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APPARATUS AND METHOD FOR PROCESSING VEGETABLE FIBERS

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2 SHEETS—SHEET 1

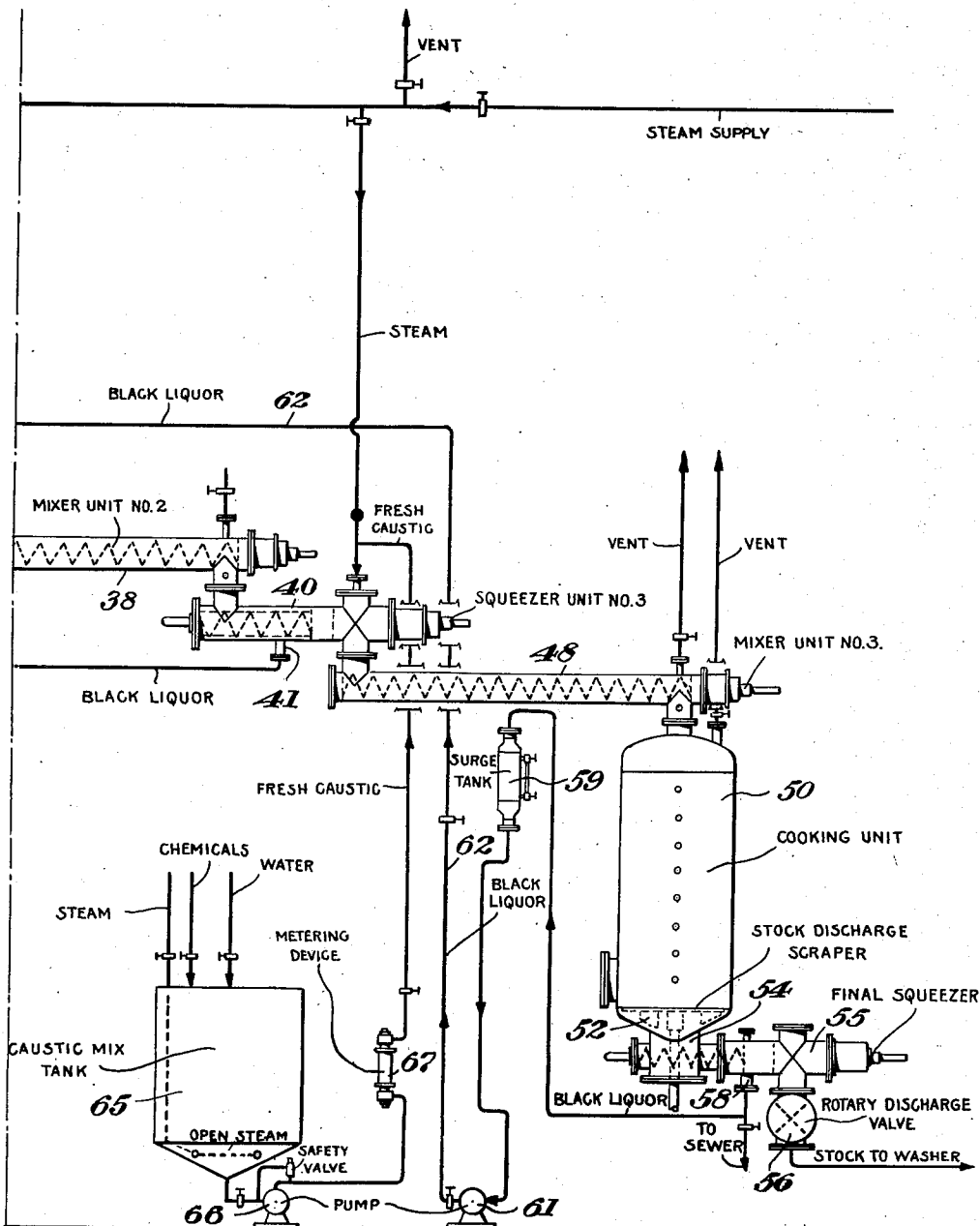


Fig. 1.

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2 SHEETS—SHEET 2

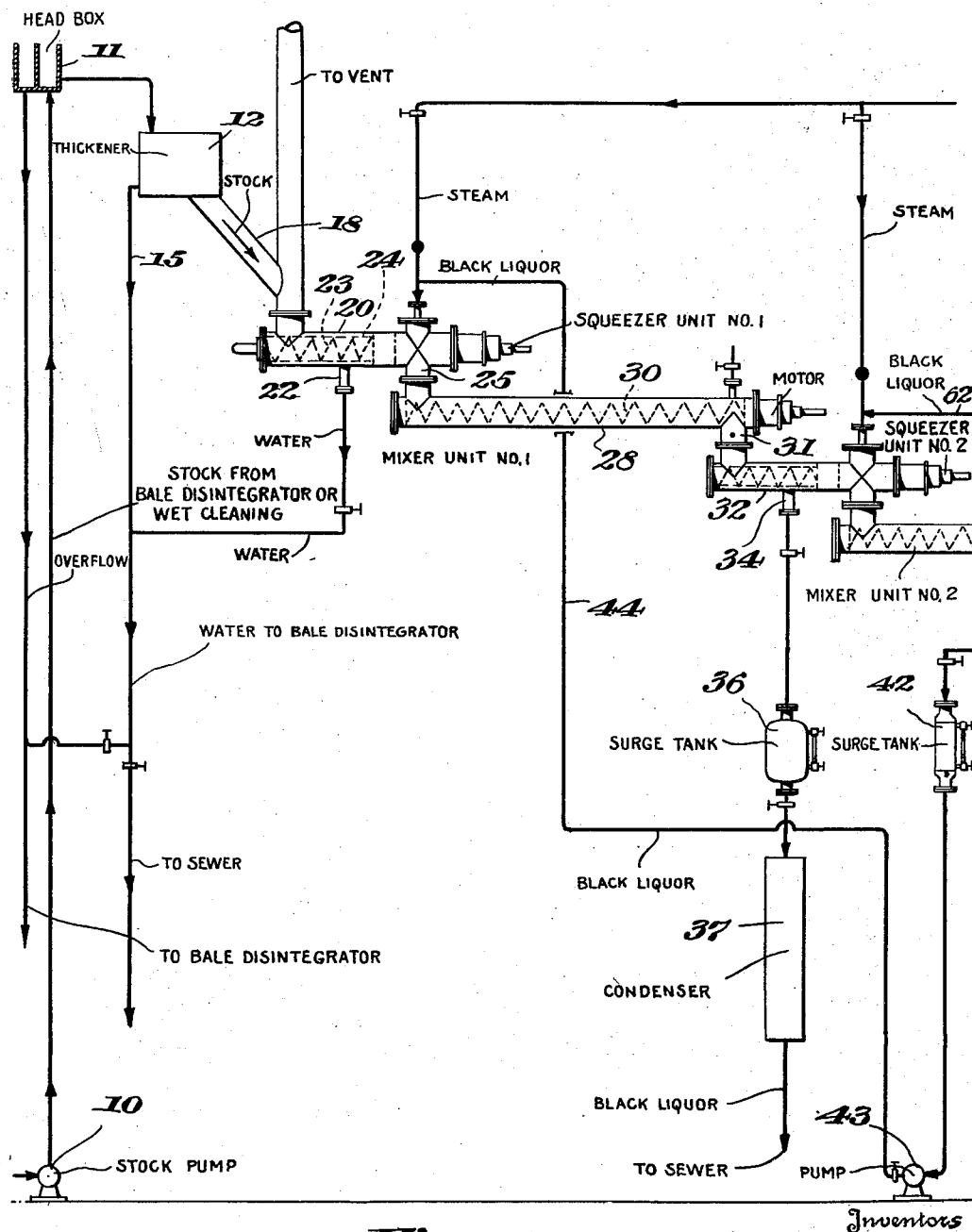


Fig. 1. Clarence B. Weiss
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UNITED STATES PATENT OFFICE

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APPARATUS AND METHOD FOR PROCESS-
ING VEGETABLE FIBERS

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2 Claims. (Cl. 92-7)

1

This invention relates to improvements in the treatment of vegetable fibers to produce a purified cellulose suitable for use in the various fields to which cellulose is adapted, and especially for the preparation of cellulose derivatives. It is an object of the invention to provide an improved process for the treatment of cotton linters and like fibrous material with alkaline agents to remove undesired substances and to alter the physical characteristics of the cellulose, especially with respect to the viscosity of solutions thereof.

In the processing of cotton linters, it is found highly advantageous to perform the alkaline treatment in at least two steps, the treatment conditions being so selected that the effect of the first step is limited principally to the removal of incrusting substances and other impurities, thereby facilitating the subsequent reduction of viscosity by the alkali employed in the final step. It is also desirable to employ substantially fresh alkali in the final step and to use in an earlier stage the partially spent liquor derived from the final step. Such treatment not only has the advantages which might be expected with countercurrent processing, such as more or less complete utilization of caustic, reduction of processing time, and simplification of equipment, but enhances the purity and color of the finished product.

Batch and semi-continuous processes are not readily adaptable to multi-step processing of the character described, and continuous processing embodying the conventional counter-flow principle offers mechanical difficulties owing to the nature of the material being treated. It is therefore proposed, as part of the instant invention, to conduct the alkaline treatment by causing a liquid suspension of fibers to flow continuously through a closed system including two or more treatment zones in which elevated pressures and temperatures may be maintained, the alkaline agent flowing with the fibers in each of the treatment zones, and being expressed from the fibers on discharge from each zone for return and re-use in a preceding treatment zone. For convenience, this method may be described as stepwise countercurrent treatment, the fiber being progressively treated with fresher alkali as the purity of the fiber increases. We have found that in the practice of the invention in this manner, it is possible to exhaust or substantially exhaust the alkaline agent, so that the black liquor withdrawn at the conclusion of the first processing step is practically spent and

2

may be discarded without substantial loss of free caustic.

It is a feature of the invention that most of the suspending liquid is expelled from the stock before introduction of the stock into each of the several treatment zones. Consequently the stock is quickly and uniformly permeated by the treating liquid added to the partially de-watered stock in each zone. This accounts, in large measure, for the exceptional uniformity of product and the substantial reduction in processing time achieved by our method.

It will be perceived that expulsion of liquid under pressure, as described, permits effective treatment of the fibers with a small amount of liquid, capable of absorption by the fibers; such small percentages of treatment liquid are highly economical, but must be expressed from the fibers to allow absorption by the fibers of the liquid in the succeeding stage of treatment.

The squeezing of the linters incident to the expulsion of liquid therefrom is also found to assist greatly in the removal of hull bran and of foreign particles, such as pieces of bolls, leaves, cotton stalk, etc. Thus we have found that while such particles are swollen with caustic, they may be readily broken down by the application of pressure, dissolved in the treatment liquid, and thus removed from the linters. Similarly, waxes and waxlike products may be more readily removed from the fibers if the latter are pressed and twisted while swollen with hot caustic.

Thus by continuously processing cotton linters in the manner described, we have been able to produce cellulose of remarkably uniform viscosity, regardless of substantial variation in the source and character of the linters. Furthermore, we have been enabled to reduce the total time required for treating the linters from a matter of hours to a matter of minutes, and at the same time to effect surprising economies in treating agents. In addition to the saving incident to exhaustion of the alkali, the cellulose produced by the continuous method of the present invention is relatively free from color, and substantial savings in cost of bleaching are effected.

These and other features and objects of the invention may be achieved by the typical practice hereinafter set forth, it being understood that no limitation of the scope of the invention is intended by the use of specific language to describe the same. One embodiment of apparatus, suitable for such practice of the invention,

is illustrated in the accompanying drawings, in which:

Figures 1 and 1a are a diagrammatic representation of a system suitable for the processing of cotton linters and like fibers, Figure 1a being a continuation of Figure 1.

A suspension of linters in water or other liquid is fed by a pump 10 to a head box 11 in which is maintained a constant supply of the suspension, any excess being returned to a bale disintegrator or other preliminary apparatus. From the head box the linters suspension is delivered to a thickener 12, which may be of conventional construction, in which the consistency of the suspension is increased, the drained liquid being withdrawn through outlet 15. Discharge conduit 18 of thickener 12 delivers the partially dewatered stock into the inlet end of a device 20, called for convenience a squeezer, which functions to compress the stock, thereby expelling liquid from the linters, excess liquid being withdrawn through outlet 22 and combined with liquid from outlet 15 of the thickener for return to the bale disintegrator.

The expression of liquid from the fibrous material prior to treatment with the alkaline agent is important to effective practice of the invention, and a squeezer similar to that shown at 20 is associated with each step of the process, releasing entrained liquid for re-use in a preceding treatment zone and delivering to the succeeding zone a concentrated stock from which the impurities dissolved in the preceding zone have been largely removed, such stock being readily dispersible in and capable of reacting quickly with the fresher reagent. While various devices for compressing fibrous slurry to extract liquid are available for this purpose, it is preferable to employ a squeezer constructed and functioning similarly to that shown in our copending application for U. S. Letters Patent, Serial No. 729,056, filed concurrently herewith. In such a squeezer, the discharge of fibrous material is resisted by the frictional drag of the denser material near the outlet end, the compacted fibers plugging the outlet of the squeezer and thereby offering the resistance to movement which is required for expulsion of liquid from the incoming stock. The fibrous plug also functions as a barrier to prevent discharge of steam from the system, as hereinafter explained.

Thus as shown schematically in the accompanying drawings, the stock is fed by a screw conveyor 23 from the inlet end toward the outlet end of the squeezer, the screw working in a perforated cylinder 24 through which liquid may be expressed. The conveyor terminates short of the rear end of the cylinder to permit the formation adjacent the squeezer outlet of a plug of compacted fiber, this fibrous plug being gradually extruded at the outlet end and discharging downwardly through outlet conduit 25, the plug being continually replenished by the feeding of fresh fiber against its forward face. A picker device (not shown) effectively disintegrates the fibrous plug, eliminating knots or lumps, facilitating the formation of a suspension in the succeeding treatment zone, and promoting intimate contact between the treating agent and the individual fibers.

From squeezer 20 the fibers are discharged into an elongated casing defining a cooking chamber, called for convenience a mixer 28, affording the first of a series of at least two treatment zones,

three such zones being employed in the illustrated embodiment of the invention. Partially spent liquor derived from the output of a succeeding treatment stage or zone, constituting the alkaline agent, and steam under pressure, supplied through steam pipes for establishing a predetermined temperature in the first treatment zone, are admitted to squeezer 20 adjacent the outlet end and beyond the fibrous plug, and thence into mixer 28.

The mixer employed in the first and in any subsequent treatment stage may be constituted by a cylindrical compartment or casing having a screw conveyor 30, whereby the stock or suspension is agitated while being fed continuously from the inlet toward the outlet end of the mixer, the conveyor being so constructed and designed that the material remains in the mixer for the requisite period of time. This period may vary widely, but is preferably chosen, with due regard to other processing conditions, so that the operation in the first mixer is substantially limited to the removal, or partial removal, of incrusting materials and the like.

At the discharge end of the first mixer 28, the partially purified material is discharged through conduit 31 into a second squeezer unit 32, which may be similar in structure and function to squeezer 20 hereinbefore described. The liquid issuing from outlet pipe 34 of squeezer 32, comprising completely spent black liquor, flows through a conduit comprising surge tank 36 and condenser 37, and may then be discarded. A pressure relief valve is located in the passage connecting the surge tank with the condenser. The fibrous stock issuing from squeezer 32 is discharged into the inlet end of second mixer 38. Partially spent liquor from the succeeding stage, and steam for establishing the desired conditions of temperature and pressure, are admitted to mixer 38 through the outlet end of squeezer 32 as hereinbefore described with reference to mixer 28 and squeezer 20.

The stock is fed continuously in mixer 38 from the inlet toward the outlet end, for example by means of a screw conveyor, and is discharged at the outlet end of the mixer into a third squeezer 40, constructed similarly to the squeezers hereinbefore described. The treatment applied to the fiber in mixer 38 is essentially similar to that effected in mixer 28; the purification of the fiber being continued substantially to completion by the use of alkali of greater strength and freshness than that employed in the first stage. The partially spent liquor discharged from outlet 41 of squeezer 40 is passed through a surge tank 42 and a pump 43, being returned to the inlet end of mixer 28 by pipe 44. The squeezed stock, together with fresh caustic and steam is discharged from the outlet end of squeezer 40 into a third mixer 48 which constitutes part of the third and final treatment zone. As hereinbefore explained, the conditions established in the final zone are conducive to more intense or more extensive treatment and are so selected as to alter and improve the characteristics of the fiber, adequate purification having been effected in the preceding zone or zones by the use of the partially spent liquor derived from the final zone. For instance, a substantially higher temperature may be maintained in the final zone, whereby reduction of viscosity of the cellulose product is facilitated. We prefer, however, to supply steam of uniform pressure, within a range of 60 to 200 pounds per square inch to each zone, and to pro-

5

vide for a substantially longer period of treatment in the final zone. Thus if the period of treatment in each of the earlier zones is 6 minutes, the material may remain in the final zone for about 10 minutes, giving a total time for treatment of 22 minutes for the three zones. Treatment times may be easily regulated to establish satisfactory processing conditions by adjustment of speed of the screw conveyors in the several mixers, and by interrupting the conveyor flights to retard the feeding rate, in the known manner.

The minimum treatment time in each of the purifying zones should not be less than one minute. Treatment for about 4 minutes in each purifying zone is usually sufficient in a system employing two such zones and a final viscosity regulating zone. Treatment for a longer period of time will not adversely affect the quality of the pulp, but may unnecessarily increase consumption of caustic. In the final or viscosity regulating zone, at least 5 minutes should be allowed, the maximum period being about 90 minutes. The treatment periods will, of course, vary with change in caustic concentration and temperature, and it is necessary to correlate these several factors to obtain optimum results.

The initial supply of alkali, as introduced into the final stage in solution in an appropriate amount of water, will be determined largely by the grade of the linters being processed and the amount of impurities contained therein. Thus 100 pounds of linters may require from 5 to 12 pounds of sodium hydroxide for adequate purification and viscosity reduction, 6 to 8 pounds usually being adequate for grades of linters most commonly treated. Excessive caustic usage over that used up in the chemical reactions which occur is obviously uneconomical, whereas an inadequate caustic usage will not result in adequate purification of the linters. Thus for proper viscosity control in processing a given grade of linters, it is necessary to correlate only the factors of time and temperature, increasing either factor effecting greater reduction in viscosity in the final stage and promoting the purifying action in the earlier stages.

To provide for an extended treatment time in the last zone, mixer 48, which may be constructed similarly to the preceding mixers, discharges into a cooking unit 50, having a capacity substantially greater than the capacity of the mixers, mixer 48 and cooking unit 50 affording in effect a continuous treatment chamber having a capacity substantially greater than that of the preceding chambers or zones. By altering the level of material in unit 50, the treatment time may easily be regulated; this may be effected by controlling the rate of withdrawal of materials from the unit for example by adjusting the speed of operation of the final squeezer 55. The cooking unit may comprise an elongated tank or cylinder through which the stock moves continuously by gravity, and in a relatively quiescent state, being fed into the upper end and withdrawn from the lower end of the unit. To facilitate withdrawal of stock, a scraper or other conventional discharging device 52 may be provided in the lower end of the cooking unit. It will be noted that the conjoint use of mixer 48 and cooking unit 50 in the last processing stage contributes materially to the production of pulp of uniform viscosity, the mixer insuring effective contact of the agent with the fibers to initiate the reaction promptly and uniformly, and unit 50 providing for accurate adjustment of the duration of the reaction.

6

The stock is delivered to the final squeezer 55 through the outlet conduit 54, and thence through a rotary discharge valve 56 to storage for subsequent processing. The black liquor discharged from outlet 58 of squeezer 55 is returned through a surge tank 59 and a pump 61 to supply line 62 for delivery into the inlet end of mixer 38 of the preceding stage, as hereinbefore described.

The alkaline agent used in the last treatment stage is prepared in a caustic mix tank, in which the alkali is diluted with water to the desired concentration, and in which additional chemicals, such as wetting agents and the like, may be introduced. The materials fed into the mix tank are suitably heated, preferably by steam, and the mix is delivered through a pump 66 and a metering device 67 to the inlet end of mixer 48 of the final stage. Any alkaline agent of a concentration suitable for the treatment of vegetable fibers may be employed, the commonly used sodium hydroxide being the preferred agent. By way of example, we may use as the agent 2.5% caustic solution in amount sufficient to establish a liquor to lint ratio of about 2.4:1 to 3.2:1 in the several treatment zones. Concentration of caustic is usually reduced in the last zone to about 1.0% to 2.0%, and in the second zone to about 0.5% to 1.0%; the liquor discharged from the first zone will preferably contain not over a small fraction of 1% of caustic.

These and other conditions and factors involved in the practice of the invention may vary widely in accordance with prior practice in this field, provided that the selected conditions are such as to promote purification of the fiber in the preliminary treatment zone or zones, and improvement of fiber characteristics, including viscosity, in the final zone.

It will be observed that the process is continuous throughout, and that it lends itself particularly well to the production of cellulose of uniform viscosity. Thus, on initiating treatment of a batch of cotton linters, the viscosity of the final product may be determined from a sample, regulation of the viscosity being readily effected by adjustment of operating conditions in the final treatment zone, preferably by varying the time of treatment in that zone, as hereinbefore indicated. Any conventional method of determining viscosity may be employed, for instance the method described in detail in "Industrial and Engineering Chemistry," vol. 23, page 136, 1931.

For example, an average batch of linters, processed in three steps for periods of 4 minutes, 4 minutes, and 30 minutes, respectively, employing in the last step a 1% caustic solution and a liquor to lint ratio of 7.5:1, the steam pressure in each stage being 100 pounds per square inch, will yield cellulose having a viscosity of the order of 3,000 seconds. If the steam pressure is increased to 200 pounds per square inch, the viscosity of the cellulose will be reduced to about 100 seconds. However, if the processing time in the final stage be decreased from 30 minutes to about 6 minutes, and steam at 200 pounds per square inch be employed, the final viscosity will be increased to 3,000 seconds. It is thus apparent that the viscosity of the final product may be varied over a wide range by appropriate selection of processing conditions, and that these conditions may be quite readily adjusted to yield any desired product.

It will be appreciated that satisfactory practice of the invention may be effected with the use of only two stages, or that the number of puri-

fyng or preliminary stages may be increased. Benefits of the invention are obviously not realized, however, unless there are provided at least two treatment zones, the treating liquor being withdrawn, at least in large part, from the fibrous material at the discharge end of each such zone for return to a preceding zone or, in the case of the first zone, to waste.

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

1. In apparatus for use in the continuous alkaline treatment of cotton linters in the preparation of cellulose, a closed system comprising a plurality of elongated, generally horizontal cooking casings affording a succession of alkaline treatment zones, each casing having an inlet passage and a remotely located outlet passage, a conveyor in each casing for causing a suspension of linters in an alkaline liquid agent to flow continuously through each of said casings in succession from the inlet to the outlet passage thereof, a cooking tank affording a final alkaline treatment zone, having an inlet passage and a remotely located outlet passage, a squeezer associated with each outlet passage for receiving and continuously compressing discharged linters to expel most of the liquid therefrom, each squeezer save that associated with the cooking tank communicating with the inlet passage of the succeeding casing to deliver thereto compressed linters, conduits continuously delivering expressed liquid from each squeezer save that associated with the first casing to the inlet passage of a preceding casing, a conduit continuously delivering to the inlet passage of the cooking tank a fresh alkaline liquid agent, whereby the linters are treated in the successive casings and cooking tank with a liquid agent of progressively increasing freshness, and steam pipes introducing in each of said casings and said cooking tank steam at a pressure of from 60 to 200 pounds per square inch, said cooking tank having a capacity substantially greater than that of the preceding casings, whereby the treatment time in said cooking tank is substantially greater.

2. In a process for the alkaline treatment of cotton linters in the preparation of cellulose, the steps which comprise feeding a slurry of cotton linters in a liquid into and through a compression zone, continuously mechanically compressing the linters in said zone to an extent sufficient to expel therefrom most of the liquid and to form the linters into a dense plug, continuously extruding the fibrous plug so formed from the compression zone, disintegrating the plug as it is extruded, and discharging the fibrous particles derived from the plug into a first alkaline treatment zone containing an aqueous caustic solution, mixing the solution and the fibers to effect uniform impregnation of the fibers with the solution while continuously feeding the fibers through and out of said first treatment zone, maintaining in said first treatment zone a temperature corresponding to that of saturated steam of from 60 to 200 pounds pressure, delivering the fibers

from said first treatment zone into a second compression zone, compressing the fibers in said second compression zone to an extent sufficient to expel therefrom most of the solution entrained in the fibers and to form the fibers into a dense plug, continuously extruding the fibrous plug so formed from the second compression zone, discharging the fibrous particles derived from the last named plug into a second alkaline treatment zone containing an aqueous caustic solution, mixing the solution and the fibers to effect uniform impregnation of the fibers with the solution while continuously feeding the fibers through and out of said second treatment zone, maintaining in said second treatment zone a temperature corresponding to that of saturated steam of from 60 to 200 pounds pressure, delivering the fibers from said second treatment zone into a third compression zone, compressing the fibers in said third compression zone to an extent sufficient to expel therefrom most of the solution entrained in the fibers into a dense plug, continuously extruding said last named plug from the third compression zone, and continuously returning the solution expelled from the fibers in said third compression zone to said first alkaline treatment zone to provide the aqueous caustic solution therein.

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