March 16, 1937. F. R. CHESLEY, JR. 2,073,682

PROCESS OF TREATING VEGETABLE FIBROUS MATERIAL FOR THE PRODUCTION OF CELLULOSE FIBER

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Fig. 1.

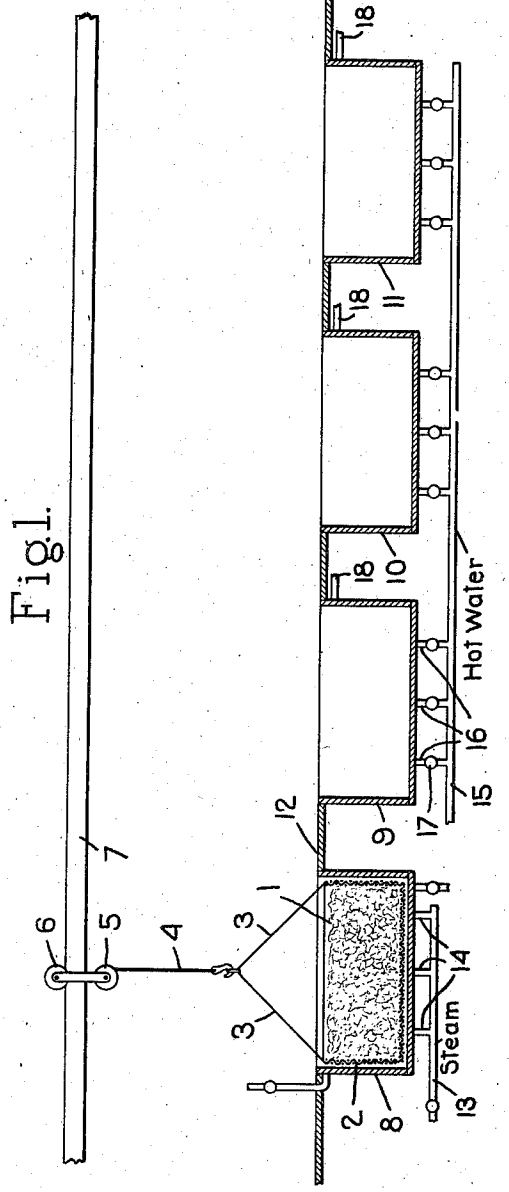
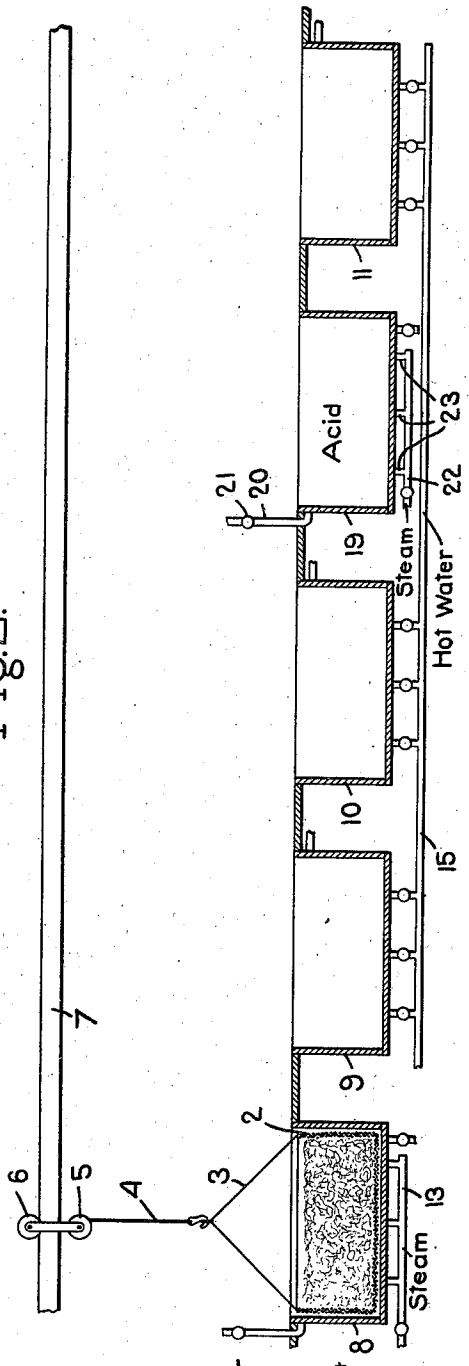


Fig. 2.



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PROCESSES OF TREATING VEGETABLE FIBROUS MATERIAL FOR THE PRODUCTION OF CELLULOSE FIBER

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11 Claims. (Cl. 92—17)

This invention relates to improvements in the production of cellulose materials, and the object thereof is broadly to provide a process for treating vegetable fibrous material, including woods especially coniferous woods, in such a manner as to remove the resins and gums from the fibrous material without affecting the cellulose constituents thereof, whereby the cellulose, whether in the form of fibers or otherwise, may be treated by usual methods and/or machines to produce economically and profitably materials used in the various industrial arts in which cellulose is employed.

The process herein described is adapted to the treatment of vegetable fibrous materials, including plants, fiber-producing shrubs, and the like, for the production of cellulose fibers used in the textile arts, and may also be employed in the production of cellulose pulp adapted for treatment to produce other products, such as paper, or when nitrated to produce the bases for explosive compositions.

The invention includes the process of decorticating and degumming fiber-bearing plants, such as ramie, flax, jute, sisal hemp, etc., by means of which the resins and gums are removed, the bark released in such a manner that it can be easily separated and the fibers individualized without destroying or deteriorating the cellulose constituents of the material.

By the present process relatively inexpensive materials and apparatus may be employed and the time required for treatment greatly shortened as compared with other processes which are commercially used.

In view of the fact that it has heretofore been difficult to produce ramie fibers which can be used in the textile industry and which when used requires special machines, the present process will be described specifically in relation to decorticating and degumming of ramie and the like, and so individualizing the fibers thereof in a relatively inexpensive and efficient manner as to prepare the fibers for direct treatment by usual machines employed in the treatment of cotton.

In the usual treatment of ramie, the ramie stalks are decorticated by being fed into a machine having rapidly revolving blades which break the stalks into small pieces and leave the bark and fiber in long ribbons, part of the gum being squeezed out by the beaters. The ribbons are then subjected to the action of caustic soda solution in a heated closed vessel or boiling kier under steam pressure. After having been boiled a sufficient time to remove the gum, the material is lifted out, the alkali neutralized and the fiber thoroughly washed to remove all traces of the chemicals, and the fiber finally dried. Thereafter, the fiber is softened by passing it through a breaker, usually in the form of a machine having complementary fluted rollers. The fiber thus treated is subjected to the operations of dressing and roving and when used for textiles is spun by a wet spinning process.

Ramie fibers when produced by usual processes have serrated or toothed edges which present a hairy appearance and when the fibers are rubbed together in subsequent treatments, such as spinning, they become torn and also are easily broken when bent.

It is believed that the serrated or hairy characteristics of the fibers are caused mainly by the extraction of the waxes which are contained in the natural fibers, and one of the principal objects of the invention is to provide a process of treatment of the fibers of ramie, and other fiber-bearing plants of this character, in such a manner that the waxes will not be removed from the fibers, thereby not only preserving their lustrous appearance, but also preserving the natural strength of the fibers. This is accomplished in the present invention by subjecting the fibrous material, such as the stalks of ramie, hemp, flax, or other plants, preferably cut into desirable staple lengths, to the action of an aqueous, weak alkaline, solution in the presence of aluminum, capable of dissolving the gums and resins contained in the fibrous material, but which is incapable of dissolving the waxes contained in the fibers, the solution being of insufficient causticity to affect the cellulose of the fibers. Inasmuch as the individual fibers of such plants are in the form of a single cell comprising mainly cellulose, treatment by the present process results in the production of fibers in which the waxes are retained, the strength of the fibers preserved, and their lustrous appearance maintained.

Usually the fibers of ramie, flax, hemp, and the like, are bleached subsequently to the decorticating and degumming process. By the present invention decortication, degumming, and bleaching may be performed concurrently by the inclusion of a bleaching chemical in the solution which effects the decortication and degumming of the fibrous material, a suitable neutralizing agent being employed to remove the excess of bleaching material. Alternatively, the bleaching of the fibers may be made subsequent to the decortication and degumming as will hereinafter more fully appear.

Any suitable apparatus may be employed for performing the present process. An illustrative apparatus is shown in the accompanying drawing, in which,

Fig. 1 is a view, mainly in vertical section, of a series of kiers, and a suitable basket, to contain the material, supported upon a trolley and adapted to be transferred from one kier to another; and,

Fig. 2 is a similar view of suitable apparatus

adapted to be employed when the fibers are bleached subsequently to decortication and degumming.

In the performance of the process by the apparatus illustrated herein, the fibrous stalks 1 of ramie, hemp, flax, or the like desirably are first cut to desirable staple lengths, and placed in a basket 2 having a reticulated or perforated bottom and sides and which is suspended by suitable supports 3 from a cable 4 which passes over a pulley 5 depending from a trolley 6 which is mounted to travel upon a track 7 extending longitudinally over a series of receptacles or kiers. A series of such kiers 8, 9, 10 and 11, are employed, all of which may be of substantially identical construction and located below the plane of the floor 12, the upper edges of the kiers desirably being flush with the floor.

An aqueous alkaline solution containing aluminum, either in the form of finely divided metal or in the form of a sulphate, is introduced into the first of the kiers 8. I have found by experiment that a weak alkaline solution containing the following ingredients in substantially the proportions stated gives most excellent results,

Fresh water	gallons	1000
Tribasic sodium phosphate	pounds	719
Soda ash	do	62.5
Aluminum sulphate	do	7.8

I have also found that finely divided metallic aluminum may be substituted in the solution for the aluminum sulphate in the proportion of approximately 23.3 ounces of aluminum per one thousand gallons of water.

The chemicals may be added to the water when cold and before the basket, containing the material to be treated, is introduced, but better results are obtained when the material to be decorticated and degummed is placed in the water and the temperature then raised to the boiling point, which is between two hundred degrees F. and two hundred twenty-five degrees F., and the chemicals then added.

Any suitable means may be provided for maintaining the solution at a boiling temperature. Desirably steam is forced through a pipe 13 and suitable branches 14 therefrom leading through the bottom of the kier into the material at a sufficient rate to maintain the temperature of the solution at a boiling point, and also to maintain a mild agitation of the material by the ebullition of the steam passing through the solution.

The boiling is continued for from approximately fifteen to approximately one hundred thirty-five minutes depending upon the following conditions:—

1. If the ramie were in its natural form, i. e., about four-foot lengths, the time allowed is about one hundred thirty-five minutes, after the solution has come to a boil, and if cut to staple lengths less than nine inches the time allowed is approximately one hundred five minutes.

2. Flax when boiled in said solution for forty-five minutes is found to be sufficiently decorticated for use.

3. Other fibrous materials may be allowed such suitable time within the aforementioned range of from fifteen to one hundred thirty-five minutes, according to their condition, and quality, to assure complete degumming.

The solution and material may be observed from time to time and when the decortication and degumming of the process is observed to be complete, bark and other materials which float upon the surface may be skimmed off.

The basket may then be raised from the kier 8 and transferred to and deposited in the kier 9 and subjected to washing. The washing in this kier desirably may be accomplished by forcing hot water through a pipe 15 and branches 16, having suitable valves 17 therein, to fill the kier 9 and continuously to overflow therefrom through an outlet pipe 18. Desirably the basket containing the material is transferred from the kier 10 successively to similar kiers or washing tanks 10 and 11 where fresh hot water is introduced from the pipe 15 in like manner.

The action of the hot weak alkaline solution in the kier 8 serves to dissolve the resins and gums of the fibrous material, thereby detaching the fibers from the box and also liberating the fibers from each other, so that they are individualized. It is believed that the presence of aluminum or an aluminum salt facilitates and speeds up the action of the alkalis in precipitating the tannic constituents of the fibrous materials and also facilitates the action of the alkali upon the soluble gums and other constituents of the fibrous material, and also in removing certain of the pigments as the fibers when thoroughly washed are substantially colorless.

If desired the fibers may be bleached by the present process during the decortication and degumming thereof by introducing into the solution contained in the kier 8 a suitable bleaching material.

In the treatment of ramie sodium hypochlorite may be employed as a bleaching agent in the proportion of approximately two ounces of sodium hypochlorite to one gallon of the solution. In such instance the fibrous material must be washed by a bath method, there being introduced between the second and third baths a boiling solution made by adding one ounce of concentrated sulphuric acid to each gallon of water in the bath.

In thus performing the bleaching concurrently with decortication and degumming, the apparatus shown in Fig. 2 is employed in which the basket 2 is carried in like manner by supports 3 upon cables 4 which pass over a pulley 5 depending from a trolley 6 upon the track 7, the steam being supplied to the kier 8 through the pipe 13 and branches 14 in the manner above described. In this apparatus the kiers 9, 10 and 11 are each supplied with hot water through a pipe 16 as aforesaid, but an additional kier 19 is interposed between the washing kiers 10 and 11 to contain the acid solution for neutralizing the bleaching agent. The proper acid solution may be introduced into the kier 19 through a pipe 20 having a valve 21 therein and the acid solution heated by steam introduced through a pipe 22 having branches 23 leading through the bottom of the tank.

When the decorticated and degummed fibers contained in the basket 2 have been suitably washed in the kiers 9 and 10 they are transferred in the basket to the kier 19 where the material is permitted to stay in the acid for a period of five minutes or more, after which the basket is transferred to the kier 11 where the bleached material is thoroughly washed, after which it desirably is thoroughly dried by a forced draft of heated air. The bleached material is then ready for mechanical processing.

The material when processed in the manner above described may be treated in usual cotton

mixing and carding machines and spun, woven, or otherwise treated, either alone or mixed with cotton, silk, or other fiber, as in usual processes.

It is important to note that in the treatment of ramie in the manner above described the fibers differ materially from ramie fibers treated by usual processes heretofore employed in that the degumming is so complete that usual carding machinery may be employed without detriment to the machines. This is particularly true of carding in which it has heretofore been deemed impossible to card ramie because the amount of gums or resins, which are retained by the fibers, will in a short time accumulate upon the card clothing and clog the machinery not only injuring it, but in many instances producing such massing of the fiber as to injure the machine. The fibers when treated by the process herein described are substantially free from serrations or hairs. Their flexibility is retained and breakage by abrupt bending is substantially eliminated. The fibers have greater tensile strength and can be spun into threads which present a lustrous appearance and when woven produces a very flexible fabric of unusual strength having the appearance of high grade linen. The fibers can be readily and uniformly dyed either before or after spinning.

A further important feature of the invention comprises the manufacture of absorbent cellulose materials from ramie, and other fibrous materials, of the character above described, by the elimination of such waxes and oils as are retained in the fibers. This may be accomplished by treating the fibers produced by the process aforesaid with a suitable solvent, such as naphtha, for the waxes and oils, retained in the fibers. It is found that when the ramie fibers are thus treated they have far greater absorbent qualities than cotton, or other usual fibrous material employed as absorbents. The product thus obtained by the present process is especially suitable for use in surgical bandages, wicks, and other purposes in which the highly absorbent characteristics of the fibrous material are advantageous.

Hemp, flax, jute, sisal, and other fiber-bearing plants may also be treated by the same process and with the same chemicals, differing only from the process of treating ramie in the period of immersion in the heated solution. The preparation of the fibers from such plants by the present process eliminates numerous treatments heretofore believed to be necessary in the preparation of the fibers for spinning, etc.

Another important feature of the present invention is the application of the present process to the treatment of wood, especially coniferous wood, in the production of cellulose pulp for use in paper making, or other industrial arts, including cellulose bases for explosive compositions.

In the production of paper pulp, wood chips have heretofore been usually treated by an acid or a caustic alkaline process for the purpose of reducing the chips to a cellulose composition, the chips being then broken up, either by crushing, beating, or by projecting the same forcibly against a steel plate. The treatment of the chips in such acid or caustic solution requires twenty-four hours or more and in such treatment the chips are boiled in the solution under high pressure involving large expense and requiring a very considerable amount of time. By the present process the wood chips are treated in the heated aqueous weak alkaline solution of the character above described at ordinary pressures, the solution being maintained at substantially the boiling point by the introduction of steam under pressure to maintain an ebulition in the kier containing the material. The time required to reduce the chips to the cellulose composition may vary from not less than three hours to not more than seventeen hours, according to the character of the wood being treated. The chips may then be reduced to pulp form by similar mechanical methods. The product will then be in the form of a pulp which may be used in the manufacture of paper. If the paper pulp thus produced is then thoroughly washed and again treated with the same solution, under the same conditions, for approximately two hours, further reduction will be accomplished which will produce substantially pure "alpha" pulp adapted for use in all arts using cellulose as a base.

While it has been found by experiment in practical commercial use that the chemicals and proportions thereof herein described in respect to the treatment of ramie may be employed, it will be understood that the proportions may be varied, and that other equivalent chemicals, or those having like properties and capable of producing substantially the same results, may be employed within the meaning and scope of the following claims. It will also be understood that other types of apparatus may be used in the performance of the process.

Having thus described the invention, what is claimed as new, and desired to be secured by Letters Patent, is:

1. The process of treating vegetable fibrous material which comprises subjecting the fibrous material to immersion for a suitable period of time in an aqueous weak alkaline solution, comprising a major portion of tribasic sodium phosphate and a minor portion of an aluminum-containing substance, capable of dissolving gums and resins within the fibrous material and of precipitating tannic substances contained in said fibrous material without affecting the cellulose of the fibers, and thereafter repeatedly washing the fibers to remove the chemicals and the foreign material which have been released by the action of the solution.

2. The process of treating vegetable fibrous material which comprises subjecting the fibrous material to immersion for a suitable period of time in a heated aqueous weak alkaline solution comprising a major portion of tribasic sodium phosphate and a minor portion of aluminum sulphate, capable of dissolving the gums and resins contained in said fibrous material, but incapable of dissolving the waxes contained in said fibers, and of insufficient causticity to affect the cellulose of the fibers, introducing steam into the solution to maintain the heated solution at substantially its boiling point, and to maintain ebulition of said liquid effective to facilitate the solvent action of said solution.

3. The process of decorticating and degumming fibrous plants which comprises immersing the fiber-bearing portions thereof for a suitable time in a heated aqueous weak alkaline solution, comprising a major portion of tribasic sodium phosphate and a minor portion of soda ash in the presence of an aluminum-containing substance, capable of dissolving the gums and resins contained in the fibrous material, but of insufficient causticity to affect the cellulose of the fibers.

4. The process of decorticating fibrous plants

and individualizing the fibers thereof which comprises steeping or boiling the fiber-bearing portions thereof, cut to desirable staple lengths, for a suitable period of time, in an aqueous weak alkaline solution, comprising a major portion of tribasic sodium phosphate and a minor portion of soda ash in the presence of an aluminum salt, capable of dissolving the gums and resins contained in the fibrous material, but of insufficient causticity to affect the cellulose of the fibers, and maintaining the solution in a state of mild agitation during said period.

5. The process of decorticating fibrous plants and individualizing the fibers thereof which comprises immersing the fiber-bearing portions of the plants, cut to desirable staple lengths, for a suitable period of time, in a heated aqueous, weak alkaline, solution comprising such major portions of tribasic sodium phosphate and minor portions of soda ash and aluminum sulphate, as will act to dissolve the gums and resins contained in the fibrous material, to precipitate tannic substances and to bleach the fibers without removing the waxes or oleaginous constituents of the fibers, but of insufficient causticity to affect the cellulose of the fibers, introducing steam into the solution in such manner as to maintain the temperature thereof at substantially the boiling point and by ebulition to cause agitation of the solution, thereafter removing the foreign material that has been released by the action of the solution, and thereafter washing and drying the fibers.

6. The process of decorticating and degumming fibrous plants which comprises immersing the fiber-bearing portions thereof, cut to desirable staple lengths, for a predetermined period of time, in a heated aqueous, weak alkaline, solution comprising the following chemicals in approximately the stated proportions:

Fresh water	gallons	1000
Tribasic sodium phosphate	pounds	719
Soda ash	do	62.5
Aluminum sulphate	do	7.8

maintaining the solution for said period of time at a temperature of approximately two hundred degrees F. to two hundred twenty-five degrees F., thereafter removing the material from said solution and washing the same to remove the chemicals and the foreign materials which have been released by the action of the solution and subseqnently drying the individualized fibers.

7. The process of decorticating and degumming fibrous plants which comprises immersing the fiber-bearing portions thereof, cut to desirable staple lengths, for a predetermined period of time, in a heated aqueous, weak alkaline, solution comprising the following chemicals in approximately the stated proportions:

Fresh water	gallons	1000
Tribasic sodium phosphate	pounds	719
Soda ash	do	62.5
Finely divided aluminum	do	23.3

maintaining the solution for said period of time at a temperature of approximately two hundred degrees F. to two hundred twenty-five degrees F., thereafter removing the material from said solution and washing the same to remove the chemicals and the foreign materials which have been released by the action of the solution and subsequently drying the individualized fibers.

8. The process of decorticating and degumming ramie and individualizing and bleaching the fibers thereof which comprises immersing the fiber-bearing portions of the ramie, cut to desirable staple lengths, for a suitable period of time, in an aqueous, weak alkaline, solution comprising major portions of tribasic sodium phosphate and minor portions of soda ash and aluminum sulphate, together with a small portion of sodium hypochlorite, acting to dissolve the gums and resins contained in the fibrous material, to precipitate tannic substances and to bleach the fibers without removing the waxes or oleaginous constituents of the fibers, but of insufficient causticity to affect the cellulose of the fibers, introducing steam into the solution in such manner as to maintain the temperature thereof at substantially the boiling point and by ebulition to cause agitation of the solution, thereafter removing the foreign material that has been released by the action of the solution, subjecting the fibers to the action of a mild solution of sulphuric acid, and then washing and drying the fibers.

9. The process of producing cellulose pulp from woods containing gums and resins which comprises immersing chips thereof for a suitable period of time in a heated aqueous weak alkaline solution, comprising a major portion of tribasic sodium phosphate and a minor portion of soda ash in the presence of an aluminum-containing substance, capable of dissolving the gums and resins contained therein, subsequently reducing the chips to pulp, and washing the pulp.

10. The process of producing "alpha" cellulose pulp from woods containing gums and resins which comprises immersing chips thereof for a suitable period of time in a heated aqueous weak alkaline solution, comprising a major portion of tribasic sodium phosphate and a minor portion of an aluminum sulphate, capable of dissolving the gums and resins contained therein, subsequently reducing the chips to pulp, washing the pulp, again treating the pulp in a like solution for a relatively short period of time, and finally thoroughly washing the pulp.

11. The process of producing "alpha" cellulose pulp from woods containing gums and resins which comprises immersing chips thereof for a suitable period of time in a heated aqueous, weak alkaline, solution comprising the following chemicals in approximately the stated proportions:

Fresh water	gallons	1000
Tribasic sodium phosphate	pounds	700 to 750
Soda ash	do	60 to 65
Aluminum sulphate	do	7 to 10

maintaining the solution for said period of time at a temperature of approximately two hundred degrees F. to two hundred twenty-five degrees F., thereafter removing the chips from said solution, disintegrating the same to produce pulp, and washing the pulp to remove the chemicals and foreign materials which have been released by the action of the solution, again treating the pulp by the same solution in approximately the same proportion for a lesser period of time, then thoroughly washing and drying the pulp.

FRANKLIN R. CHESLEY, JR.

CERTIFICATE OF CORRECTION.

Patent No. 2,073,682. March 16, 1937.

FRANKLIN R. CHESLEY, JR.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 4, first column, line 53, claim , for "subseqnently" read subsequently; line 64, claim 7, for "-----do----23.3" read----ounces---23.3; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 25th day of May, A. D. 1937.

Henry Van Arsdale
Acting Commissioner of Patents.

(Seal)