accordingly, in a third aspect, the present invention provides a method for separating bast skin into individual fibres, comprising contacting dry bast skin with at least one enzymatic and/or chemical retting agent and incubating the bast skin in a closed container for a period sufficient for at least a portion of the bast skin to separate into individual fibres.

[0020] Preferably, the bast skin is decorticated bast skin comprising fibre bundles.

[0021] Preferably, in the method of the third aspect, the dry bast skin is contacted with the enzyme and/or chemical retting agent by soaking the skin in a solution of the agent(s) and draining the skin before placing it in the container.

[0022] The enzymatic and chemical retting agents may be those known in the art. Examples of suitable enzymatic and chemical retting agents include pectinases eg “Crystalzyme”, “Flaxzym”, Caustic, Soda ash, Sodium Silicate, Oxalic Acid, and EDTA.
The degree of degumming can be altered by changing the duration of degumming and/or the enzyme concentration. The concentration of the enzyme may be in the range of about 0.1 to about 2% (w/v), depending on the required fibre fineness, the enzymes used for retting, and other treatment conditions such as temperature and time.

The residence time of incubation and the enzyme concentration can be selected to achieve the desired quality of the resultant fibres.

The bast skin used in the method of the invention may be obtained by decorticating any bast plant. The plant may be ramie, flax and hemp. Preferably, the bast plant is hemp.

The term "bast skin" as used herein refers to the fibrous skin or rind that has been removed from the bast plant, for example bast skin that is produced when a bast plant is subjected to decorticication.

The bast skin may be that produced by the method of the brief decorticication process. Preferably, the skin is that produced by the decorticication process described in International Patent Application Nos. PCT/AU97/00329 and PCT/ AU99/00858, the entire disclosures of which are incorporated herein by reference.

The substantially gas impervious container may be in the form of a bag or a like into which decorticicated skins may be placed. The bag may be formed from any substantially gas impervious material. The material may be a natural or synthetic plastic. Preferably the plastic is a flexible plastic.

The container may be formed from a gas pervious material that has been treated to make it substantially gas impervious. The container may be flexible, semi-rigid or rigid or a combination thereof. For example the container may be formed from paper, which has been treated to make it substantially gas impervious, e.g. by forming a multilayer material a layer of which is substantially gas impervious.

The method of the present invention may be carried out under controlled temperature conditions. The controlled temperature(s) may be in the range of about 25°C to about 55°C, with the optimum temperature being dependent upon the specific enzymes used for retting.

Alternatively the method may be carried out under uncontrolled temperature conditions. For example, the freshly decorticicated green skins may be placed into plastic bags or the like, the bags sealed and left in the open (e.g. in a field) for a predetermined period.

The predetermined period is selected to obtain the desired amount of fibre separation and the quality of the final fibre. The period is determined by such factors the nature, type and characteristics of the bast fibre being treated, and the temperature(s) at which the method is carried out.

Preferably, fibres treated in accordance with the method of the invention are contacted (e.g. dunked) with hot water (e.g. about 70-90°C), preferably followed by several times (e.g. 2 to 4 times) dunking in either hot or cold water. We have found that this squeezing improves the fibre fineness. The dunking and squeezing simulate the actions on a conventional wool scouring machine.

Accordingly, in a fourth aspect, the present invention provides a method according to any one of the first to third aspects of the invention comprising contacting the treated fibre with aqueous media and compressing the fibre.

The aqueous media may be water.

Preferably, the compressing step involves combined rolling and compressing of the fibres. The compressing step may involve pressure roller compression (squeezing) of the fibres.

The elevated temperature may be in the range of about 70 to 90°C in the first hot water dunking.

In fifth aspect, the present invention provides a fibre product produced by the method of the present invention.

We have found that the degree the level of degumming may be evaluated using a commercial Optical Fibre Diameter Analyser (OFDA). The degree of degumming can be assessed by measuring the width of degummed and opened fibres. This provides a speedy means of evaluating the degree of fibre separating during degumming, even though the fineness of hemp is not usually expressed in terms of fibre diameter or width. It is possible to establish a correlation between this evaluation technique and conventional techniques of measuring the residual gum content and fibre linear density.

Accordingly, in yet a sixth aspect, the present invention provides a method for determining the degree of degumming of a retted bast fibre product, comprising determining fibre width using an optical fibre diameter measuring means.

We have found that the fibres of the present invention may be spun into yarn or the like using conventional cotton spinning apparatus e.g. ring spinning, and rotor spinning. We have also found that by appropriate manipulation of the parameters of the fibre, they may be used in conventional wool spinning operations.

Prior to spinning, the fibre bundles produced by the process of the invention may be subjected to opening (e.g. using an opener) to separate and open up the fibre. The opened fibre can then be subjected to one or more carding operations.

Thus, in a seventh aspect, the present invention provides a method according to any one of the first to third aspects of the invention, comprising the additional steps of subjecting the fibre to at least one opening operation followed by at least one carding operation.

The opening and carding operations are conventional (e.g. those used for cotton) and will be known to those skilled in the art.

The fibres produced by the method of the seventh aspect may range from about 15 microns to about 30 microns. We have found that it is possible to achieve very fine fibre diameters, for example 7 to 13 micron. The invention extends to such fibres.

In an eighth aspect, the present invention provides a spun yarn formed by spinning bast fibres in accordance with the present invention.
Preferably, the bast fibre is hemp fibre. The yarn of the eighth aspect may be a blend of a fibre in accordance with invention with one or more other fibres.

In a ninth aspect, the present invention provides a fabric or material manufactures from fibre in accordance with the present invention.

In order that the present invention may be more readily understood, we provide the following non-limiting embodiments.

**BRIEF DESCRIPTION OF THE DRAWINGS**

**FIG. 1** shows a flow chart of the procedures involved in degumming and evaluating the hemp fibres;

**FIG. 2** is a graph showing the effect of incubation time on fibre width; and

**FIG. 3** is a graph showing the effect of enzyme concentration on fibre width.

**EMBODIMENTS OF THE INVENTION**

Referring to **FIG. 1**, the raw material can be either decorticated dry skin or decorticated green skin. If decorticated dry bast fibres are to be used, the skins are soaked in an enzyme solution for about 5 minutes, this is followed by draining, for about 2 minutes, the solution from the soaked skins to reduce the liquor ratio.

After draining, the skins are sealed in a container (eg plastic bag) and incubated for a pre-determined period of time. Hot water dunking of the bast fibres follows the incubation order to stop further enzyme action. This is then followed by pressure roller squeezing, further dunking and squeezing to improve the fibre cleanliness and fineness.

To evaluate the textile attributes of the treated fibres, the fibres are oiled and dried, opened gradually, conditioned under standard test conditions and tested for fibre fineness (expressed in fibre width here) on an optical fibre diameter analyser (OFDIA).

If decorticated green skins are to be used as the starting material, the skins can be sealed in a container (eg plastic bag) directly, or after being sprayed with an enzyme solution to enhance the retting performance. The remaining steps are the same as described in the previous paragraph for the decorticated dry skins.

**TEST RESULTS**

**Dry skin**

Decorticated and dry skins of the Ukraine variety (Zolotonshka) were mixed and prepared in batches of 800 grams for the evaluation experimentation. The chemicals used were:

- **Enzymes:** 
  - 0.1% (w/v) → 1 g/L (Crystalzyme from Enzyme solutions P.L) Higher enzyme concentrations were also used
  - Oxalic acid: 30 milli mole → 6.3 g/L (Oxalic acid in the form of (COOH)₂H₂O)

- **Wetting agent:** Cibaflow CIR → 0.5 g/L

- **Solution:** 10 Litres

**Green Skin**

Green hemp skins were kept in a sealed plastic bag for about a week to allow natural retting to occur under anaerobic conditions. After fibre opening and sample carding, the width of the degummed fibres was measured on the Optical Fibre Diameter Analyser, and the average width is about 22.5 microns.

**Any discussion of documents, acts, materials, devices, articles or the like which has been included in the present specification is solely for the purpose of providing a context for the present invention. It is not to be taken as an admission that any or all of these matters form part of the prior art base or were common general knowledge in the field relevant to the present invention as it existed before the priority date of each claim of this application.**

**It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as shown in the specific embodiments without departing from the scope or spirit of the invention as broadly described. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.**

1. A method for separating decorticated bast skin into individual fibres, comprising maintaining decorticated bast skin in a closed container in the presence of at least one
enzymatic and/or chemical retting agent for a period sufficient for at least a portion of the skin to separate into individual fibres.

2. A method according to claim 1, wherein the bast skin is decorticated bast skin comprising fibre bundles.

3. A method according to claim 1 or claim 2, wherein the bast skin comprises at least in part fresh green bast skin and the at least one enzymatic retting agent is a naturally occurring enzyme of the green bast skin.

4. A method according to any one of the previous claims claim 1, wherein the bast skin is maintained under anaerobic conditions for a period sufficient for at least a portion of the bast skin to separate into individual fibres.

5. A method according to claim 4, wherein the anaerobic conditions are established at least in part by the production of gas(es) by the fresh green skin.

6. A method according to any one of claim 3 to 5 claim 3, wherein the naturally occurring enzyme is augmented by at least one other enzymatic or chemical retting agent(s).

7. A method according to claim 1 or claim 2, wherein the bast skin is substantially dry bast skin.

8. A method according to claim 7, wherein the bast skin is contacted with at least one enzymatic and/or chemical retting agent(s).

9. A method according to any one of the preceding claims claim 1, wherein the bast skin is contacted with the enzyme and/or chemical retting agent by soaking the skin in a solution of the agent(s) and draining the skin before placing it in the container.

10. A method according to any one of the preceding claims claim 1, wherein the enzymatic or chemical retting agent(s) is/are selected from the group consisting of Crystallzyme, Flaxzym, Caustic, Soda ash and Sodium Silicate, Oxalic Acid, and EDTA.

11. A method according to any one of the preceding claims claim 1, wherein the concentration of the enzyme solution contacted with the bast skin is in the range of about 0.1 to about 2% (w/v).

12. A method according to any one of the preceding claims claim 1, wherein the separation is carried out at a temperature in range of about 25°C to about 55°C.

13. A method according to any one of the preceding claims claim 1, wherein the bast skin is derived from a bast plant selected from the group consisting of ramie, flax and hemp.

14. A method according to any one of the preceding claims claim 1, wherein the bast skin is derived from hemp.

15. A method according to any one of the preceding claims claim 1, wherein the closed container is a substantially gas impervious container.

16. A method according to claim 15, wherein the substantially gas impervious container is in the form of a bag or the like into which decorticated skins may be placed.

17. A method according to claim 16, wherein the bag is formed at least in part from a natural or synthetic plastic.

18. A method according to claim 17, wherein the plastic is a flexible plastic.

19. A method according to any one of the 1 to 17 claim 1, wherein the container is formed from a gas-pervious material which has been treated to make it substantially gas impervious.

20. A method according to any one of the previous claims claim 1 when carried out under controlled temperature conditions.

21. A method according to any one of claim 1 to 18 claim 1, carried out under uncontrolled temperature conditions.

22. A method according to any one of claim 21, wherein the contained bast skins are left in the open for a predetermined period.

23. A method according to any one of the preceding claims claim 1, further comprising contacting the bast skins with hot water.

24. A method according to claim 23, wherein the hot water treated fibres are contacted at least one time with either hot or cold water.

25. A method according to claim 23 or 24, wherein the fibres are subjected to squeezing to remove a least part of liquid therefrom.

26. A method according to any one of claim 1 to 23 claim 1, further including the step of contacting the treated fibre with aqueous media and compressing the fibre.

27. A method according to claim 25, wherein the treated fibres are subjected to at least 2 aqueous media contacting steps.

28. A method according to claims 26 or claim 27, wherein the aqueous media is water.

29. A method according to any one of claims 26 to 28 claim 26, wherein the compressing step involves combined rolling and compressing of the fibres.

30. A method according to claim 29, wherein the compressing step involves pressure roller compression (squeezing) of the fibres.

31. A method according to any one of claims 26 to 30 claim 26 wherein the fibre is contacted with the aqueous media at elevated temperature.

32. A method according to claim 31, wherein the elevated temperature is in the range of about 70 to 90°C.

33. A method according to claim 31 or claim 32, wherein the fibre is contacted with the aqueous media by dunking the fibre in the aqueous media.

34. A fibre produced by a method according to any one of the preceding claims claim 1.

35. A fibre according to claim 34, which has been subjected to an opening operation.

36. A fibre according to any one of claims 34 to 35 claim 34 which has been subjected to a carding operation.

37. Yarn comprising a fibre according to any one of claims 34 to 36 claim 34.

38. A fabric or material comprising a fibre in accordance with any one of claims 34 to 37 claim 34.

39. A method for separating bast skin into individual fibres, comprising maintaining bast skin under anaerobic conditions in the presence of at least one enzymatic and/or chemical retting agent for a period sufficient for at least a portion of the skin to separate into individual fibres.

40. A method for determining the degree of degumming of a retted bast fibre product, comprising determining fibre width of the retted bast fibre using an optical fibre diameter measuring means.

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