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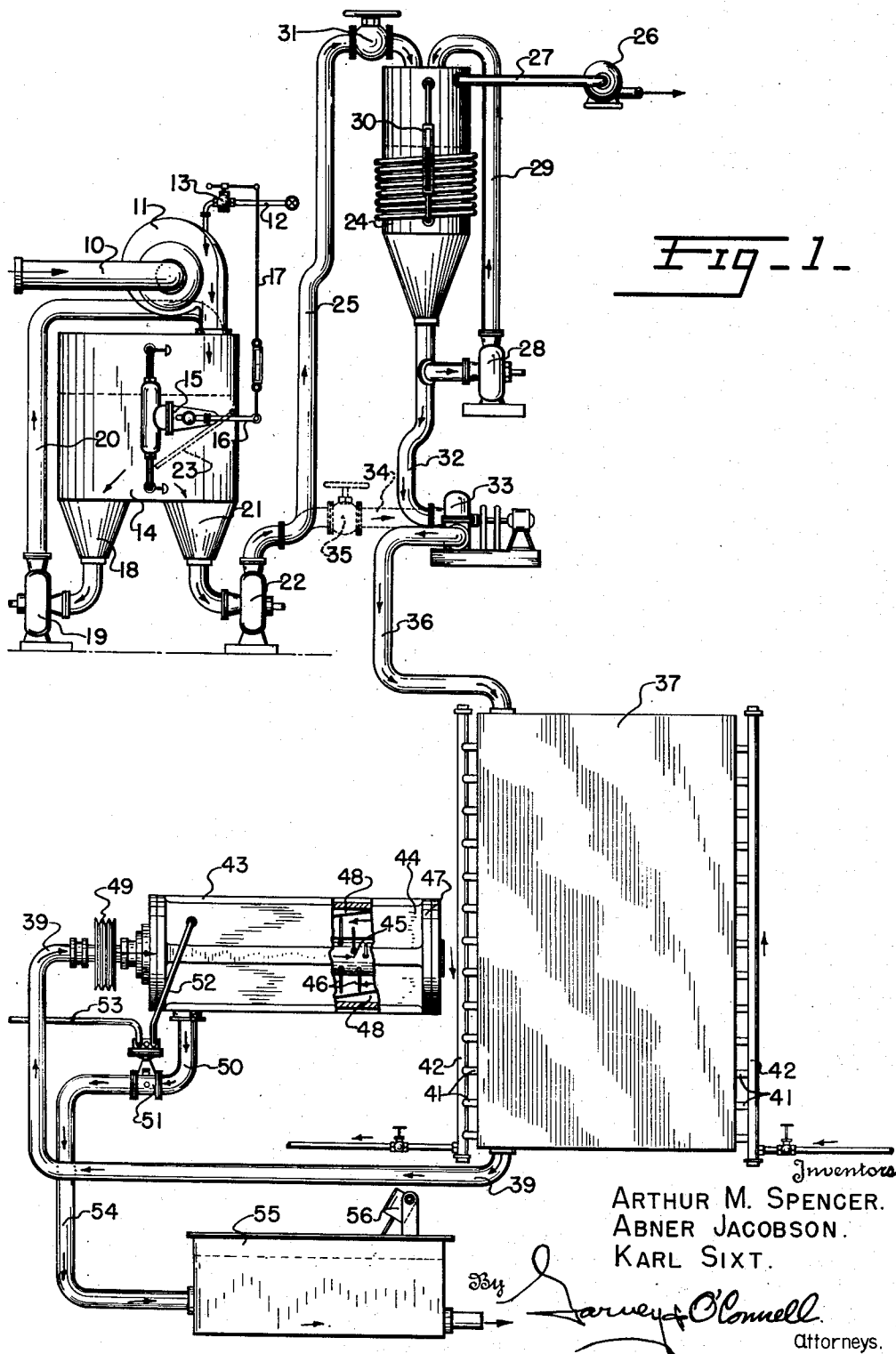
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2,668,110

METHOD FOR FIBER LIBERATION IN COTTON STALKS AND THE PULP

Filed June 18, 1948

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Fig. 3.

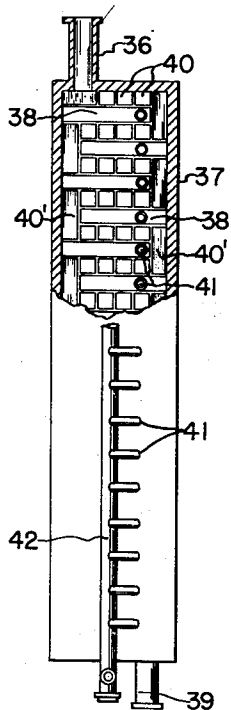


Fig. 2.

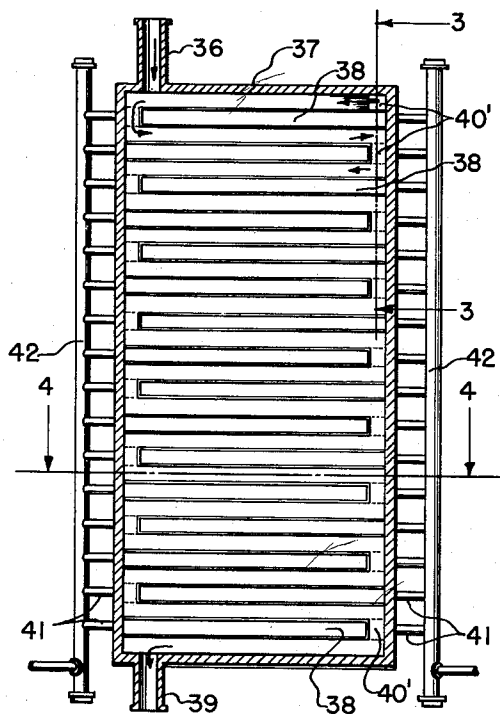


Fig. 4.



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METHOD FOR FIBER LIBERATION IN COTTON STALKS AND THE PULP

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4 Claims. (Cl. 92—6)

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The present invention relates to fiber liberation, particularly the treatment of cotton stalk for continuous rapid liberation with minimum breaking of the fiber thereof into useful products, the effective impregnation of the fiber with organic resinous, bituminous or asphaltic materials to the products in free fiber or matted state, and to apparatus for carrying out the process to form such products.

One of the objects of the present invention is to provide an improved process of digestion of the fiber, particularly cotton stalk fiber, whereby the lignin is rapidly and continuously softened and partially removed and/or softened with a wetting agent containing a minimum of alkaline chemical tending to degrade the fiber.

Another object is to provide an improved rapid digestive process for softening without degradation of the fiber.

Another object is to provide improved fiber liberation, without destruction of the fiber by cutting, tearing and abrasion, by beating of the digested stalks in a whipping type beater to separate fiber bundles into individual fibers.

Another object is to treat the pulp with emulsified organic materials such as resins, waxes, bitumen and asphalt to obtain by coagulation of the emulsion in situ on the fiber a homogeneous distribution and coating thereof.

Another object is to produce improved impregnated moistureproofed matted fibrous products of cotton stalk fiber.

Another object is to provide improved apparatus to carry out the process herein and form the improved products.

Other objects will be inherent in the following description of the invention.

Cotton stalk fiber has not been successfully marketed as commercial matted fiber products, such as wall board, insulation, roofing paper, felt, boxboard, rug liners and other paper type products because prior processes applied thereto, similar to treatment of wood fiber, tended to degrade the fiber both chemically and mechanically in treatments too drastic for the requirements of this fiber and too expensive to compete with other fiber sources.

The present process aims at merely softening the lignin without complete removal thereof and without degradation of the cellulose, and separation of the long fiber from the stalk bundles without mechanical destruction or breaking into short fibers in a rapid, continuous and economical manner.

According to the present invention, cotton stalk

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fiber is digestively softened in the presence of a wetting agent in water primarily adapted to penetrate and loosen the fiber from a normal coating of natural waxes, gums and some of the lignin. The water may also contain a small quantity of alkali which effects some chemical hydrolysis of the natural fiber, but it is preferred to omit alkali and to effect digestive softening with only aqueous wetting agent. Without alkali, some hydration of the cellulose may take place during the digestive softening; however, the present processing of the fiber in total treatment is effected relatively rapidly, so that the final character of the fiber appears to indicate that the primary action of the treatment is a softening of the natural fiber binder and relatively lesser degrees of hydration and, in the presence of dilute alkali, hydrolysis occurs in the treatment. It will be understood that the term "digestive softening" used herein is intended to indicate such primary softening action and partial removal of natural binder substance with secondary, if any, hydration or hydrolysis effects on the fiber.

As wetting agent, we prefer products which have high surface activity as well as good solvent and dispersing action upon the water insoluble natural waxes, gums and lignins associated with the cotton stalk. In general, the wetting agents are fatty derivatives. Secondary, fatty alcohol sulfonation products, esters of fatty alcohols with dibasic acids, and alkyl derivatives of aryl sulfonates and condensates of fatty acids and alcohols with alkylene oxides, are preferred types. Such wetting agents commonly available as Tergitol, Taxad and sodium lauryl sulfonate are suitable. Other common wetting agents, preferably sulfonated types, such as sodium, potassium, ammonium, triethanol amine, piperidine or morpholine salts of sulfated octyl, decyl, dodecyl, tetradecyl, hexadecyl and octadecyl, primary or secondary alcohols, may be used.

For specific description of the process and apparatus, reference is made to the drawings herein, wherein:

Figure 1 shows a general layout of apparatus elements;

Figure 2 is a vertical sectional view of the digester;

Figure 3 is a side elevational view of the digester with parts broken away to show internal construction taken on the line 3—3 of Figure 2; and

Figure 4 is a horizontal sectional view of the digester on the line 4—4 of Figure 2, looking in the direction of the arrows.

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Cotton stalk with or without bolls but without roots and generally without leaves, partially dried from field or other storage, is chopped into suitable lengths and conveyed, preferably in an air conveyor (or other type of conveyor), through duct 10 into the suction of blower 11. Water containing .1 to 5% of wetting agent, such as Tergitol, is drawn from a storage tank (not shown) and passed through pipe 12 with flow controlled at a rate set by valve 13 and sprayed onto the dry chopped cotton stalks emitted from the blower 11. The wetted mass passes into the atmospherically open top of mixing tank 14. The tank 14 is maintained at a fixed level of fluid and stalks by a conventional type float 15 having an arm 16 attached to the float and actuated by the liquid level to throttle the fluid inlet valve 13 through a connecting rod 17. The tank 14 has a double conical bottom, in one cone 18 of which fluid and stalk is picked up by the suction of a pump 19 and recycled to the top of the tank 14 through pipe 20 to secure complete agitation and wetting of the stalk, as well as considerable release of entrapped air bubbles, and in the other cone 21 the preliminary wet and deaerated stalk and fluid is withdrawn by the suction of a pump 22 for passage to the next step of the process.

A baffle 23 is placed in the tank 14 slanting above the outlet cone 21 to divert the incoming and recycle stalk suspension towards recycle outlet cone 18 so that most of the stalk is recycled for thorough wetting and the stalk and fluid which passes into outlet cone 21 has been thoroughly wet by having most of the outlet material recycled and wet by ultimately passing beneath the baffle 23.

It is to be noted that the wetting liquid described here is wetting agent and water. If alkali is to be used, then the wetting liquid, as made up, may further include up to 3%, usually less than 1% of alkali, such as caustic soda or soda ash. The alkali may, of course, be added at any subsequent point up to the digester.

Preliminarily, wet stalk and fluid are passed from pump 22 to a deaerating tank 24 by way of pipe 25 and subjected to a vacuum maintained on tank 24 by a vacuum pump 26 through pipe 27. The tank 24 has a conical discharge bottom and a part of the stalk and fluid is picked up and recirculated by pump 28 and recycle line 29 to the top of the tank 24 to aid in the deaeration. The stalk suspension is regulated to a preselected height in the tank 24, as observed through a sight glass 39 by control of the rate of passage through the tank of the fluid in order to keep the level in the tank below the vacuum outlet into pipe 27 and facilitate gas separation by falling and breaking of the fluid in dropping from both lines 25 and 29 through the top of the tank. The rate of flow may be controlled by throttling valve 31. To enhance deaeration and initiate penetration and digestive softening of the stalk by the wetting agent, the tank 24 may be heated by conventional jacketing or coils for steam or other heat transfer fluid.

The thorough deaeration of the fluid is particularly desirable herein to avoid oxidation and degradation of the fiber since no reducing chemical, such as sulfite, is generally present which would inherently remove oxygen.

The deaeration described is thorough and takes only a fraction up to a minute or two whereby the stalk is thoroughly wetted, deaerated and, with the application of heat, penetration and digestive softening will have begun.

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The preliminarily treated suspension then passes through pipe 32 to a slush pump 33 capable of moving the fluid suspension continuously against the high pressures developed during further digestive softening and against the suction of the vacuum in tank 24.

A line 34 is shown dotted with a control valve 35 which may be used as a by-pass if the vacuum treatment is to be dispensed with in such operations where alkali is used, where the operation is to be speeded up, or where the character of the product desired renders this feasible.

The preliminarily treated suspension of stalk in fluid is passed by high pressure pump 33 into the top of digester 37 by way of pipe 36. In the digester 37, the fluid suspension is passed in continuous flow through a tortuous path over the surface of several heated platens defining heating stages wherein the temperature of the fluid is continuously raised, reaching an ultimate digestive temperature of 250° to 350° F., preferably about 300° to 330° F., developing a pressure of 25 to 150 pounds per square inch. The fluid suspension is passed rapidly through digester 37 so that the fiber is under digestive softening treatment of 5 to 15 minutes, preferably about 10 minutes; more rapid or slower passage through the digester is possible with considerable sacrifice of either economy of operation or the quality of the product for many uses.

The digester 37 is shown in detail in Figures 2, 3 and 4 and generally consists of a ruggedly constructed rectangular tank capable of withstanding the pressures mentioned. The interior of the digester 37 has mounted therein a series of vertically disposed horizontal platens 38, each defining a shelf with alternately disposed openings at opposite sides whereby, after passing over one platen 38, the fluid may descend to the next, passing over each for suitable heat treatment and then passing out at the bottom through outlet 39.

The space defined between each platen is further subdivided by vertical parallel division plates 40 into a set of parallel alleys, alternately open at the end of each alley so that each may communicate with the adjacent alley. Thus, on the surface of a platen 38, the suspension will pass from side to side of a single platen up and back through the communicating alleys and finally drop through an opening 40' in the platen to the next lower platen, thus following a tortuous path in each space between platens, repeating on the next lower platen space. Each platen 38 is internally heated by super-heated steam or other heat transfer fluid supplied thereto and withdrawn by pipes 41 on opposite sides of the digester from large leads and return heater pipes 42 supplied with heating fluid from any suitable source, such as a steam boiler (not shown).

As thus described, the material passed through the digester in a continuous stream is subjected to indirect heat whereby high temperature and pressure is developed in the fluid adequate to soften the cotton stalk fiber bundles for ultimate separation into individual fibers.

The suspension of softened fiber bundles passes under the high pressure and temperature of the digester through pipe 39 to a rapidly rotating beater 43 for ultimate separation of fibers.

The beater 43 forms the subject matter of co-pending application, Serial No. 33,697, filed June 18, 1948, now Patent No. 2,592,481, issued April 8, 1952, and is specifically designed to separate

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fiber by beating with rubber whips cooperating with baffle type walls in a soft impact separation, without macerating, tearing or abrading action, to distinguish from such usual beater effects.

While more detailed construction is described in the copending application, for purposes herein, it is pointed out that the beater comprises an elongated tank of irregular polyhedral straight sides 44, for example, hexagonal or octagonal or eccentric, such as heart-shaped.

The beater tank has mounted for rotation therein an inner shaft-like duct 45 carrying, internally and externally fastened to the wall of said rotating duct, spirally or axially mounted rubber whips 46 for rotation therewith. One end of the rotating duct 45 communicates with pipe 39 to receive digestively softened fiber suspension and pass the same through the interior of the duct 45, rotatively beating the fiber by the flailing action of the rubber whips 46 extending into the duct 45. The duct is open at the end 47 of the tank so that the partially beaten fiber flows out from the rotating duct into the body of the tank, reverses its direction of flow, and passes back over the outside of the duct 45 and continues to be beaten by whips extending from the outside of duct 45 and is thrown against the straight side or eccentric walls 44 of the tank. The passage of the pulp further is baffled by a series of spiral vanes 48 mounted from the walls of the tank. These vanes cooperate with the whips and the throwing effect on the fiber suspension thereby against such baffling to enhance the gentle non-abrasive or non-cutting separation of the fiber bundles into individual fiber. Thus it is to be noted that no abrasive teeth or cutting action, other than flexible whipping and irregular baffling, to stop short the rapidly thrown fibers, is present in the construction described. The duct 45 is rapidly rotated through a pulley 49 driven by a belt from a motor (not shown). The whips used herein are commonly rubber hose and, as these are flexed in opposite rotative effects at various angles, fluid is alternately drawn into the hose and expelled, thus adding to the turbulent movement of the fiber in suspension to aid the ultimate fiber separation.

During the beating, as described, the fluid is still maintained under the heat and pressure of the digester.

It is to be noted that the duct 45 may be rotated in either direction. When the whips are spirally disposed, as well as the baffle vanes, the rotation may be in a direction to give a helical thrust effect, either to aid the normal passage through the beater, or to oppose such passage to give a greater force effect.

The fluid pulp passes out of the beater through a pipe 50 and through a pressure reducing valve 51. This valve is automatically operated by the pressure developed in the beater so as to regulate the flow to maintain the pressure in the beater and digester substantially constant, the pressure control to the valve being communicated through duct 52 with a pilot exhaust of pressure control fluid through duct 53.

The separated fiber now passes at atmospheric pressure through duct 54 into emulsion treating tank 55, conventionally fitted with an agitator 56 for mixing emulsion with the pulp.

For certain purposes the pulp may be otherwise treated, such as by washing and drying.

For purposes of producing water resistant organic emulsion treated pulp, emulsion such as emulsified wax, tar, pitch, resin or asphalt sus-

pending in water in an oil in water type of emulsion with acid, alkaline or salt type of dispersing agents is added and homogeneously mixed with the pulp. Thus a 1 to 5% suspension of 50 penetration asphalt, 70% chlorinated wax, or low temperature coal tar are suitable materials to be applied as emulsions. Various suspending agents such as caustic soda, triethanol amine, petroleum sulfonic acid, sodium lauryl sulfate, are used in forming the emulsion. After thorough mixing of the emulsion and fiber, the emulsion is broken in situ by adding to the tank alkali such as caustic soda, mineral acid such as sulfuric acid, hydrochloric acid, or phosphoric acid or salts, such as sodium chloride or aluminum sulfate, depending on the character of the emulsifying agent, to neutralize or coagulate the same and thus break the emulsion precipitating the organic material in situ on the individual fibers to give fine surface coating thereof.

The following examples illustrate the practice of this invention:

Example I.—A 3% aqueous suspension of 50 penetration petroleum asphalt using sodium lauryl sulfonate as the dispersing agent is added to an individual long undegraded fiber suspension in water of cotton stalk fiber as emitted from the beater described above and stirred to homogeneity. To this mixture of emulsified asphalt and fiber suspension is added dilute sulfuric acid to a pH of 6.5 and the emulsion will break and the asphaltic particles will homogeneously coat the fiber in suspension. The coated filaments are then separated from the water and dried on a Fourdrinier machine or other fiber matting equipment to be formed into such products as water repellent boxboard, wall board, felt, shingles, roofing paper, and other common paper products. Other known alkaline and alkali salt dispersing agents including lime, caustic soda or trisodium phosphate may be used to disperse the asphalt and other mineral acids may be used to neutralize the same to effect coagulation.

Example II.—The method of Example I is repeated, using an acid dispersing agent for the asphalt, i. e., petroleum sulfonic acid and the acid is coagulated by neutralizing with caustic soda or ammonia. Other acids, such as sulfuric, or phosphoric or organic acids such as stearic, or alkyl sulfonic acids may be used as acid dispersing medium.

Example III.—The method of Examples I and II are repeated, using a 3% dispersion of 70% chlorinated paraffin wax to give a water repellent and termite proofed wall board or other building materials or paper products.

Example IV.—The method of Example III is repeated, using phosphoric acid as dispersing agent for the chlorinated wax, and the phosphoric acid is ultimately neutralized to thus precipitate the wax in situ with sodium arsenite to form a salt of the phosphoric acid containing arsenic. The homogeneously coated filaments hereof are waterproof as well as flameproof and thus are an improved building material. The method of this example may be repeated, omitting the chlorinated wax to impart fireproofing salt coating without the water repellancy.

Moreover, various fireproofing salt combinations, such as arsenic and other metal salts with carbonic acid, may be substituted.

The treated pulp of the several examples is ultimately matted into the several products on a Fourdrinier machine into shingles, wall board,

roofing paper, insulation, etc., with or without further lamination.

Various modifications will occur to those skilled in the art and it is intended that the foregoing description be regarded as exemplary and not limiting, except as defined in the claims.

What we claim as new is:

1. Method of liberating the fiber of cotton stalk in its natural undegraded long fiber form comprising suspending chopped cotton stalk in a fluid slurry consisting of water containing as the active digestive component 0.1 to 5.0 per cent of a wetting agent, continuously heating the fiber bundles in said aqueous solution to a temperature in the range of about 250 to 350° F. under substantially the pressure developed at said temperature for a period of about 5 to 15 minutes, and then non-abrasively beating the fiber under the same heat and pressure to gently separate the digestively softened bundles into substantially individual fibers.

2. Method of liberating cotton stalk fiber into their naturally undegraded long fiber as contained in cotton stalk without substantial chemical degradation comprising forming a suspension of small cotton stalk sections in a fluid slurry consisting of water containing as the active digestive component 0.1 to 5.0 per cent of a wetting agent, de-aerating said suspension of the cotton stalk in said liquid, rapidly heating the suspension under the pressure naturally developed to a temperature in the range of 250 to 350° F. for a period of time of about 5 to 15 minutes sufficient to merely soften the fiber and its natural coating of lignin, waxes and resins without substantial chemical attack thereon, and then separating the softened fiber bundles into substantially individual fibers still containing their natural lignin, wax and resin coating by non-abrasively beating the fiber under the same heat and pressure.

3. Method of liberating cotton stalk fiber into their naturally undegraded long fiber as contained in cotton stalk without substantial chemical degradation comprising forming a suspension of small cotton stalk sections in a fluid slurry consisting of water containing as the active digestive component 0.1 to 5.0 per cent of a wetting agent, de-aerating said suspension of the cotton stalk in said liquid, rapidly heating the suspension under the pressure naturally developed to a temperature in the range of 250 to 350° F. for a period of about 5 to 15 minutes sufficient to merely soften the fiber and its natural coating of lignin, waxes and resins without substantial chemical attack thereon, then separating the softened fiber bundles into substantially individual fibers still containing their natural lignin, wax and resin coating by non-abrasively beating the same under the same heat and pressure, and finally homogeneously mixing the fiber suspension with a water resistant binder substance compatible with the natural coating upon the fibers and then separating the binder coated fiber from the liquid suspending medium and forming the same into firmly bonded fibrous sheets.

4. A matted and firmly bonded product in sheet-like form, consisting essentially of natural undegraded long cotton stalk fiber, each fiber

being coated with its naturally occurring coating of resinous, waxy and lignin coating substances and further coated with a water-repellent binder substance to securely adhere the fibers into the sheet-like product, said product being formed by digestively softening chopped lengths of cotton stalk suspended in a fluid slurry consisting of water containing as the active digestive component 0.1 to 5.0 per cent of a wetting agent, heating said suspension to a temperature in the range of 250 to 350° F. for approximately 5 to 15 minutes and under a pressure developed at said temperature, non-abrasively beating said softened fiber bundles to separate the same into substantially individual fibers while suspended in said aqueous medium under the same temperature and pressure at which they were softened, and then adding the water-repellent binder substance to said individual fiber suspension and separating the said binder coated fibers from the aqueous suspending medium and forming the same into firmly bonded sheets.

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