Sept. 6, 1938. J. H. ESTES 2,128,928

ART OF FIBER LIBERATION

Filed June 29, 1932

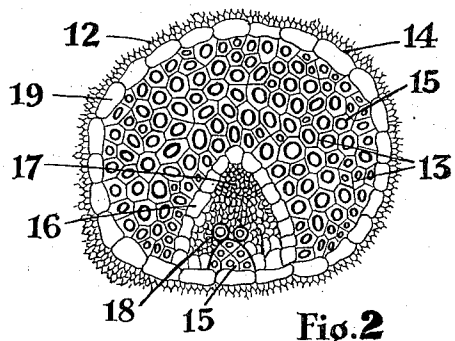


Fig. 2

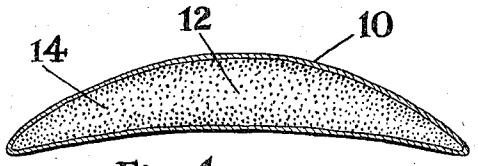


Fig. 1

Fig. 4

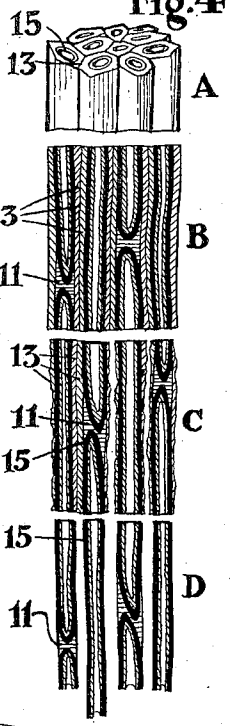
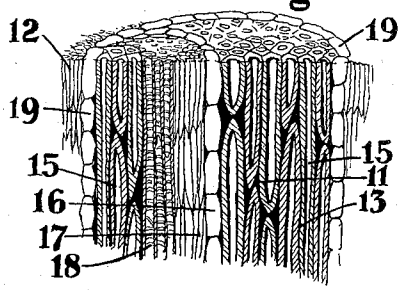


Fig. 3



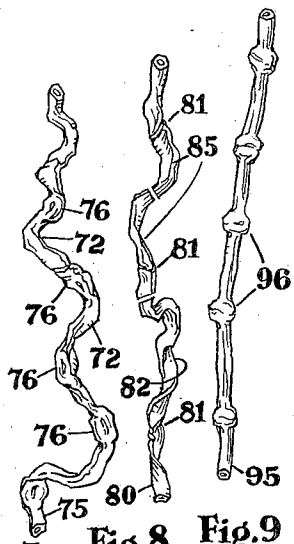


Fig. 7 Fig. 8 Fig. 9

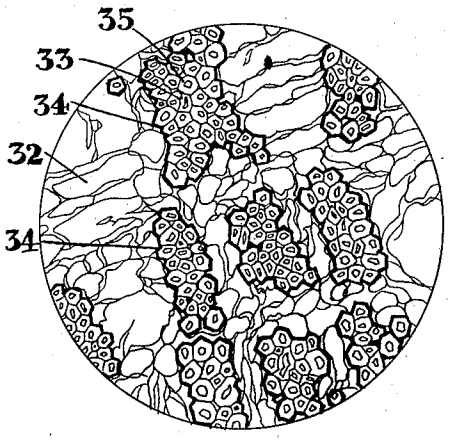


Fig. 6

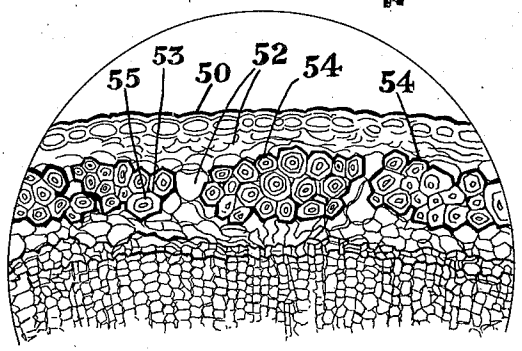


Fig. 5

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2,128,928

ART OF FIBER LIBERATION

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Application June 29, 1932, Serial No. 620,054

10 Claims. (Cl. 92—14)

This invention relates to improvements in the art of fiber liberation and in fibrilized products thereof.

More particularly the invention involves the treating of "fiber" with a liquid composition which, by reacting chemically with certain parts of the fiber, and by avoiding reaction with certain other parts, makes at low cost a fibrilized product having new or greatly improved properties.

The invention deals with the mutual relations of ultimate cells of the fiber; and it provides for their selective and limited separation, viz., separating certain ultimate cells from certain other ultimate cells which lie adhesively at microscopic distances beside them; and it provides for the doing of this without injury to either of the said separated cells, and without breaking or weakening the endwise adhesions of such cells. The successful achievement of this selective control has brought with it the promise of great benefit to industry and the arts.

The treatment is herein described with reference to what are known to commerce as long vegetable fibers. Having successfully applied the invention experimentally to a great number of such, both of bast and of leaf origin, it appears to me probable that the invention is applicable with benefit to all bast and leaf fibers. And it has also been applied with advantage to some other kinds of plant fiber.

Preferably the invention is applied to the "fiber" after any customary or suitable retting or decorticating process; that is, after the cortex and woody portions of the stalks, or the pulp in case of leaf fiber, have been removed.

In "fiber" seen at this stage, or after the further processes practiced to prepare it for spinning, that which the eye observes as a typical one of the separate filaments, however small, is nevertheless an aggregation of smaller filamental elements (fibrils) whose individual identity is practically lost in the snugness and tightness of their parallel nesting together. Each fibril consists of a succession of elemental tubular cells joined end to end with intervening strong cementitious filling matter between them. The mass of such fibrils which thus coheres as a filamentary unit is commonly called "a fiber."

In the plant the fibrils grow in these cemented-together groups, called bundles. Each "fiber" produced by the retting process may contain one or several of these bundles, which each in turn may contain few or many fibrils. The tubular fibril cells are of microscopic diameter,—of the order of one to two one-hundredths of a millimeter in many kinds of plants. But they differ greatly in length, ranging from one or two millimeters, in some kinds of plants, to twenty or more centimeters in others. The invention provides for removing, from the microscopic interstices, cementitious matter which intervenes laterally between cells, thus freeing the fibrils from each other; without at the same time weakening the endwise cementitious connections of the cells; and without chemically degrading the tubular walls of the cells; thus leaving the freed fibrils individually intact and strong.

This selective erosion of intercellular matter produces a sort of chemical catamorphism, throughout a mass or batch of fibers of convenient size for economical industrial handling, such that each "fiber" becomes split or sub-divided. As the action is progressive the fineness of splitting can be controlled, to a degree; but if carried to the end it constitutes a sub-dividing of the fibers into individual strings of single connected cells (fibrils),—or even into single long cells in the case of ramie or other plants having cells of comparable extreme length. Also the product from the original heterogenous material may contain, in some instances, cells of other variety than that above described, e. g. cells which are not ordinarily in a strict sense considered to be part of a fibril, although the string be comparable to one or a small number of fibrils in diameter. I therefore call the invention a fibrilizing process, and the fiber-bundle fragments "splits".

The improvement by which I have accomplished this involves the excluding of caustic and other strong alkali from the treatment; the excluding of acid; the making of a colloid which is so perfectly a hydrophilic colloid that it can penetrate fully the microscopic interstices and thus thoroughly can wet the heterogeneous materials that make up the "fiber", and is of such composition that it can serve as a protective colloid for a selected portion of the fiber; the making of an alkali reactive solution whose notable and essential characteristic is that its alkali is extremely weak, within limits; the supplementing of this, for certain kinds of fibers, with a sodium sulfite characteristic; and the careful control, according to certain principles, of the conditions and duration of application of the solution to the fiber.

The accompanying drawing, which is more or less diagrammatic, illustrates the invention by showing plant and fiber structure on the basis

of photomicrographic representations in standard treatises:

Figure 1 represents a cross section through a leaf of sisal, the dots being fiber bundles;

Figure 2 represents a cross section through one of the said fiber bundles, greatly enlarged;

Figure 3 represents in perspective a combined longitudinal and transverse section through such a fiber bundle; and

Figure 4 is a schematic drawing on a still larger scale, large enough for details to be shown indicating the three kinds of material between which the selection is made, and representing at A, a fragment of a "fiber", in side elevation, at B a similar fragment in section corresponding to the longitudinal portion of Figure 3; and at C and D the split condition at successive stages of treatment, viz., at C a portion of such a fragment upon which the process of the invention has operated to a certain extent, sufficient to free some of the ultimate cells of their lateral junctures, making separated fibrils; and at D a portion of such a fragment on which the reaction has proceeded further, so that the lustrous cellulose of each ultimate cell is cleaned of all of its lateral surrounding lamellae of cementitious matter, but remains with its own cell walls and its endwise cementitious junctures intact, having fully withstood the chemical attack.

Figure 5 represents a cross section through a fragment of a stalk of flax, greatly enlarged, with introduced heavy outlines indicating fiber bundles such as customarily constitute spinning units or filaments; and

Figure 6 represents similarly a fragment of a stalk of jute, in the bast region, showing by introduced heavy outlines the agglomerated bundles of bast fiber, each bundle representing a spinning unit or filament.

Figure 7 represents a fibril of ramie, as it appears when seen microscopically, having knobs, between which bends have been made, giving kink to the fibril;

Figure 8 shows similarly a typical development of kink in a fibril not having knobs, as of jute or hemp; and

Figure 9 shows similarly a fibril, as of istle, sisal or Sunn hemp, in which knobs existing therein have been made prominent by the treatment.

Figure 1 shows a typical fiber-bearing leaf of a kind of plant (in this instance sisal, *Agave sisalana*) whose fiber is strong and long but has hitherto been used mainly for coarse work, as cordage, because it has not been capable of economical sub-division for fine work. The epidermis 10 surrounds a pulpy mass of parenchymous tissue 12 in which are embedded a multitude of fiber bundles 14. Any one of these bundles 14 is a filamentary unit as now customarily used in industry, called "a fiber" of sisal. But each "fiber" is really a closely agglomerated group of numerous fibrils, as represented in Figure 2. By the invention this group can be handled in mass with others as a mass of fibers and each fiber in the mass be individually fractioned into sub-groups of its fibrils, or even into its individual fibrils. These fibrils consist of relatively long tubular cells, Figure 3, the length of cell, in the case of sisal, being one hundred times the cell diameter. But as that proportionately great length really amounts to only 2.5 millimeters (the average for sisal) it is clear that a separation of cell from cell in the individual fibril would not leave a unit of practicable length for spinning into a strong thread. The cells 15 are of cellulose, hard and strong, with walls thickened by interior growth so that they are called sclerenchymous. Other kinds of long cellulose cells seen in these figures are the bundle sheaf cells 16, phloem or bast cells 17, and the tracheids or wood cells 18. The envelope of starch cells 19 is of no industrial importance; but the various cells 15, 17, 18 have permanent value. In the plant, and in the fiber bundle after its preparation treatment for spinning, as applied hitherto, they remain still cemented together in their bundle.

Figure 4 illustrates schematically the relative arrangements of adjacent fibrils in leaf fibers, and will serve equally to indicate the same for bast or stalk fibers. However, it should be noticed that, for convenience of illustration, the lateral dimensions are exaggerated, as to thickness of filling between cells; and that the number of cell-ends represented is greater than would ordinarily occur in the length of fiber which is portrayed. Microscopic investigations of structure, aided by differential staining, have shown the cells 15 surrounded by cementitious lamellae whose chemical character varies progressively as one proceeds outward from the pure cellulose of the cell wall, whether proceeding outward laterally of the cell or endwise of the cell. It is the purpose and effect of the invention to remove the middle lamella 13 and more or less of its adjacent lamellae also marked 13 which intervene between cells 15, in order to release the cells 15 from their mutual lateral attachment; and yet to avoid removing or substantially weakening those bits 11 of cementitious material which stand between any cells 15 which lie end to end. The latter constitute endwise junctures making a longitudinal succession of cells into a fibril. Both deposits of cementitious matter 11 and 13 usually consist of pectic substances of various kinds, except that in some kinds of plants 13 sometimes has strong ligneous characteristics.

Figure 5, illustrating the bast region of a stalk of flax shows the epidermis 50 of the cortex which surrounds the mass of parenchymous cells 52 in which are found the bundles 54 of sclerenchymous cells, which bundles survive the retting process. The heavy lines around the bundle are introduced to indicate the limits of the bundles portrayed, each of which bundles, in ordinary practice, may constitute "a fiber" or filamentary spinning unit of flax or linen.

Figure 6 is similarly a portion of a cross section of the bast region of a stalk of jute, as shown by photomicrograph in a standard treatise, in which the agglomerate character of each spinning "fiber" unit or filament 34 is manifest, consisting of a group of sclerenchymous cells 35 of bast tissue with intervening parenchymous 32. The coarseness of fabric woven from jute, as burlap, is well known. The invention, by fibrilizing the bundles 34, splitting them into smaller groups or into individual fibrils, makes from jute a spinnable fibrous product from which cloth can be made of very different aspect, because the separate filaments are so soft and flexible.

Fiber bundles of other stalks and leaves, which the process of the invention can likewise split, producing industrially available filaments having the improved qualities herein indicated, are typified by these showings notwithstanding the divergences which exist between kinds of plants, and sometimes between grades of fiber from the same kind of plant. The procedure of the in-

vention is to attack the middle lamella 13, 53 or 33 with a weak alkali capable of moving it by solution and/or chemical attack; while retarding the chemical attack on the cell wall 15 and its more intimate enclosing lamellae which the chemicals used are capable of affecting; and while avoiding the use of chemicals which might attack the materials constituting the endwise juncture 11 between cells. As the effects must be gained by penetration by the protecting material along with the attacking material into spaces of extremely small dimensions, a first requisite is the making of a colloid whose continuous phase (in this case water) will be perfectly adsorbed on interstitial surfaces and which is so perfect a colloid that its dispersed phase (in this case a neutral soap) will follow in for giving the desired protection.

For the making of such a hydrophilic colloid I have found it satisfactory to put finely divided well dried neutral soap into water which is boiling. The boiling water has a temperature and a concomitant agitation which will assure the making of a hydrophilic dispersion if the quantity of soap put in be of a proportion ranging between .2 and .5 of one per cent of the quantity of water. With a percentage of soap higher than 1% there is probability of making a hydrophobic mixture or, at any rate, a mixture which does not penetrate among fibrils completely and operate uniformly. With less than the proportion stated, the desired hydrophilic colloidal state may not arise. If the soap be put into cold water which is later heated there is also danger of missing the desired result, because the soap may become hydrolized. But the making of a hydrophilic colloid as stated evidently carries the soap thoroughly into the interstices between fibrils, as openings between them become available during the treatment, and protects the cells from the attack of the alkali of the solution.

For the alkali I prefer to use sodium carbonate in the weak hydrated form called sal soda wherein each molecule is associated with ten molecules of water; and of this crystalline substance I take a quantity in the range between .9 of 1 per cent and 1.3 per cent of the quantity of water into which it is to be put. The equivalent range of anhydrous sodium carbonate would be from a third to a half of one per cent, and this is the strength or concentration of alkali in the liquid which that amount of sal soda produces. This, like the soap, is to be ground fine and put into the boiling water.

Preferably the soap and the alkali should be ground and mixed preliminarily, to be put into the water together, for experiment has shown me that the process works less well when the chemicals are not pre-mixed.

The fiber, preferably in quantity weighing about 5 or 10 per cent of the water, may be put into the boiling liquor thus prepared, and the vigorous boiling continued in an open vessel for about a half hour. Boiling in a kier or a closed vessel is to be avoided, as not providing a desired access of atmospheric oxygen. If an oxidizing agent be added in a closed vessel the results are likely to be not uniform.

The fiber is to be removed from the boiling liquid when it can be ascertained, by rubbing a sample between the fingers, that its initial harsh feeling has changed to a slimy, slippery, soft and silky feeling. Over a wide range of fibers I have found that the proper duration of boiling ranges between 25 and 45 minutes; and that the stated half hour is about right for most fibers.

Upon removal from the liquor the fiber is to be rinsed, drained and dried.

If treated with acid the product will be weakened, or even broken into short lengths.

Weakening may also result from having alkali too strong in the liquor. And, if treated too long by the specified proper liquor, the fiber will lose in strength, which is due presumably to progress by the alkali in converting cellulose of the cells 15 to oxycellulose which is brittle. This converting reaction is retarded by that quality of the protective colloid which draws it to the surfaces of the cell, and thoroughly wets those surfaces, and adsorbs the protective film of neutral soap strongly thereon. Coupled with this are the concomitant weakness of the attack of the alkali on the cellulose, because of the low alkali concentration; and the constant progress meanwhile, during this retardation, of the business of removing the alkali-soluble pectic bodies. As the chemical reaction with and/or solution of these pectic bodies progresses, parts of the middle lamellae disappear, and thus the firm lateral junctures between cells diminish. Progressively the ultimate fibrils come to be only weakly connected to each other, a state in which they may later become separated by mechanical rubbing; and progressively entire separations occur as the action of the liquor continues. Separations first occur by breakages through weaker places in the midst of a fiber bundle, leaving the bundle in split fragments. If the treatment be continued long enough, every fibril will come apart from every other. And then, by a further continuance of the treatment, there follow other results to the individual fibrils, as mentioned hereinafter.

The fineness of sub-divisions may be controlled selectively by selecting the time for discontinuing the treatment; stopping it, for instance, a little early, or a little late, to get respectively a coarser or a finer sub-division of fibril bundles, or continuing it still longer to get sub-division into individual fibrils. But in each case the retention by the cells of their native tensile strength, and their endwise connections, leaves them workable as long filaments (fibrils). These retentions of length and strength are important features which distinguish the product of the present invention.

The explanation underlying this important feature is, I believe, that some among the numerous kinds of pectic bodies, which are present in plant stalk and leaf fibers, are soluble in acid, and some in alkali. It is known however that wood fiber can be broken into elements short enough for making paper by either acid alone or by soda alone. Alkali attacks cellulose, converting it to oxycellulose by adding oxygen; and the tubular walls of the ultimate cells 15, 35, 55 consist of cellulose of microscopic thickness. However, it happens that those pectic cementitious bodies which stand laterally between the fiber cells are the ones capable of being taken into solution by the treatment with very weak alkali above described; while those pectic cementitious bodies which connect the ultimate cells endwise resist alkali. They yield to acid. But because caustic alkali is excluded from the formula provided by the invention; and strong concentrations of soda ash are excluded; and also acid is excluded; and the penetrative protective colloid is provided; under these conditions the composition is capable of completing its work of dissolving the pectic

middle lamella before damaging effect upon the cellulose has become appreciable.

The selectivity of action is especially important in dealing with kinds of fibers which have not hitherto been sub-divisible without sacrifice of strength, because some of such are inherently much longer and stronger and are at the same time less costly than the kinds of fiber commonly used.

Some fibers are not completely fibrilized by the treatment above set forth. This depends upon the nature of the plant and to some extent the age or toughness of the individual plant. For unusual toughness the concentration of alkali may be increased and used near the higher limits stated, where the fiber is of a kind with which the above treatment is usually satisfactory. These include jute, pita, sisal, ramie, flax, hemp, bear grass, *Furcraea macrophylla*, Mauritus hemp, Colombian smooth leaved pita, bow string hemp; and for these the above formula of mild carbonate concentration with a protective colloid will satisfactorily suffice.

But in some kinds of plants the cementitious material which stands laterally of the ultimate cells in a fiber bundle has a ligneous component such that it is not sufficiently removed by a weak alkali solution as defined above. These include manila, New Zealand flax, lowest grade sisal (butts and aged stock), cabuya, Colorado River hemp, and other very strong fibers which have never heretofore been made available on a commercial scale for fine spinning so far as I am aware, because of inability to make the subdivision.

For the successful dealing with such it has been discovered that the process operates best when the described liquor is supplemented by addition of a weak concentration of sodium sulfite. It is then effective to remove the ligneous element as desired while still avoiding attack on the cellulose of the cell and on the matter which connects the cells endwise. In such case I modify the proportion of sal soda in the formula given above, so that the total percentage of Na_2O from both the carbonate and the sulfite will be about the same as from the carbonate alone in the formula above given. Otherwise the materials and procedure are similar.

When the crystalline sodium carbonate is used the following proportions serve:

	Per cent
Neutral soap, well dried	.2 to .5
Sal soda	.5 to 1
Sodium sulphite	.2 to .75

These are to be preliminarily ground and mixed together and then put into boiling water 100%. In the liquor thus formed the fiber is to be boiled vigorously until a sufficient removal of the lateral cementitious material is discernible by the testing of a sample by feel as above described.

If the formula be varied by using the sodium carbonate in the form of the monohydrate Na_2CO_3,H_2O, its percentage relative to the water would be in the vicinity of .3 to .8% of the water; or, if used in the anhydrous form of soda ash Na_2CO_3, the range of about .2 to .7% of the water into which it is put would be equivalent to the preferred formula.

The desirability of excluding caustic alkali having been explained above, in order to avoid having such character or strength of alkali as will appreciably attack the cellulose, it remains to mention two exceptions; first, that such infinitesimal degree (if any) of hydrolysis as occurs incidentally upon the putting of sodium carbonate into aqueous solution has no appreciable effect, and may be treated as a nonentity. Second, a weak concentration of sodium hydroxide may be safely employed to provide the desired alkalinity, if this be in conjunction with the sodium sulfite, because the latter acts as a retarding agent, and in effect renders nugatory the potential attack of the caustic on the cellulose. In that case the sodium hydroxide NaOH may be taken within the range of .1 to .5% and sodium sulphite Na_2SO_3 within about the range of .2 to 1%. Being ground and mixed together and put into 100% of boiling water, a sufficient alkalinity is present for dissolving the lateral pectic bodies, and sulphite for dissolving the ligneous bodies, without affecting the endwise cementitious matter of cells. But the tendency of the sodium hydroxide to degrade the cellulose into oxycellulose is restrained because the sodium sulphite, acting as a reducing agent, seizes upon a large part of whatever oxygen is found in the liquid and so preempts it from use in a reaction with the cellulose.

The sodium sulphite has its best effect of separating cells laterally when used in conjunction with weak alkali as herein set forth. This has the notable industrial benefit that it renders available, for fine spinning, the fibers of various kinds of plants which are known to have especial merits but have not been thus available before. Obstacles have been the extreme difficulty and high cost of getting them finely divided, if indeed this has been possible at all except by the use of alkali in such strength as has disintegrated or too much weakened the product. Among such are New Zealand flax, manila, cabuya, istle, lowest grade sisal.

The discovery has followed that the known great strength of some of these tropical fibers can be retained in the sub-divided fiber and utilized in cloth and cords of very great strength as compared with the cotton and other fibers which have been the only material hitherto available for the desired small sizes. Applied in the manufacture of automobile tires, the cords may be both smaller and stronger. Applied in cloth the woven fabric may be fine and flexible; but also, because of the nature of certain fibers rendered available by the invention, and hitherto available only in harsh and coarse form, as jute, manila and sisal, cloth may be made from these fibers which proves to be surpassingly soft and silky. Such cloth may be close woven, and even be tight against passage of air or water; greatly surpassing cotton and linen in strength, and yet being less than cotton in cost.

An important matter from the industrial standpoint is the getting of uniformity of product, as regards degree of sub-division. A feature of the process which contributes to this is the perfection of the hydrophilic colloid, and this is promoted both by the observance of the stated limits and the maintenance of a boiling condition of the water when the mixture is introduced. Another contributing feature is the maintaining of a condition of vigorous ebullition during the treatment. This tends to equality of application of the process to all parts of the fiber.

The treatment may be stopped when the described stage has been reached as between some but not all of the fibrils of a bundle. The fiber bundle will then be fractioned into smaller groups of fibrils; and for some industrial uses

this will be sufficient and be even better than the extreme sub-division which is obtainable by continuing the treatment till all individual fibrils are separated.

In the plant stalk whose fiber is to be used, the said fiber bundles stand in the bast region, around the pith and/or woody center; and the whole is surrounded by the cortex or bark-like exterior. In the case of flax, jute and some other the center and the cortex are loosened by the fermentative decay, called retting, of the weak parenchymous cells surrounding the fiber bundles and intervening between them. The separation from wood and cortex is completed by pounding, combing and pulling the fiber, in the mechanical operations of hackling and scutching; or in some cases by human chewing; while in other cases, as manila, the initial separation from unwanted tissue is purely mechanical. But these steps merely segregate the fibers (bundles 14, 34, 54) from unwanted matter. Such partial success as may have been attained in splitting bundles, whether by long continued bio-chemical decay in the retting, or by treatment in boiling solutions of alkali and/or acid, or by maceration, has been always, I believe, accompanied by injury to and deterioration of the fibrous product, and has resulted in the weakening of the cellulose cells, or their endwise junctions.

The fibrilized product of the present invention is distinguished from all such by its cells being of their original strong quality of cellulose, their endwise junctures unaffected, and their physical structure unimpaired by crushing, maceration or breakage.

The surfaces of cleavage left on the elements separated by the invention will be, in some cases, the pure cellulose surfaces of the individual cells in the fibrils, as in Figure 4—D. If the eroding treatment be carried less far, the cell surfaces may retain some of their surrounding cementitious lamellae, as indicated by the rough lines in Figure 4—C, or indeed the process may have been stopped when the original bundle, splitting first at its thinner or weaker places, had as yet separated only into group fragments, each containing several fibrils. In either event the product is spinnable into finer threads than while the original bundles remained undivided. And in either case the distinction is noted, over such fibers as flax, for example, where reduction of the retted product is executed by various methods of bruising and fracture, that the unitary filament produced by the present invention has its cells and connections substantially whole in longitudinal extent, with separation and surfaces made gently by restricted erosion; and that the mass contents are homogeneous in form.

The relative lateral thickness of cementitious matter is greatly exaggerated, in the drawing; and it is to be expected that in ordinary commercial practice substantially the whole of this really very thin substance will usually be removed from the outside of the filamentary units produced by the fibrilizing treatment. Microscopic diametric measurements of fibrils freed by the process of the invention, in certain experiments made under my direction over a wide range of kinds of fibers, have shown figures markedly less than the figures (measured from middle lamella to middle lamella as they lay in the retted fiber) published by previous investigators as diameters of the same kinds of fibers. This indicates that, in the fibrilized product, cementitious matter which was surrounding the cell laterally has been removed.

Comparison for loss of weights indicates the same. In an illustrative instance, the product, being splits as indicated at Figure 4—D, was found to have the same composition chemically as the fiber had before treatment, except that the greater part of the pectic substances were absent; and the fats, waxes and gums were also absent. Before treatment the analysis showed:

	Per cent
Cellulose	76.1
Pectic bodies	11.3
Ligneous matter	1.4
Fats, waxes, gums	5.4
Moisture	5.8

The pectic substances are known by differential staining to be located mostly within the fiber-bundle and between the fibrils, where they constitute the lamellae of cementitious matter; and the fats, waxes and gums are mostly or wholly outside of the fiber-bundle. As left by the retting or decorticating process the fiber of a plant is frequently in units too stiff and coarse to be handled well for spinning, because the fiber units are agglomerations of bundles held together by the gums and waxes. Therefore "degumming" processes of various sorts are used to break these down into fiber-bundles. Such processes, and also processes proposed for breaking a fibre bundle down into its ultimate fibers, if using acid or strong alkali will weaken or injure the cellulose, or the endwise junctures of cells.

The pectic bodies in the removed lamellae have relatively little strength compared with the cellulose of the sclerenchymous cells. They are porous; and thus the eager wetting powers of the soap dispersion are believed to have quick access to the cellulose walls for their protective adsorption thereon. But if the agitative boiling be continued too long the progress of the alkali in degrading the cellulose is observable, with a marked reduction of strength of the cells and the resulting product.

However the boiling treatment may with advantage be continued beyond the stage of erosion of lamellae and separation into individual ultimate fibrils. An extra ten minutes results in the introduction of a new characteristic into the fiber, viz., kinks, represented in Figures 7, 8 and 9, which are comparable in effect to the kink of wool, making the fiber, when spun, hold together better than heretofore, and so be easily spinnable into finer counts of thread. In the case of ramie, Figure 7, this contrasts with ordinary ramie fibers, which without this treatment are characteristically straight and smooth, and difficult for textile use. And from this there results cloth which in some respects closely resembles woolens, having qualities of kink, warmth, and great strength, but being also insect proof and more absorbent of moisture than is wool; and this cloth may be of very light weight. The kinked ramie fiber or threads can be used either alone or intermingled with woolen.

The introduction of kink by this process, by over-cooking, is observed variously:

In a ramie cell 75 there are knobs 76 at intervals; and the kinks 72 arise from bends which develop in the shaft of the cell between the knobs. In jute and hemp (Figure 8) where no knobs are observed in the fibril 80 (made up of cells 85 joined at 81) sinuous forms 82 arise. In the case of these particular fibers, as the length of a cell

85 is of the order of a hundred or a thousand times its thickness, breaks are made in the drawing so that successive fiber junctures 81 can come into the picture with their cementitious material intact. In some other fibers, Figure 9, it appears that knobs are present morphologically in the cell 95, and that they are developed as at 96 by the erosion of the cementitious lamellae surrounding them, corresponding to 13 of Figure 4, instances being seen in istle, sisal, and Sunn hemp. These effects tend to make the respective fibers more spinnable.

Figures 7, 8 and 9, as also Figure 4, have been drawn after careful observation of the working of the invention, and microscopic study, and represent the subject matter as the structure is believed to be.

I do not know the explanation for the development of this kink, but I suggest the following as being probable: The cellulose wall of the fibril cell is uneven as regards thickness, and, when the surrounding cementitious material has been removed by the treatment, water of the solution acquires access to the cellulose wall, which, being a colloid, swells under the access of water and, being uneven in thickness, swells unevenly, which produces contortions. The presence of this kink, however brought about, and the development of protuberances, indicated in Figure 9, which are comparable in industrial results, and may for practical purposes herein be included under the general term of kink, is an extremely important element of industrial utility in that it enables these various kinds of fibers, hitherto relatively smooth, to become strongly inter-engaged when reduced to fibrils and spun, thus making strong threads, even though the count be very fine.

By removing pectins and the pectic lamellae which surround the ultimate cells, the process of the invention incidentally removes whatever is adhering to those lamellae, and this includes the fats, waxes and gums, and any bits of bark, dirt, or oil, on the outside of the fiber-bundle. In so doing it removes matters which attract insects because of their food value or furnish basis for obnoxious small plant growth, as molds and mildews. The absence of food values in the fiber makes the products manufactured from the treated fiber, as clothing, be therefore insect, mold and mildew proof, unless some other insect or plant food be added during later steps of manufacture.

Another effect of removing the lamellae while preserving the endwise junctures of ultimate cells is that it uncovers the color and lustre of the pure cellulose of the walls of the latter. And this, because of the freedom from deteriorative matter above stated, appears to be capable of very permanent continuance. In particular it is not destroyed by laundering, as are "mercerized" effects. But this lustre is not obtained from such parts of the cellulose as remain covered; nor from such parts as become degraded into oxycellulose, by reaction with alkali; or into hydrocellulose if there were ammonia present; or become pitted by erosion of the natural cellulose surface under the alkali attack, which is at work close by in its task of dissolving the pectic bodies. For the successful avoidance of such ill effects in the cellulose, the above described colloidal hydrophilic dispersion of neutral soap is important, for close adsorption selectively by the cellulose, as is also a proper concentration of the alkali at a degree where it will erode the pectic cementitious matter and yet not be strong enough either to coagulate or disrupt the protective film of soap (within the time necessary for the desired operation) or so as to get at the cellulose and attack it effectively (within the time needed for practicable operation). Two of the fibers named have a slightly different material in the walls of their ultimate cells, viz., jute, which has ligno-cellulose and flax, which has pecto-cellulose, but as all or practically all of the others have cellulose, and as the behavior of the cellulose and the pecto-cellulose and the ligno-cellulose is substantially the same, in the matters with which this specification deals, the term "cellulose" is used in the claims to refer to them indiscriminately.

Another effect of converting the fiber to fibrillized form with the cellulose surface of the cell wall exposed is that the remarkable absorptive power of pure cellulose surface for water and aqueous liquids, as human blood, becomes available; and it is observed that the fibrillized product of various plant fibers as jute or ramie, masses of which never heretofore have been considered absorbent, are found to be very absorbent, more so, for example, than standard "absorbent cotton". And likewise cotton, which is a seed hair, responds to the treatment above described for fiber-bundles by becoming more highly absorbent,—and this at a cost which, for the above described treatment, is markedly less than the methods at present customary for making "absorbent cotton". Inasmuch as the methods hitherto known to me for making absorbent cotton have involved the use of caustic alkali and long boiling and no protective colloid for the cellulose surface, I think that the superior results of the process which I have described above, when applied to the seed hairs, may be due to the protection which the colloid renders against attack by mild alkali, in the one case, as contrasted with the defenseless condition of the cellulose surface against the strong attack of caustic alkali in the other case, so that the natural surface may have been degenerated or eaten away, as hitherto prepared.

Another observed characteristic of the fibrillized product is the homogeneity of a mass of the fibers which have been treated together, in the sense that the splits of the fiber-bundles display a marked uniformity of diametric size and of surface and chemical characteristics,—to an extent which renders them distinctive in this respect among bast and leaf fibers known to commerce and industry.

The product of the described treatment is likely to be in most cases merely a step-product, to be followed by other steps of manufacture, as dyeing, spinning, weaving; yet in some cases, as where intended for use as an absorbent, the product of the treatment may be the final form in which the fiber is to be used. Therefore, in the claims the general term "product" is used to refer equally to a mere step-product, or indeed it may be, a piece of woven cloth, for the described treatment for fibrillizing the fibers is applicable whether before or after spinning and weaving, and the same is true of the process when used for cleaning or otherwise treating other plant fibers, as seed hairs.

So also the term "decorticated" is used with general signification to indicate a removal of bark or skin, and of wood, pith, pulp or other unwanted material to which the fibers are connected by nature, regardless of whether the particular method used be fermentative, chemical or mechanical; and this term does not imply a limitation to that stage immediately following the

decorticating, but may include later stages in which the fiber may already have been spun or woven; and even the analogous cases where fibers, as seed hairs, are naturally free from seed, bark, pulp, etc. The decortication need not be complete; and in the case of manila, for example, where the decortication is so imperfect that the fiber is rated only as of low grade, the described treatment nevertheless produces satisfactory fibrillizing results.

The requirement of the process for neutral soap is served by the so-called "neutral soap" of commerce, which sometimes is very slightly alkaline.

The process operates best, in my observation, under a boiling condition of the liquid in the open air; but this situation can be approximated at a temperature somewhat less than boiling in which a strong and universal circulation of the liquid among the fibers is promoted by other means.

The resulting filamentary fragments of fiber-bundles are conveniently said to be "chemically split" because the division occurs on the longitudinal lines, and these filamentary fragments themselves are referred to as "splits" notwithstanding that the division occurs by removal of material leaving a surface of erosion, as distinguished from the more ordinary sense of the word "split" which signifies a longitudinal fracture made by mechanical force of spreading and rending, leaving a surface of fracture.

And the term "catamorphosis" is used, by analogy from terminology employed in geology, to signify the breaking down of the form of the fiber by its splitting into fragments,—by erosion of lateral bonds between cells.

Statements of quality refer to weights, unless otherwise expressed.

Although I have spoken in detail only of treating fiber at the stage after its retting and before its combing, carding and spinning, the invention is not limited to that. It may for example be applied to fiber which has already been woven into cloth; for example, a sheet of ordinary burlap, a stiff fabric woven from jute, is made flexible, soft and absorbent by receiving the above described treatment.

The instances in which the invention introduces new industrial effects and improvements, in the art of dealing with fiber, have been found so numerous and so varied that the describing of the invention requires mention of at least some of them to indicate the widespread and diverse fields in which the invention has already been found useful. Among these are its:

Rendering available for fine spinning certain kinds of fibers, for which only coarse spinning has heretofore been practicable, illustrative instances being found in pita, sisal, manila, New Zealand, and many other fibers which are scientifically known to have valuable qualities but which have not been utilized because of difficulty in spinning. The term "pita" used in this connection refers not only to the forty or so botanical varieties in whose name the word pita occurs, but also specifically to the variety having no botanical name, so far as I know, which is found in the Magdalena Valley in Colombia and has a very long, strong and fine fiber hitherto practically unmanageable.

Producing sub-divided (fibrilized) "fibers" without disproportionate reduction of strength, illustrations being found in hemp, jute, flax, pita, sisal, ramie.

Dealing with difficult fibers at commercially practicable cost, in some cases where cost has heretofore been a prohibitive factor; and utilizing a larger proportion of the raw material, i. e. with less wastage to tow, in the case of some fibers which have heretofore been spun, a feature found particularly valuable with ramie; also with hemp and jute, which, however, have not heretofore been made into fine cloth.

Imparting a pronounced absorbent quality, of the order of absorbent cotton, to some inexpensive fibers hitherto having no such quality; producing also an observable increase of elasticity of the spun or woven product; removing characteristic individual odor such as heretofore has persisted offensively in the unfibrilized fiber, and been subject to regeneration by moist air as in hemp, jute and flax; preserving and bringing out to view a beautiful native lustre of the fiber, in some instances not realized heretofore.

Also, preparing the fiber before bleaching so that it will retain permanently the whiteness produced by the bleaching.

An incident is that it enables the manufacture of the fiber to proceed with fewer steps, e. g. to eliminate the customary washing and souring before the usual bleaching, with saving of costs; and another manufacturing benefit is the rendering of the woven product free from shrinkage, obviating the usual cloth pre-shrinking operation, because the invention incidentally pre-shrinks the fiber.

Other features are the rendering of the fiber immune to insect attack; the softening of varieties called "hard fiber", so that they become notably more flexible and pliable, and when in the mass are softer to the touch; and the introducing of "kink" or equivalent characteristic which makes certain fibers easily spinnable which heretofore have been unspinnable, or spinnable only with difficulty, or without holding power, of which ramie is an illustration.

In some cases there is a substantial reduction of net over-all cost by the incidental production of valuable by-products, an instance of which is in cases where pectic bodies, boiled out, can be utilized.

I claim as my invention:

1. A process for liberation of plant fibrils comprising the immersing of a mass of decorticated natural fiber-bundles in a solution boiling in atmosphere, in which solution to 100% of water there are about .2% to .5% (two-tenths to five-tenths of one per cent) of neutral soap and about .4% (four-tenths of one per cent) of sodium carbonate, the said treatment continuing with erosion of lateral cementitious connections between cells in the fiber until separation of fibers into fibrillized fragments occurs.

2. A process for liberation of plant fibrils comprising the preparing of a solution by putting, into boiling water, of a comminuted mixture of neutral soap about one-half of one per cent of the water, and of sodium carbonate, about .4% (four-tenths of one per cent) of the water; and, when these are well dispersed in the water, immersing in the boiling solution a mass of natural fiber-bundles; and continuing the boiling until by erosion of lateral cementitious connections a splitting of fibers results.

3. A process for liberating plant fibrils, comprising a subjecting of the fibers to an eroding of alkali-soluble cementitious matter, naturally standing between their ultimate cells, by a hot alkali aqueous solution in which the strength of alkali is substantially that of a concentration therein of one-third of one per cent to one-half of one per cent of sodium carbonate, meanwhile

protecting the cellulose of the fibrils, from degeneration by reaction with the alkali, by providing in the same solution an hydrophilic dispersion such as results from the introducing of neutral soap in quantity within the range one-fifth of one per cent to one per cent of the weight of the solution.

4. A process as in claim 3, in which the concentration of alkali is substantially one-half of one per cent of sodium carbonate.

5. A process as in claim 3 in which the alkali characteristic is provided by introducing sal soda in quantity substantially in the range of nine-tenths of one per cent to one and three-tenths per cent of the water.

6. A process as in claim 3 in which the quantity of neutral soap is approximately one-third of one per cent of the weight of the solution.

7. A process as in claim 3, in which the eroding reaction is stopped by removal and rinsing of the fiber at a stage of slimy feeling of the fiber.

8. A process as in claim 3, in which while the eroding reaction is being executed the solution is maintained in vigorous ebullition, is open to atmosphere, and contains the fiber immersed.

9. A process as in claim 3, in which the hydrophilic dispersion is made by putting the soap in finely divided form into water which is already boiling.

10. A process for the rendering of plant fibers more absorbent for aqueous liquids, comprising the boiling of an aqueous solution containing sodium carbonate, of a concentration approximately in the range of one-third of one per cent to one-half of one per cent of the water, in which solution the fiber is immersed, by the alkali of which solution covering matter is removed from the surface of the cellulose of the fiber; and further comprising the protecting that cellulose meanwhile from chemical degeneration by the alkali, by providing in the same solution an hydrophilic dispersion of a substance inert with respect to the cellulose but which substance the cellulose selectively adsorbs in preference to the alkali, such as results from the introducing of neutral soap in quantity within the range one-fifth of one per cent to one per cent of the weight of the solution.

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