United States Patent [19]

Trocino

[11] **3,781,187**

[45] **Dec. 25, 1973**

[54] **METHOD OF SEPARATING BARK COMPONENTS**

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[73] Assignee: **Bohemia Lumber Company, Incorporated,** Eugene, Oreg.

[22] Filed: **Mar. 8, 1972**

[21] Appl. No.: **232,737**

[52] **U.S. Cl.** **162/93,** 162/98, 241/24, 260/412.8

[51] **Int. Cl.** **C11b 11/00,** D01c 1/00

[58] **Field of Search** 162/93, 98, 91, 74, 162/72, 55, 26, 24, 9, 5, 4; 241/14, 24, 28; 260/412.8, 412.5

[56] **References Cited**

UNITED STATES PATENTS

2,662,893	12/1953	Kurth	260/412.8
2,781,336	2/1957	Zenczak	260/412.8
2,926,115	2/1960	Van Beckum	162/93 X
3,616,201	10/1971	Trocino	161/262

Primary Examiner—S. Leon Bashore
Assistant Examiner—Richard V. Fisher
Attorney—Stephen W. Blore et al.

[57] **ABSTRACT**

Douglas Fir bark is extracted with a hot neutral organic solvent to separate the wax from the bark. Thereafter, the residue is heated to desolventize the same and expand the cork fraction substantially in size. Such facilitates efficient subsequent mechanical classification of the cork, bark fiber, and nonfibrous phloem fractions of the bark to a high degree of purity and quality.

10 Claims, 2 Drawing Figures

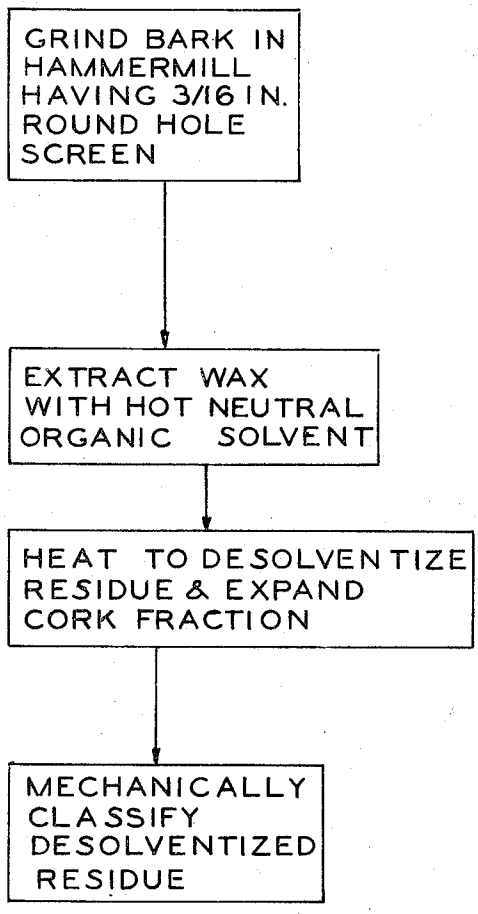


FIG. 1

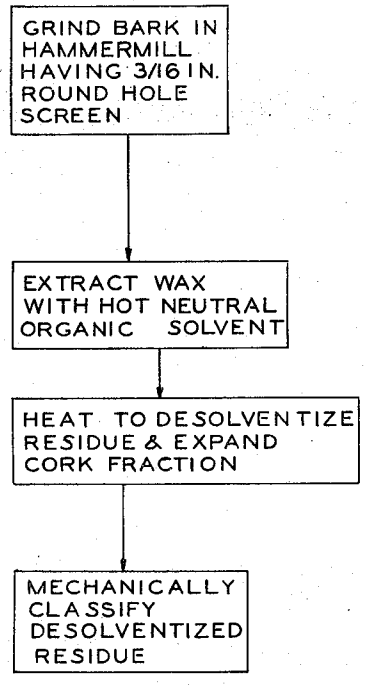
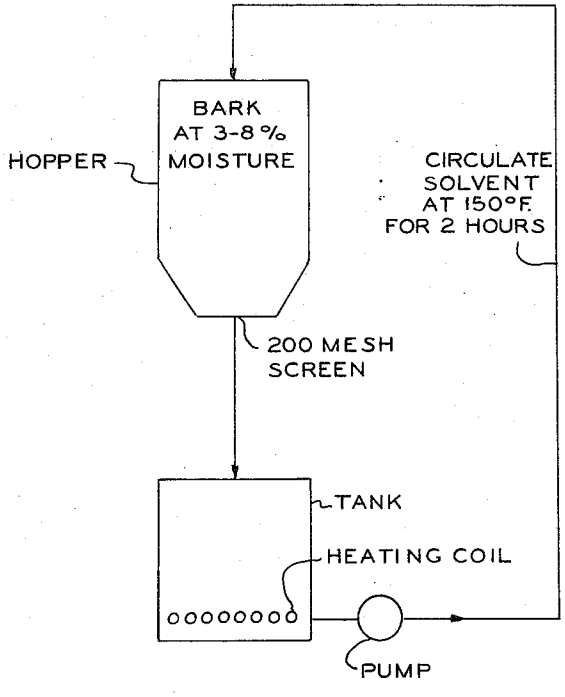

GRIND BARK IN HAMMERMILL HAVING 3/16 IN. ROUND HOLE SCREEN
EXTRACT WAX WITH HOT NEUTRAL ORGANIC SOLVENT
HEAT TO DESOLVENTIZE RESIDUE & EXPAND CORK FRACTION
MECHANICALLY CLASSIFY DESOLVENTIZED RESIDUE

FIG. 2


BARK AT 3-8% MOISTURE
HOPPER
CIRCULATE SOLVENT AT 150°F. FOR 2 HOURS
200 MESH SCREEN
TANK
HEATING COIL
PUMP

METHOD OF SEPARATING BARK COMPONENTS

CROSS REFERENCE TO RELATED APPLICATION

Pending application, Method of Extracting Wax from Bark, Ser. No. 232,944, filed Mar. observation 1972, concurrently herewith, by Frank S. Trocino, applicant herein.

BACKGROUND OF THE INVENTION

This invention relates to a method of separating the various components of the bark of trees and, more particularly, to a method of separating strata deteriorates components in commercial volumes of high purity and quality.

The bark of trees and particularly, the bark of the Douglas fir consists principally of cork, parenchyma tissue and sclerenchyma or bast fibers. The cork is so called because it is a spongy, resilient, low density material resembling ground cork from the Mediterranean oak in appearance and in many of its properties. The cork contents of Douglas fir bark varies from 32 to 50 percent depending on the location and age of the tree.

The parenchyma tissue is a friable phloem consisting principally of sieve tubes. Such tissue readily disintegrates to a fine powder. The sclerenchyma or bast fibers comprise a tough fibrous portion of the phloem occuring in tightly cemented bundles which, upon grinding or subjection to mild attrition, are reduced to the individual or ultimate fibers. Such individual fibers, viewed under a microscope, are red-brown in color, short and sharply pointed and appear to have a spindle shape with a length approximately ten times their diameter. Variations in particle sizes of individual fibers have been noted from 0.016 to 0.090 inch in length and from 0.002 to 0.008 inch in thickness at their midsection with the bulk of the particles having an average length of about 0.054 inch and an average thickness of about 0.005 inch. The bast fiber content of Douglas fir varies between 35 and 48 percent by weight on an air dried basis. The individual fibers are very resistant to comminution and tend to maintain their identity even when subjected to severe pulverizing action by which other portions of the phloem are reduced to powder.

The above three constituents of the bark are intimately commingled with each other and are present in the varying portions noted depending on such factors as the age of the chemical climate conditions and soil conditions. Bast or bark fibers are particularly useful in the formulation of thermosetting phenol-aldehyde holding compositions, the fibers being incorporated in substantial proportions to serve as an extender materially to reduce the cost of the compositions. The fibers also impart high flexural and tensile strength to the molded product and give increased impact resistance thereto.

Past attempts to separate the bark components have made use of various comminutive devices to reduce the particle size of the bark, followed by various methods for separating or classifying the solid material, such as milling, screening or shearing. In a typical mechanical procedure, raw bark is ground or milled at a controlled moisture content to a suitable particle size. It is then fractionated mechanically by screening, winnowing or other procedures which separate it into fractions comprising predominately bast or bark fiber, parenchyma and cork. These procedures, however, while useful for some purposes, have been too complex for widespread

commercial acceptance. In addition, a clear separation of the three components has never been achieved. Where mechanical fractionation has been carried out to the degree necessary to secure relatively pure fractions of the individual bark components, the yield has been so small that it has been commercially impractical.

Prior art chemical procedures for treating bark have relied upon the differential solubility of the bark components in organic and inorganic solvents. In these procedures, the extracted residue is a mixture of the our components of the bark. In Gregory et al. U.S. Pat. No. 3,245,869, aqueous alkali is used to separate the bast fibers. In this process, a substantial proportion of the bark is dissolved away from the fiber, leaving that component as a solid residue. Nonfibrous parenchyma and cork components not removed by the chemical treatment are rendered friable so that they may subsequently be removed by screening and milling. The process, however, does not achieve a complete separation of the components, some of the parenchyma tissue remaining encrusted upon the fibers and some wood and cork fractions remaining intermixed therewith. The characteristic red-brown color of the clean, pure bast fibers is not achieved.

In my U.S. Pat. No. 3,616,201, I disclosed that if the wax content of Douglas fir bark is reduced to below about 3 percent by weight by solvent extraction, the residue, if dried and ground, makes a very satisfactory extender for thermosetting adhesives used in the formulation of bonded cellulosic products. I have since discovered that extraction of the bark using a neutral organic solvent permits the bark components themselves to be separated with an efficiency not heretofore possible.

Accordingly, it is the primary object of the present invention to provide a method of separating the various bark components, including cork, bast or bark fiber and nonfibrous phloem, in commercial volumes of high purity and quality.

It is a further object of the present invention to provide such a method wherein there is no chemical alteration and no portions of the bark are dissolved, thereby to utilize 100 percent of the bark.

It is a still further object of the present invention to provide a method of separating the cork fraction wherein the quality of the cork obtained is comparable to that of Mediterranean oak.

A still further object of the present invention is to provide a method of separating bark components wherein the bast or bark fibers are obtained free of any encrustation by parenchyma tissue.

It is a still further object of the present invention to provide a method of separating bark components that requires an initial hammermill grinding, but no subsequent grinding, milling or shearing to facilitate ultimate mechanical classification.

SUMMARY OF THE INVENTION

My method of separating the cork, bark fiber and nonfibrous phloem fractions of the bark of trees comprises reducing the bark to the form of small pieces and extracting the mixture so obtained with a neutral organic solvent, thereby separating from the bark wax which is soluble in such solvent. The wax solution is separated from the residue, which is thereafter heated to desolventize the same and substantially expand the

pieces of cork therein. Mechanical classifications of such heat-desolventized residue can then be employed selectively to segregate the mixture into cork, bast fiber and comminuted nonfibrous phloem with no subsequent grinding, milling or shearing required. The components are separated to a purity and quality not heretofore obtainable in commerical volumes.

The solvent extraction of the bark and the subsequent desolventizing of the residue expands the pieces of cork to facilitate this separation. A quality of cork is achieved comparable to that of Mediterranean oak. Good yields of the bast or bark fibers are also achieved, and such are of high purity and quality.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 illustrates a flow diagram of the process in accordance with the teachings of the invention.

FIG. 2 illustrates schematically the solvent extracting step.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The first step in my process is to grind the bark in a hammermill having a screen with holes about three-sixteenths inch in diameter. The mixture thereby achieved is then dried to between about 3 and 8 percent moisture by weight, although this step is not mandatory. Achieving this degree of dehydration is preferable in that it facilitates ultimate recovery of the solvent.

The mixture is then extracted with a neutral organic solvents examples of suitable ones being hexane, bezene, the chlorinated hydrocarbons, napthalenes, toluene and the like. I have found a combination of aliphatic and aromatic hydrocarbons particularly suitable for this step, a mixture of this constituency have a preferred concentration of aromatic hydrocarbons between about 10 and 50 percent. An example of such a solvent which I have found suitable is that sold by Standard Oil Company of California as SOCAL No. 226. This solvent has the following characteristics:

Molecular weight:	98
Vapor density:	3.3
Boiling Point:	50% at 208° F. 90% at 213° F.
Dry Point:	225° F.
Specific Gravity	6.29 lbs./gal.
Flash cup:	closed 24° C.
Aromatics:	11%

The solvent is heated to a temperature of about 150° F. and is percolated through the mixture of ground bark for about 2 hours, the solvent being introduced to the bark in a hopper having a 200 mesh screen at its bottom and being permitted to flow therethrough by gravity. Solvent passing through the mixture is reheated to 150° F. by a heating coil in a holding tank and thereafter recirculated for the desired time. See FIG. 2. The solvent is circulated through the bark mixture so as to remove between about 3 and 6 percent of the wax based on the dry weight of the bark. Whole Douglas fir bark has between about 5 and 10 percent wax by weight on a dry basis, and I have found that removing approximately 60 percent of the total wax satisfactorily prepares the bark for classification as described hereinafter. A longer period of extraction would dissolve more of the wax, but I have found this unnecessary.

The surfaces of the bast fibers in the bark mixture are cleaned by the action of the percolating solvent during the extraction, the solvent dissolving the adhering and encrusting material. This facilitates ultimate classification of the components.

After the two hour extracting period, the bark is drained of solvent, placed in a dryer and heated to about 500° F. for 2 to 4 minutes to dry off the solvent. The solvent can then be condensed, recovered and reused. Heating the bast fibers to 500° F. in the desolventizing step conditions them to that temperature, thereby to make the fibers suitable for end uses requiring an ability to withstand this temperature.

The desolventizing of the bark residue after extraction of the wax expands the pieces of cork therein substantially in size. The process of desolventizing expands the individual cork cells, puffing them up visibly in size as the heat evaporates the solvent. The wax can subsequently be recovered from the solvent, thus making possible 100 percent utilization of the bark.

After desolventizing the bark, the wax extracted residue is then mechanically classified using standard classification equipment, but without the necessity of any further grinding, milling or shearing to achieve ultimate separation of the cork, bast fiber and nonfibrous phloem fractions. The corky fraction of the bark, having expanded substantially in size during the desolventizing of the wax extracted residue, is more resilient than its original counterpart, and this makes classification much easier. I have found the cork obtained to be of a quality competitive with Mediterranean oak. It is of better quality than that obtainable by all prior mechanical segregating processes and this, I believe, is due to the expanding thereof which takes place in the desolventizing step and which increases the resilience of the cork fraction. The bast fibers achieved in the process are cleaner and of a better quality than those achieved in the aqueous alkali process above mentioned. Furthermore, complete recovery of the solvent and subsequent recovery of the wax therefrom insures that the instant process provides no ultimate disposal problem as in the aqueous alkali process, wherein the caustic solution with the dissolved constituents therein must ultimately be burned.

Example 1

Eighty-three pounds of Douglas fir bark were ground in a hammermill having a 3/16 inch round hole screen and subsequently dried to 16.5 percent moisture. The mixture was extracted for two hours with SOCAL No. 226 solvent at 150° F., 2 gallons per minute of the solvent being pumped through a hopper containing the bark mixture as illustrated in FIG. 2. The extracting yielded 5.1 percent wax by weight from the bark on a dry basis. The bark residue was thereafter desolventized by being placed in a 500° F. dryer for four minutes. Classification in a Prater classifier realized the following yields of bark components as a percentage of the weight of the desolventized bark:

Cork — 41%
Bast fibers — 29.5%
Powder — 29.5%

Example 2

Seventy-eight pounds of Douglas fir bark were ground in a hammermill as in Example 1 and dried to 6.4 percent moisture. The mixture was extracted with SOCAL No. 226 solvent at a temperature of between 150° and 157° F. for 2 hours at a flow rate of 2 gallons per minute. The yield of wax was 3.6 percent by weight

on a dry basis. The extracted residue was desolventized in a dryer as in Example 1 for 2 to 3 minutes. Classification in a Prater classifier yielded bark components in the following amounts as a percentage of weight of the desolventized bark:

Cork 35%
Bast fibers 47%
Powder 18%

The two examples illustrate that the amount of cork recovered varies with the yield of wax which is to be expected since most of the wax is in the cork in Douglas fir.

I claim:

1. A method of treating bark containing cork, bark fiber and nonfibrous phloem fractions, comprising

reducing the bark to the form of small pieces comprising a mixture of pieces of cork, bark fiber and nonfibrous phloem;

extracting said mixture of pieces with a neutral organic solvent, thereby separating from said bark wax which is soluble in said solvent and leaving a residue of wax-extracted bark;

then heating said residue to desolventize the same and expand said pieces of cork substantially in size, said bark fiber and nonfibrous phloem fractions remaining substantially unchanged in size; and

mechanically classifying said heat-desolventized residue selectively to segregate said pieces into cork, bark fiber and finely comminuted nonfibrous phloem.

2. A method as in claim **1** in which said bark is ground in a hammermill to pass a 3/16 inch round hole screen.

3. A method as in claim **1** in which said bark is Douglas fir bark.

4. A method as in claim **1** in which said solvent comprises a mixture of aliphatic and aromatic hydrocarbons.

5. A method as in claim **4** in which the concentration of aromatic hydrocarbons in said solvent is between about 10 and 50 percent.

6. A method as in claim **1** in which said solvent is percolated through said bark pieces.

7. A method as in claim **6** in which said solvent is percolated through said bark pieces at a temperature of about 150° F.

8. A method as in claim **7** in which said solvent is percolated through said bark pieces for about 2 hours.

9. A method as in claim **1** in which said bark is dried to a moisture content between about 3 and 8 percent by weight prior to extracting the same.

10. A method as in claim **1** in which said bark is extracted to remove between about 3 and 6 percent of wax based on the dry weight of the bark.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,781,187 Dated December 25, 1973

Inventor(s) FRANK S. TROCINO

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 5, "Mar. observation" should be --March 8,--;

line 12, "strata deteriorates" should be --such--;

line 47, "of the chemical climate" should be

-- of the tree, climatic --

line 51, "holding" should be --molding--;

Column 2, line 11, "our" should be --original--;

Column 3, line 1, "classifications" should be --classification--;

line 31, "solvents" should be -- solvent, --.

line 31, "be-" should be --ben- --; and

line 35, "have" should be --having--.

Signed and sealed this 21st day of May 1974.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents

FORM PO-1050 (10-69)

USCOMM-DC 60376-P69
* U.S. GOVERNMENT PRINTING OFFICE : 1969 O—366-334