Jan. 7, 1936. J. A. MANAHAN 2,026,584

METHOD OF LIBERATING FIBERS

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Fig. 1

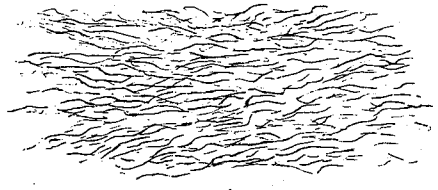

Fig. 2

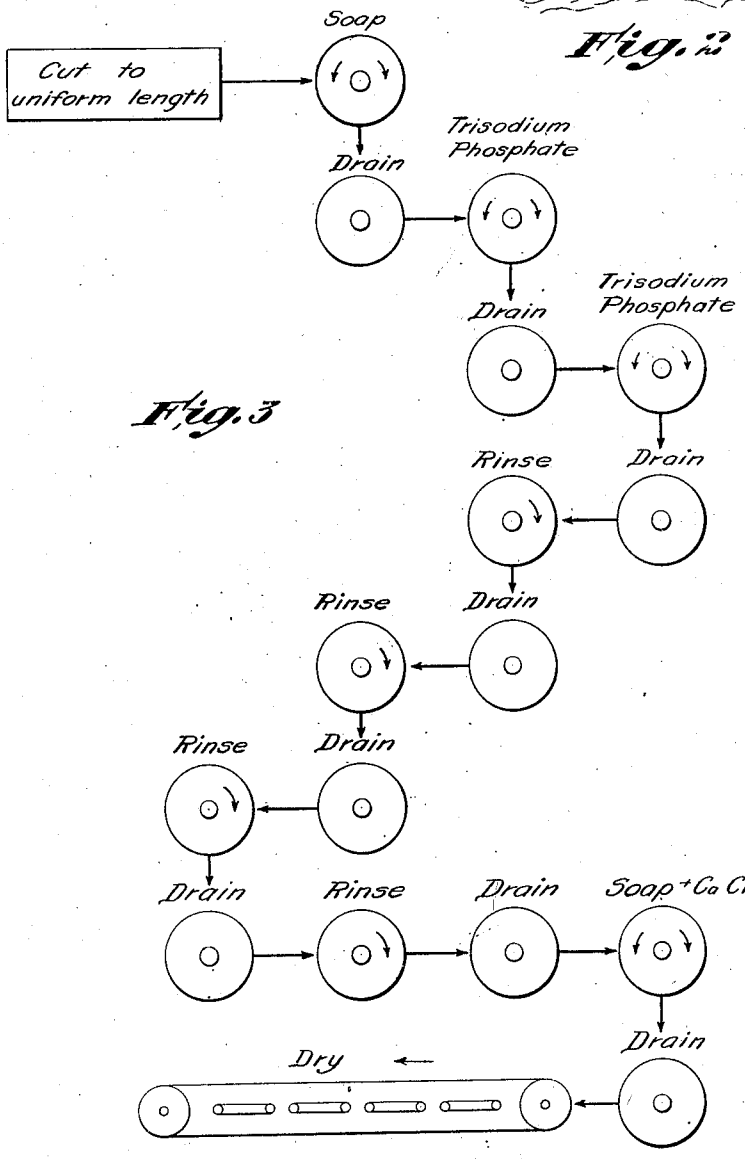

Fig. 3

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METHOD OF LIBERATING FIBERS

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19 Claims. (Cl. 92—9)

This invention pertains to an improved method or process of liberating vegetable fiber, useful for textile purposes, from lignin and other connective or incrusting tissues or substances. Among the fiber plants furnishing a plentiful supply of such fibers may be mentioned flax, hemp, jute, sisal, bagasse, esparto, and ramie or china grass. In all of these plants the useful fiber is embedded in, incrusted or coated with, or surrounded by connective tissues frequently of a gummy or mucilaginous character which must be destroyed and/or removed before the fiber can be made available for textile purposes. Commercial methods of freeing the fiber contained in some of these plants have been developed, but in most cases such methods are very slow and laborious and the nature of the treatment is often such as to impair the quality of the fiber, producing degenerated cellulose, low yield, loss of fiber strength or decrease in length of staple, while as respects some of such fiber plants, for example china grass, apparently no commercially successful method has heretofore been found.

Ramie or china grass (*Boehmeria nivea*) is a native of Eastern Asia, belonging to the nettle family and has a fiber of great length, which when freed of the abundant and very gummy connective tissue is of a beautiful silky lustre, and capable of being spun into fine yarns. This plant is easily cultivated, and the raw material, ready for fiber liberation, may be produced very cheaply.

I am aware that attempts have been made to liberate fiber of textile staple from some of the plants above mentioned by the use of chemical reagents, with the object of hastening and/or improving upon the older processes which depend largely upon slow bacteriological or mechanical operations, and which at best produce but a low yield of useful fiber, but so far as I am aware none of such chemical processes has been commercially successful. After a long series of experiments I have now devised a process capable of application for the liberation of textile fibers from any or all of the above-named plants, as well as others, and which is especially useful in the treatment of the more difficult materials such as ramie, whereby useful fiber of high quality, long staple and substantially unimpaired strength may easily and rapidly be produced. For example, long staple, substantially white fiber having a soft feel, silky lustre and capable of being spun and woven on usual cotton or woolen machinery may be produced from raw ramie at a cost not substantially exceeding that of cotton of equivalent staple.

By way of illustrative example, in the accompanying drawing I have indicated my improved process as applied to the preparation of ramie fiber, and in the drawing,

Fig. 1 is a view showing the general appearance of the dry, coarse, stiff, and straw-like raw ramie cut to a predetermined length ready for treatment, in accordance with my improved process;

Fig. 2 is a similar view showing the liberated fiber ready for delivery to usual textile machinery; and

Fig. 3 is a diagrammatic representation of a preferred order of steps comprised in my improved process.

While as above noted, my improved process is applicable to the liberation of fibers, and in particular bast fibers, from plants of many types, some of which have been specifically enumerated, I will describe it with particular reference to the liberation of fiber from ramie or china grass, with the understanding that a generally similar process is applicable to the liberation of fibers from other plant materials, which in most instances are more readily liberated from their connective tissues than is ramie.

With this understanding my present process preferably involves the following steps, it being noted that the dry raw material as received is of considerable length, averaging for example, more than twelve inches, and that since uniformity of fiber length is conducive to the production of yarn of high grade, and since existing cotton or woolen machinery is designed for working fiber which is comparatively short, I find it desirable, though not essential, as a preliminary step in my process, to cut up the raw stock to a substantially uniform length, selected in accordance with the nature of subsequent treatment to which the liberated fiber is to be subjected.

However, this preliminary step of cutting up the raw stock may be omitted and the raw material, as received, regardless of length, may be subjected directly to the chemical treatment about to be described.

Referring to the drawing, Fig. 1 shows the raw material 1 as having been cut to uniform length, for example one and one-half inches, if the material is to be worked upon cotton machinery. This raw material is dark in color, stiff, straw-like, and harsh, and in coarse strands of cohering fibers or "bundles" of non-uniform cross section and diameter.

Whether or not the raw material is cut to uniform lengths I now proceed to treat the material with a hot dilute water solution of a neutral soap, a tallow soap, preferably,—a one per cent. solution being preferred, and this liquor is maintained substantially at 212° F. during the treatment of the raw material, which lasts for a period of approximately thirty minutes. While a soap solution, diluted to one per cent. soap, is effective, I have found that the percentage of soap may be varied to some extent, for example, to five per cent. without detriment, although such increase

seems to have little beneficial effect, but I believe that a solution substantially less than one per cent. soap, is not desirable. The process may be carried out in a rotary drum, or other appropriate receptacle, for example, capable of being turned first in one direction and then in the other, or of being rotated continuously in one direction, as desired. For most effective results I believe that the receptacle should be arranged to turn about a vertical axis, as proper circulation of the liquid may thus be obtained without unduly tangling the fiber, but it is to be understood that any suitable apparatus in which the material may be exposed to the action of the hot reagent liquor may be employed. If the receptacle turn continuously in one direction, it may be furnished with relatively fixed blades or other stirring devices to assure rapid circulation of the liquid. Preferably the drum is driven at a rate of approximately three to four turns per minute during the period while it is undergoing treatment.

As a further safeguard against snarling or tangling the fibers, I may place them in porous bags, nets, or perforate baskets or other receptacles and circulate the liquid in one or opposite directions through such porous or perforate containers, for example by a pressure pump, as in certain well known systems of dyeing.

At the end of this period the soap solution is drained off and the container is immediately filled with hot water containing approximately one per cent. of a mild alkaline salt capable of softening the gummy and incrusting substances surrounding the fiber. Preferably I use trisodium phosphate ($Na_3PO_4.12H_2O$). The drum is again driven at approximately the same rate for about forty minutes, after which the liquid is drained off without opening or uncovering the drum. The drum is immediately filled a second time with a similar hot solution of trisodium phosphate, and the operation repeated for another period of approximately forty minutes, the temperature of the drum contents being kept at approximately 212° F. throughout the treatment with the alkaline reagent. It is to be noted that during treatment with the alkaline trisodium phosphate, after a previous soap treatment, a violent foaming takes place, and this foaming is, I believe, an important factor in separating the gummy and other extraneous substances from the useful fiber, the foam perhaps acting more or less mechanically to support the gummy materials out of contact with the fibers, or to emulsify such gummy materials and prevent recoalescence of the gummy substance.

The liquid is again drained off without uncovering the drum, and the fibrous material is repeatedly rinsed in plain water at boiling temperature and drained, as many as seven rinsing waters preferably being employed, the drum being turned in one direction at a rate of approximately seven revolutions per minute for a period of five minutes during each rinsing.

After draining off the final rinsing water, the drum is filled with a hot one per cent. soap solution similar to that first employed but with the addition of some suitable softening reagent, usually of a waxy or oily nature, for example one-tenth of one per cent. of coconut oil, and with the further addition of approximately one-tenth of one per cent. of calcium chloride ($CaCl_2$). The drum is again driven for approximately fifteen minutes while keeping the temperature of the contents at approximately 212° F. and then drained, and the fiber, now completely freed from connective tissue and incrusting matter, and of a very light, nearly white shade, of a soft and silky lustre, and with a tendency to curl very suggestive of that of the best cotton or of wool, is now dried, preferably slowly and without substantial agitation, by placing it upon a wire screen or conveyor belt and subjecting it to a drying medium, such for example as warm or predried air.

The fiber as thus prepared is comparable to cotton or wool in working qualities, being somewhat hygroscopic by reason of the calcium chloride treatment, and having the necessary softness for machine operation by reason of the small percentage of oil or wax applied prior to drying. Moreover, since the fibers are all of substantially the same predetermined length, it is possible to work them into yarn with far less difficulty or waste, than is possible with fibrous materials of non-uniform character.

In treating fibrous materials, other than ramie, I find that it may be desirable to vary the length of the several periods of treatment from those above instanced. For example, in treating sisal the treatment in the trisodium phosphate bath may be of the order of one hour's duration, while in treating flax and hemp each period in the trisodium phosphate may be of the order of forty minutes. The other treatments may likewise be varied in length in substantially the same proportion.

I am aware that it has previously been proposed to employ trisodium phosphate as a fiber liberating medium for paper making, where a very short staple, unsuitable for textile purposes, is required, but for some reason unknown to me a single treatment with this reagent alone apparently fails when applied to the liberation of bast fiber for textile purposes, at least on a commercial scale. While I do not know the exact reason for this failure, I have observed that the trisodium phosphate solution appears to have a marked solvent effect upon the connective tissue during a certain period of treatment, after which, perhaps, saturation takes place, and that thereafter the action appears to be reversed and the gummy material is redeposited upon the fiber, causing the fiber to adhere in masses and leaving it in a condition wholly unsuitable for textile purposes. On the other hand, if the raw material be first treated with a soap solution, as above described, and particularly if the trisodium phosphate treatment be divided into a plurality of successive stages, each terminating before the saturation period is reached, and if precautions be observed to prevent the temperature of the material from dropping substantially in the intervals between successive steps in the operation, the fiber is completely freed from the gummy material even though the period of treatment may vary very considerably, and there is apparently no danger of a re-precipitation of the gummy substance upon the fiber after it has once been removed. Possibly, the observed effect may be due to a physio-chemical action,—i. e., a chemical or solvent action and a physical or emulsifying action, in which some of the reagent material may be partly ionized so as to become effective in dissolving the gummy substance, while on the other hand, the soap, being a colloid, may act to disperse the gum and subsequently act as a stabilizer to prevent recoagulation of the latter. I prefer, as above described, to carry out the treatment with the trisodium phosphate in two or more successive stages, using a fresh solution at each stage.

While I believe that the second treatment with the soap solution and the calcium chloride and oil may, under some circumstances, be dispensed with, I find that by treating the liberated fiber in this way it is left in a better condition for subsequent textile operations, for example, it is softer and more pliable and the addition of the calcium chloride, even in very small amount, to the solution, makes the fiber somewhat hygroscopic so that it absorbs sufficient moisture from the air to keep it from becoming harsh and brittle, and serves as a mordant to assist in drying.

I have found that this or an equivalent step is very desirable to the proper working of the fiber during the subsequent process of yarn and cloth manufacture.

I have hereinabove referred to the use of trisodium phosphate as the preferred liberating reagent, and have found that its mild alkaline action is conducive to the best results thus far obtained. However, desirable though less valuable results (from the commercial standpoint at least) may be obtained by the use of other soluble alkaline reagents, for example tripotassium phosphate (K_3PO_4), sodium carbonate (Na_2CO_3), borax ($Na_2B_4O_7$) or ammonium sulphide ($(NH_4)_2S$), the substances which I have thus found useful for the purpose falling within the general category of mild alkaline salts.

While I have found a soap solution to be very effective for the purpose of pretreating the fiber before cooking it with the alkaline reagent, I have observed that certain other substances, for example naphthalic acid, castor or coconut oil, having an emulsifying action, in the presence of alkali, upon the gummy substances of the plant, may be employed in place of the soap, and I regard emulsifying reagents in general as useful in this particular.

Furthermore, while I have mentioned calcium chloride as a desirable hygroscopic agent, I may instead use sodium chloride (NaCl), ethyleneglycol or glycerine with good results, and believe that any cheap hygroscopic substance, harmless to fiber color or strength might be employed. I also contemplate the use of oils or waxes, which may be sprayed on or otherwise applied to the fiber, for example, after drying, as substitutes for the soap or the hygroscopic substance, or both in the final stage of the process, such oils or waxes, for example, sulphonated castor or coconut oil, being well known in the textile arts as reagents for softening fibers and making them more amenable to the spinning process.

I further contemplate an increase in the number of successive treatments with the alkaline reagent, either with or without a decrease or increase in the length of each period of treatment as within the scope of the invention.

After the rinsing operation the fiber is very light in color, quite curly or crinkly, resembling the best cotton or wool in this particular, and, being even more absorbent than cotton, is very readily bleached to a dazzling white, and takes dyes with the greatest ease.

While I have hereinabove described my process in its specific details, I wish it to be understood that variations in the process, as for example by the substitution of materials; by a change in the order of process steps; and by variation in the periods of treatment of the material at the successive stages of the process, may be made without departing from the spirit of the invention as expressed in the appended claims.

I claim:

1. That method of liberating plant fiber from connective tissue which comprises treating the raw material with water containing an emulsifying agent, thereafter treating the material with a water solution of a mild alkaline salt, rinsing the material, and treating the material with a softening agent prior to spinning.

2. That method of liberating plant fiber from connective tissue which comprises violently agitating the raw material in hot water containing a colloidal emulsifying agent until the fiber is thoroughly saturated with said agent, drawing off excess fluid, agitating the drained material in a hot aqueous solution of an alkaline salt of phosphoric acid, draining and rinsing the material, treating the material with a hygroscopic agent, and drying the material.

3. That method of liberating plant fiber from connective tissue which comprises as steps subjecting the raw material to hot water containing a reagent capable of emulsifying the gummy substances contained in the connective tissue after liberation, drawing off the surplus liquid, agitating the material in a hot solution of a mild alkaline salt capable of softening and separating the gummy substances from the fiber, draining off the liquid, rinsing the material in hot water, and drying the material.

4. That method of liberating plant fiber from connective tissue which comprises keeping the material at a temperature approximating 212° F. while it is undergoing the several successive steps of the process, first agitating the material in a bath containing a colloidal emulsifying agent, capable of dispersing the gummy substances of the connective tissue when such gummy substances are liberated, until the material is saturated with said agent, thereafter agitating the material in a bath containing a soluble alkaline salt capable of softening the connective tissue and liberating the gums contained therein, and thereafter rinsing the material.

5. That method of liberating plant fiber for textile purposes which comprises as steps saturating the raw material with water containing soap in solution, thereafter treating the material with water containing trisodium phosphate in solution, and rinsing and drying the material.

6. That method of liberating plant fiber from connective tissue which comprises as steps saturating the raw material with hot water containing substantially one per cent. of soap in solution, thereafter treating the material with hot water containing approximately one per cent. of trisodium phosphate in solution, rinsing the material, again treating it with a hot soap solution, and drying the material.

7. That method of liberating plant fiber from connective tissue for textile purposes which comprises as steps treating the raw material with a hot aqueous solution of soap, thereafter treating the material with a hot aqueous solution of trisodium phosphate, rinsing the material, treating the material with a hot aqueous soap solution containing a hygroscopic substance, and drying the material.

8. That method of liberating plant fiber for textile purposes which comprises as steps treating the raw material with hot water containing substantially one per cent. of soap in solution, thereafter treating the material with hot water containing approximately one per cent. trisodium phosphate in solution, rinsing the material with hot water, again treating the material with hot water containing approximately one per cent. of

soap in solution and also approximately one-tenth of one per cent. of calcium chloride in solution, and thereafter drying the material.

9. That method of liberating plant fiber for textile purposes which comprises as steps cutting the previously dried raw material into substantially uniform predetermined lengths, agitating the raw material thus cut into uniform lengths in a hot dilute soap solution, draining off the solution, treating the material with a hot dilute solution of trisodium phosphate, rinsing the material in hot water, treating the material with a dilute soap solution containing a hygroscopic substance, and thereafter drying the material.

10. That method of liberating plant fiber for textile purposes which comprises as steps placing the cut raw material in a perforate container, causing a hot dilute soap solution to circulate through said container for a period of the order of thirty minutes, draining off the solution, successively circulating through the material, for periods of approximately forty minutes, respectively, hot dilute solutions of trisodium phosphate, subjecting the material to repeated rinsings in hot water with drainings between the several rinsings, agitating the material in a hot dilute soap solution containing calcium chloride, and drying the material.

11. That method of liberating plant fiber from connective tissue which comprises as steps agitating the raw material in water at approximately 212° F. containing substantially one per cent. soap for a period of the order of thirty minutes, draining off the solution, agitating the material in a one per cent. water solution of trisodium phosphate at a temperature of approximately 212° F. for a period of approximately forty minutes, draining off the solution, repeating the boiling and agitation in a solution of trisodium phosphate for a similar period, draining off the solution, rinsing the material by agitating it for periods of approximately five minutes each, with intermediate drainings, but without uncovering the receptacle, in plain hot water, agitating the material for approximately fifteen minutes in hot water containing one per cent. soap and one-tenth of one per cent. calcium chloride in solution, draining off the solution and air-drying the material without substantial agitation.

12. That method of liberating plant fiber from connective tissue which comprises as steps placing the raw material in a perforate container, forcing a fluid reagent to circulate through the container, said reagent being a boiling solution of trisodium phosphate, discontinuing the treatment with said reagent before the latter has become saturated with gummy constituents of the raw material, circulating a rinsing liquor through the material, and repeating the above steps until the fiber has been substantially freed from gummy substances.

13. That method of liberating plant fiber from connective tissue which comprises as steps circulating a boiling solution of trisodium phosphate through the raw material to be treated, discontinuing such circulation of the solution before the latter has become saturated with gummy substances, draining off the solution, rinsing the material under treatment, and repeating the above steps until the fiber has been substantially freed from gummy substances.

14. That method of liberating plant fiber from connective tissue which comprises as steps circulating a hot solution of trisodium phosphate through the raw material to be treated for a period less than that beyond which re-precipitation of gummy substances would begin, draining off the solution, rinsing the material in hot water without permitting the temperature of the material substantially to fall, and repeating said steps until the fiber has been substantially freed from the gummy substances.

15. That method of liberating plant fiber from gummy connective tissue which comprises as steps agitating the raw material in a boiling water-solution of trisodium phosphate for a period of approximating, but less than, that beyond which re-precipitation of gummy substances would begin, draining off the solution and rinsing the material in hot water without permitting the material substantially to drop in temperature, repeating said steps until the fiber is substantially free from gummy substances, and thereafter treating the fiber with a hygroscopic agent.

16. That method of liberating plant fiber from connective tissue for textile purposes, which comprises as steps subjecting the raw material to the action of water at approximately 212° F. and containing approximately one-tenth of one per cent. of trisodium phosphate in solution, rinsing the material with hot water without permitting the temperature of the material substantially to drop and without exposing it to the air, repeating the preceding steps, thereafter further rinsing the material in hot water, and drying the material.

17. That method of liberating plant fiber from connective tissue for textile purposes, which comprises as steps causing water at approximately 212° F. and containing approximately one-tenth of one percent. of trisodium phosphate in solution to circulate in contact with the raw material for a period of appproximately forty minutes, draining off the liquor without permitting the temperature of the material substantially to drop, circulating a fresh trisodium phosphate solution of substantially the same strength and temperature through the material for a similar period, again draining off the liquor without permitting the temperature to drop, and repeatedly rinsing the material with hot water.

18. That method of liberating plant fiber from connective tissue which comprises as steps repeatedly subjecting the material to be treated to a boiling water-solution of a mild alkaline salt, each successive treatment being in a fresh solution and each such treatment being followed by a rinsing in hot water, the entire process being carried out without substantial drop in temperature of the material from beginning to end and without free exposure of the material to the open air.

19. That method of liberating plant fiber from connective tissue which comprises as steps first treating the raw material with a foam-producing reagent, thereafter treating it with a boiling solution of a mild alkaline salt, rinsing the material without permitting its temperature substantially to drop, again treating it with a boiling solution of a mild alkaline salt, rinsing the material, treating it with a softening reagent, and drying the material.

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