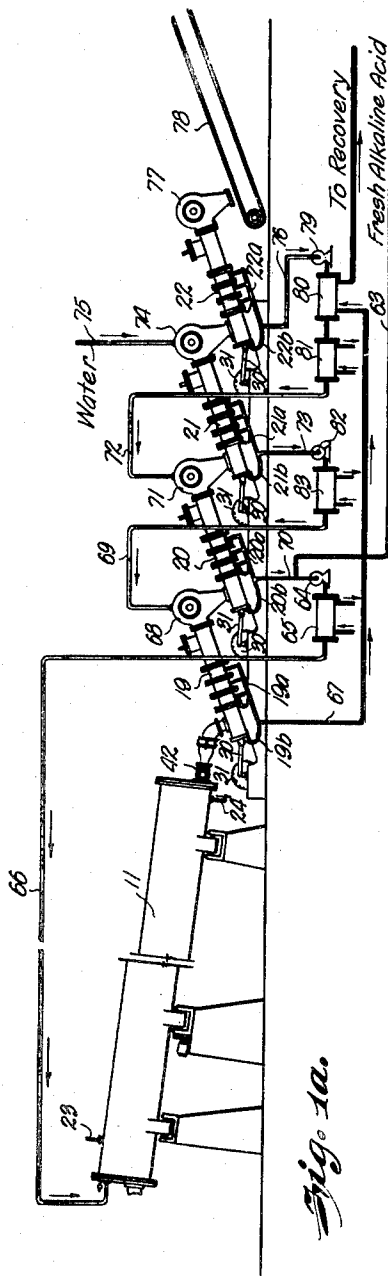
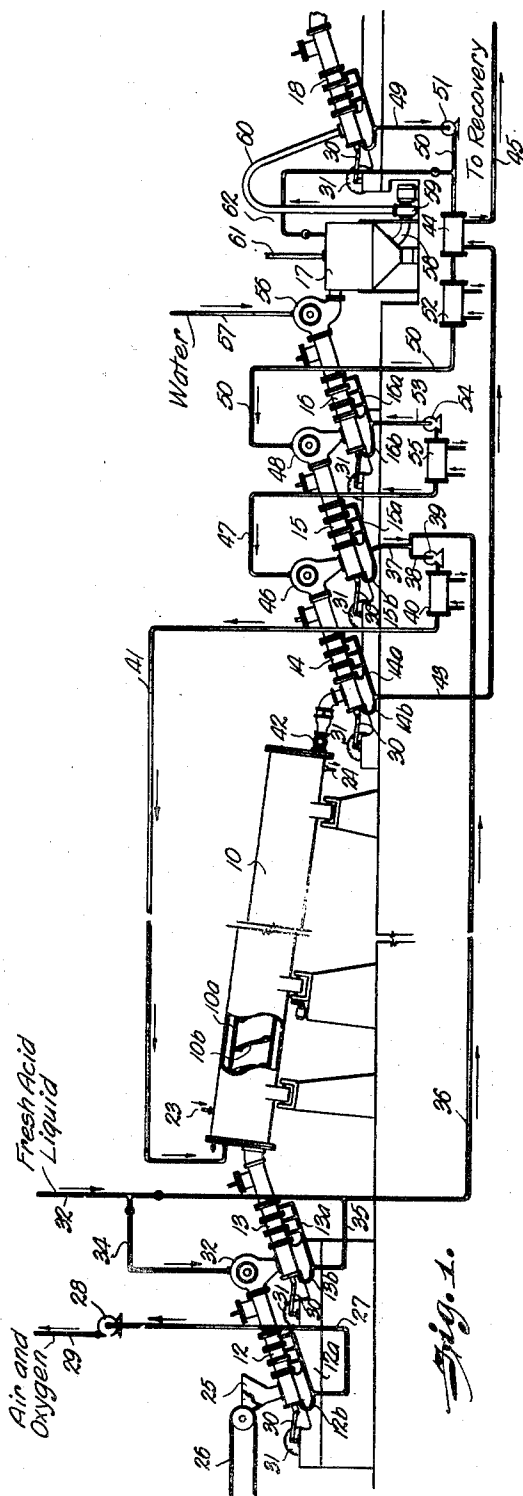


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CONTINUOUS DIGESTION APPARATUS FOR THE PRODUCTION
OF HIGHLY PURIFIED CELLULOSE
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CONTINUOUS DIGESTION APPARATUS FOR
THE PRODUCTION OF HIGHLY PURIFIED
CELLULOSE

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This invention relates to improvements in the continuous digestion of cellulosic-bearing materials for the recovery of a highly purified cellulose. The basic concept resides in a two-stage digestion apparatus, the first stage acid and the second alkaline, accompanied by a preliminary stage of air removal, liquor saturation and moisture control, an intermediate stage of acid removal accomplished preferably by filter pressing and highly agitated washing, and a final stage of filter pressing and washing to remove the alkaline liquor. The material undergoing digestion throughout most of its travel is kept at a medium consistency or in a relatively dense mass while being impregnated with treating liquors by rotation of the digesters, and at very high consistency liquor extraction by the pressure imposed upon the mass or mixture in the washing and filtering presses. The apparatus, while developed and applicable particularly to the digestion of bagasse, is equally useful for the recovery of cellulose from other materials.

An object of the invention is to provide an apparatus in which a treating fluid is uniformly dispersed throughout the mass of cellulose-bearing material while air and oxygen-containing fluids which would cause objectionable oxidation in the acid digester are removed therefrom.

Another object is to provide an apparatus through which the saturated mass passes including successive digesters consisting of inclined concentric vessels, the inner one rotating, the outer stationary, and an internal open screw affixed to the inner wall of the rotating vessel whereby the volume of the inner vessel, the rate of rotation, the pitch of the screw and the degree of inclination of the digesters determine the length of the digestion period.

A further object is to provide an apparatus in which are interposed a plurality of filter press units for washing and extracting treating fluids from the fibrous mass while maintaining the same at a relatively high consistency and at substantially the temperature and pressures of digestion.

Other and further objects will appear from the following description.

Fig. 1 and Fig. 1a show a side elevational view of a continuous apparatus which has been separated centrally in order to include the entire mechanism on a single sheet of drawing.

The apparatus is divided for convenience in the description into its essential parts which comprise an acid digester 10, an alkaline digester 11, a charging unit consisting of presses 12 and 13, intermediate presses 14, 15 and 16, a dissolving tank

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17, charging press 18, and a plurality of terminal presses 19, 20, 21, and 22.

The structure of digesters 10 and 11 is preferably the same and consists of two concentric cylindrical shells inclined from the horizontal to facilitate passage of material therethrough. The outer shells are stationary, the inner shells 10a rotatable and have attached to their inner walls open screws diagrammatically shown at 10b in digester 10 which tumble the mass with rotation of the inner shell and cause it to advance along the length of the digesters. Between the inner and outer shells are fluid tight seals which form an annular jacket surrounding the inner vessels and through which a heating medium is passed to control the temperature of treatment. This medium is introduced to the jackets through pipes 23 and is discharged through pipes 24. Cellulose-bearing material, after its moisture content has been adjusted to substantially 50%, is introduced to the charging hopper 25 at the left end of Fig. 1 by means of a conveyor belt 26. The mass of material is charged from the hopper through the press 12 where it is relieved of oxygen-containing fluids such as air and the like which are exhausted from the press through pipes 12a, 12b, pipe 27, pump 28 and vented to the atmosphere through pipe 29. The construction of the presses 12 and 13 for charging material to the first digester, the intermediate presses 14, 15 and 16, charging press 18 for the second digester, and the terminal presses 19, 20, 21 and 22, is of the same general character. They consist of a tubular body or shell in which a piston is reciprocated by means of connecting rods 30 reciprocated from a power source diagrammatically shown as gear wheels 31 or screw feeds may be substituted for the pistons. The purpose to be accomplished is to compact the material to form a dense plug and advance the plug into the inner rotating vessel. The shell portion of the presses is either constricted toward its discharge end by tapering or the size of the discharge opening of the press is reduced by an adjustable valve. Slotted openings in the walls of the presses and in the case of reciprocating presses, also apertures in the faces of the pistons permit egress of the fluids pressed from the mass as the consistency and density of the material is increased. These extracted fluids are withdrawn from the presses through pipes 12a, 12b, 13a, 13b, etc. After extraction of the air in the first press 12, the material is discharged into a shredder 32 where a rotating wheel breaks up the congested material and intimately mixes therewith fresh acid supplied to the shredder from

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any convenient source through pipes 33 and 34. The fibrous mass saturated with acid is then passed through press 13 into the inner shell of digester 10. The congested plug of material in press 13 affords a pressure-tight closure at the charging end of the digester. Excess fluid extracted from the material passing through press 13 drains off through pipes 13a and 13b through line 35 and pipe 36 to the intermediate presses at the discharge end of the digester as hereinafter explained.

Saturation with acid is not necessary. There may be cases where the material may not be saturated with acid prior to entering the inner chamber, saturation taking place readily and rapidly within the chamber.

Acid-saturated material introduced into the digester is tumbled by rotation of the inner shell and advanced during rotation by the open screw 10b at a uniform rate of travel throughout the length of the digester. The rate of rotation and the length of the digester, as well as temperatures within the vessel and the concentration of liquor produce loosening and separation of the incrustants from the fibers. As an additional source of treating liquor to the digester 10 acid passing through pipe 36 is combined with diluted acid withdrawn from press 15 through pipe 37. The components are combined in pipe 38 and are charged by pump 39 through heat exchanger 40 and pipe 31 to the inlet end of the digester where they are mixed with the cellulose-bearing mass introduced thereto by press 13.

After digestion with the acid the mass is discharged through a valve 42 into press 14. In this press a substantial portion of the liquid content of the digested mass is removed by increasing its density. The liquids separated from the solids in the press are drained out through pipes 14a and 14b to a common withdrawal pipe 43 thence through heat exchanger 44 and pipe 45 to a recovery system not shown. In the recovery system the chemical is extracted from the liquor and returned to the digester for reuse.

After preliminary extraction of the liquid in press 14 the congested mass is again broken up in repulper or shredder 46 where it is intimately mixed with dilute acid supplied through pipe 47. This mixture is passed through press 15 where a considerable portion of the fluid is again separated from the solids, the former withdrawn through pipes 15a, 15b and pipe 37, the latter broken up in shredder 48 and again mixed with dilute acid supplied from press 18 through pipes 49 and 50 in which are interposed pump 51, heat exchanger 44 and heat exchanger 52.

The mixture of dilute acid and cellulose-bearing material is then charged to the final press 16 of the intermediate series or washing presses where it is again compacted to extract the liquid. From this press the liquid is withdrawn through pipes 16a, 16b, pipe 53, and is charged by pump 54 through heat exchanger 55 and pipe 47 to shredder 46. The congested solids discharged from the press are broken up in shredder 56 and are there mixed with water supplied through pipe 57. After mixture with the water the acid digested solids pass to a dissolving tank 17 where a separation is made of the incrustants and solids by high speed agitation. Acid-treated pulp is withdrawn through pipe 58 and is directed by the motor driven centrifugal pump 59 through line 60 to press 18 by which it is charged to the digester 11.

Tank 17 is equipped with a gas vent 61. Also

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between tank 17 and pipe 50 is a by-pass line 62 controlled by suitable meters and valves which enable recirculation of sufficient liquor to keep the discharging consistency from the dissolver 17 within pumping range.

With the acid-treated mass of semi-pulp introduced to the digester 11 is charged an alkaline liquor such as sodium hydroxide. This is pumped from a convenient source through pipe 63, pump 64, heat exchanger 64 and pipe 66 which connects to the inlet end of the digester. In the digester the alkaline liquor and partially digested pulp are intimately mixed by rotation of the inner shell and the action of a screw corresponding to that shown at 10b in digester 10. The function of this screw is to advance the mass slowly from one end of the digester to the other as the inner shell rotates, affording the necessary time period for reaction of the treating liquid with the solids. Upon discharge from the second digester through a valve 42 the mass is congested in a filter press extractor 19 from which the separated liquid is withdrawn through pipes 19a, 19b, and pipe 67. The congested solids are separated in shredder 68 and are there mixed with dilute alkaline liquor supplied through pipe 69. The mass is again extracted in press 20, liquids being withdrawn through pipes 20a, 20b and line 70, the solids again broken up in shredder 71 and mixed with dilute liquor supplied through pipe 72. The liquor extraction is again performed in press 21 from which the liquid is withdrawn through pipes 21a, 21b and pipe 73 while the solids are passed to shredder 74. In the shredder 74 the digested pulp is combined with water introduced through pipe 75 and the mixture is directed to the final press 22 from which the liquid is drawn off through pipes 22a, 22b, and pipe 76, while the solids pass to shredder 77, thence to conveyor belt 78. The dilute alkaline liquor withdrawn from press 22 is recycled through pipe 76, pump 70, heat exchangers 80 and 81, pipe 72 to the shredder or repulper at the discharge from the second terminal press 20. Fluids withdrawn from press 21 through pipe 73 are recycled by means of pump 83 through pipe 69 to shredder 68 of press 19. Fluids withdrawn from press 20 through pipe 70 are returned as previously described through pump 64 to the inlet end of the digester.

Thus it will be seen that in both the intermediate washing presses 14, 15 and 16 between digesters 10 and 11 and in the terminal presses 19, 20, 21 and 22 diluted fluids are passed counter-currently to the direction of flow of the mass of solid materials which have undergone treatment in the separate digestion stages.

Having described generally the apparatus and the manner in which it functions, the processes practiced therein will be briefly described. While the apparatus as suggested was designed primarily for the treatment of bagasse which is a fibrous product in the manufacture of sugar from sugar cane, it can be used as well in the treatment of other types of vegetable fiber including non-resinous woods, such as cottonwood, aspen, birch, beech, chestnut, gum, elm, maple, ash and the different types of oak or coniferous woods such as spruce, pine, fir, hemlock and cedar. Many of these woods contain objectionable pitches or resins. Besides sugar cane, seed hair fibers, leaf fibers, fruit fibers or grass fibers such as ramie, bamboo, wool, flax, hemp, jute, nettle, corn straw, esparto sisal, pineapple, palm, banana, and coconut, to mention but a few, may be used as charging material. The apparatus is particularly

adaptable for the pulping of plants containing fibro-vascular bundles and parenchyma or pith cells.

In brief, materials to be pulped in the apparatus are initially given a uniform treatment with an acid solution in a relatively inert, reducing or non-oxidizing atmosphere after which the treated materials are flowed countercurrently in a multistage washing and defibering operation. The fluid is then extracted and the material digested with an alkaline solution. After the second digestion with the alkaline solution it is again washed countercurrently to remove the treating liquor after which it may be bleached, screened, riffled and dewatered for use in any type of high grade cellulose products.

Attempts have been made to treat vegetable fiber containing cellulose in continuous multistage operations to obtain a product having a highly pure cellulose content. Such methods have had only a limited degree of success due to unequal water content, non-uniform operation, difficulties encountered with undissolved incrustants present in the fiber, incomplete washing and defibering and attempts to bleach after the acid step instead of delaying the bleaching operation until after the alkaline step. By following the chemical digestion steps with countercurrent multistage, high consistency washing and dewatering operations, greater overall efficiency, higher quality and better uniformity in the final product is obtained.

The cellulose obtained by processing materials in this type of apparatus is adaptable for use in the manufacture of many products owing to its exceptional uniformity, purity and high white color. Among such uses is the production of better quality of cellulose derivatives including explosives, artificial silk, pyroxylin products, photographic and other types of films, plastics and the like.

Generally speaking the method includes an initial treatment of the vegetable fibers to produce within the stock to be treated a uniform water content (substantially 50%). The stock is then treated with a mixture of water and sulfur dioxide in the first digester 10 in the absence of free oxygen or air while maintaining the mass under medium to high densities. The temperature during this initial acid digestion is closely regulated by the circulation of a heating medium around the inner rotating vessel preferably between 98° C. and 150° C., depending upon the liquor ratio, chemical concentration, materials used and other varying factors. A temperature of 110° C. to 130° C. and liquor ratios of 4:1 to 7:1 employing acid of 1.003 to 1.005 specific gravity at 15.5° C. are usual. Pressures within the digester are substantially above atmospheric and normally range from 40 to 200 pounds, depending upon the liquor ratio, concentration of sulfur dioxide gas and temperature.

It is important that as much as possible of the air or free oxygen in the charging stock be removed prior to the acid treatment. This is accomplished by compressing the material in charging press 12 into a dense mass and evacuating the air with a vacuum pump as shown at 28, or in any other suitable manner, before charging the stock to the digester.

The initial acid treatment is preferably carried on as a continuous step, the charging stock after removal of the air or oxygen is introduced at one end of the rotating vessel and discharged at the opposite end. The travel through the

digester is determined by the length of time necessary to obtain adequate acid treatment. The digester in which the acid treatment takes place, as previously suggested, is equipped with an internal screw or other means for advancing the materials slowly and uniformly in order that thorough mixing of the liquor and fiber is obtained, and the material advanced continuously from the inlet to the discharge end during acid treatment. After treatment with the acid the material is thoroughly washed in presses which make up the intermediate multistage washing operation. Diluent liquid in this washing operation is passed countercurrently to the flow of the acid-treated fibers to remove therefrom the acid, sugars, incrustants and other contaminating substances. Cases may arise in the treatment of certain types of stock where the plurality of intermediate high consistency presses between the two digestion stages are replaced by low pressure or vacuum filters where the stock contains an excess of fluid gummy or resinous substances not easily removed by presses of this type. A dissolving tank or blow tank with the ordinary vacuum or conventional type washers may in such cases prove more satisfactory than the high consistency presses. The important factor to avoid is the extrusion of fluid gummy resinous substances in the filter orifices of the high pressure filters.

The acid treatment in the first digester is calculated to partially or wholly eliminate or transform sucrose, invert or other sugars, pentosan, and other carbohydrates, as well as coloring and mineral matter and other undesirable incrustants insoluble or not removable by subsequent treatment with the alkaline solution. The acid treatment prepares the material for later digestion with the alkaline solution and bleach. The high dilution and agitation accompanying the treatment separates the fibrovascular bundles, parenchyma cells, knots or other fiber bundles aiding in the removal of the incrustants and purification of the cellulose.

The intermediate washing operations which follow both the acid, alkaline and bleaching operations, if practiced, should be performed multistage, preferably countercurrent at high consistencies and at approximately the treating temperatures with water and dilute liquor.

The alkaline treatment is calculated to render soluble remaining incrustants and mineral matter such as silicates, additional coloring matter and substances which are not dissolved by the previous acid treatment. The alkaline liquid serves to convert the acid-treated pulp stock to a very highly purified and light colored cellulose pulp. Bleaching treatment, if necessary, serves to convert the highly purified cellulose to a high white color. Final customary and conventional steps not shown in the drawings may be used in the manufacture of purified cellulose, such as screening, riffing, dewatering, drying and sheeting to prepare the product for storage, transportation or subsequent use in industries requiring the highly purified cellulose.

The acid employed in the acid treating step is a mixture of sulfur dioxide (SO_2) and water (H_2O) producing sulfurous acid (H_2SO_3). The proportions previously specified are adapted primarily for the treatment of bagasse. The acid treatment should be effected in the absence of air or free oxygen to avoid or reduce to a minimum the formation of sulfuric acid (H_2SO_4) which is detrimental to the cellulose, especially at the

high temperatures employed and rapidly deteriorates the apparatus.

Following the acid treatment, and the high consistency washing operations performed in presses 14, 15 and 16, the fibrous material is subjected to high dilution and agitation within dissolver 17. This separates the fibrovascular bundles, parenchyma cells, knots and other resistant fiber bundles aiding in the removal of incrustants, purifying the cellulose and preparing the material for later treatment with alkali.

The alkaline treatment is preferably with a mixture of water and sodium hydroxide (NaOH). As in the acid digestion step, the alkaline treatment is conducted in a closed vessel which may or may not be jacketed, although the jacket rotatable digester equipped with a conveying screw is preferred in order that the material be uniformly heated and completely united with the alkaline liquor. The alkaline solution employed need not be in excess of 30 pounds of sodium hydroxide (NaOH) per 100 pounds of original oven dry weight of fiber and in normal operation the proportion of sodium hydroxide (NaOH) will range between 16 to 24 pounds per 100 pounds weight of original oven dry weight of fiber. A concentration of from 2% to 4% is normal, about 2.5% being preferred. The fibrous mass undergoing digestion in the alkaline stage should be maintained at temperatures from approximately 100° C. to 150° C. and for a time period sufficient to reduce it to a proper condition. When treating bagasse from one to four hours is usually adequate and from one to two hours at approximately 140° C. with low concentration produces good results. The temperatures and time of treatment will depend upon the concentration of alkali used. Temperatures up to 180° C. may be used, but the advantage of high temperatures, principally in shortening the period, is usually offset by the loss in yield and danger of injury to the material at excessive temperatures. The consistency of the material in the alkaline stage, that is, the ratio of solution to fiber, will depend upon the concentration of chemical employed. Generally, a ratio to 6 to 12 parts of solution to one part of fiber based upon over dry weight gives satisfactory results.

Generally the higher the density of the solution the lower temperature required, and with higher temperatures and greater density of alkaline liquor the fiber is more likely to be attacked by the chemical to an objectionable degree. The fibrous material and solution of sodium hydroxide are introduced at the charging end of the digester in a manner corresponding to that employed in the acid treating stage. The operation in the alkaline stage as in the acid stage is continuous, treatment being completed by the time the digested mass is discharged from the vessel. Care should be taken to reduce the pressure gradually to avoid damage to the cellulose. For this reason the last washing operation is performed at relatively low temperature and pressure with warm or cold water to cool down the mass before discharging to the atmosphere.

After the alkaline treatment the digested mass is discharged into the countercurrent multistage washing operation preferably at high consistency and at substantially digestion temperatures.

The diluted solutions of acid and alkali may be subsequently treated for extraction of the chemicals. Methods of extraction are conventional, including separation of gases and evaporation of the water, incineration, and causticizing with cal-

cium. The use of pure water in the washing operation will prevent contamination with minerals such as iron, manganese, calcium, or other mineral substances.

The final product from the two-stage digestion discharged from the shredder 77 onto conveyor belt 78 will be found to be a highly purified uniform cellulose low in ash, pentosan, low ether extractable matter and a very light color. Bleaching, sheeting and drying of the product are additional steps which may be performed if desired according to the use to which the material is to be put.

While but a single type of alkaline solution has been suggested, it is contemplated that other conventional alkaline methods may be substituted according to the type of fiber.

Since the invention is directed primarily to the apparatus, processes adaptable for use in the apparatus have been but generally described and to a great extent have been limited to operations upon bagasse.

The equipment including the presses, shredders, digesters, and dissolving tank, as well as the connections between these devices, are equipped with suitable valves, vents, pressure gauges, thermometers and other auxiliary equipment essential to proper control and operating conditions.

In the acid and alkaline stages when sulfur dioxide and sodium hydroxide are referred to it is intended to cover such mixtures or dilutions as are necessary to obtain the desired results with the particular stock being treated. Likewise, in the use of sulfur dioxide or sulfurous acid in the acid-treating stage the proportion or dilution of the treating liquor is not intended to be a limiting factor.

In other words, the invention has to do with an apparatus divided for convenience into a plurality of essential steps, an initial step of proportioning the water content, a second step of extracting air and oxidizing gases, an acid digestion step followed by a plurality of successive steps of countercurrent high consistency washing and shredding operations in which a diluent chemical is flowed countercurrently and in which temperatures and pressures are maintained substantially those of the digestion step; a shredding dilution and final extracting step for removing acid liquor and compressing the stock preparatory to charging it to the alkaline digestion, an alkaline digestion step followed by a plurality of washing and shredding operations performed again at high consistency and at substantially the temperature and pressure of the alkaline treatment.

In general any type of press may be used to form a compact plug and advance it into the next stage such as reciprocating or screw presses, and therefore where presses are mentioned it refers to any type that may be used to accomplish the above purpose.

From the foregoing it will be seen that the invention is well adapted to attain all of the ends and objects hereinbefore set forth together with other advantages which are obvious and which are inherent to the apparatus.

As many possible changes and embodiments may be made of the invention without departing from the scope thereof, it is to be understood that all matter herein set forth should be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim:

1. Apparatus for treating cellulosic material, comprising a pair of digesters each having an

inlet and an outlet, means supplying the material to be treated continuously to the inlet of one digester, at least two presses connected in tandem between the outlet of said one digester and the inlet of the other digester, means including a pipe connected intermediate the two presses for continuously supplying washing liquid to the material as it passes from the first press to the second press, means for continuously recovering the liquid expressed from the material by the second press and supplying the recovered liquid to the first digester.

2. Apparatus for treating cellulosic material, comprising a pair of digesters each having an inlet and an outlet, means supplying the material to be treated continuously to the inlet of one digester, at least two presses connected in tandem between the outlet of said one digester and the inlet of the other digester, a repulper intermediate the two presses, means including a pipe connected to the repulper for continuously supplying washing liquid to the material as it is repulped, and means for continuously recovering the liquid expressed from the repulped material by the second press.

3. Apparatus for treating cellulosic material, comprising a pair of digesters each having an inlet and an outlet, means supplying the material to be treated continuously to the inlet of one digester, at least two presses connected in tandem between the outlet of said one digester and the inlet of the other digester, a repulper and a dissolving tank intermediate the two presses whereby the material from the first press advances through the repulper and dissolving tank in order before reaching the second press, means for supplying washing liquid to the material as it is repulped, means in the dissolving tank for agitating the repulped material in the washing liquid, and means for continuously recovering the liquid expressed from the material by the second press.

4. Apparatus for treating cellulosic material,

comprising a pair of digesters each having an inlet and an outlet, means supplying the material to be treated continuously to the inlet of one digester, at least three presses connected in tandem between the outlet of said one digester and the inlet of the other digester, a repulper intermediate the first and second presses, means including a pipe connected intermediate the second and third presses for continuously supplying washing liquid to the material as it advances from the second press to the third press, means for continuously recovering the liquid expressed from the material by the third press and supplying it to the material as it is repulped by said repulper, and means for continuously recovering the liquid expressed from the repulped material by the second press.

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