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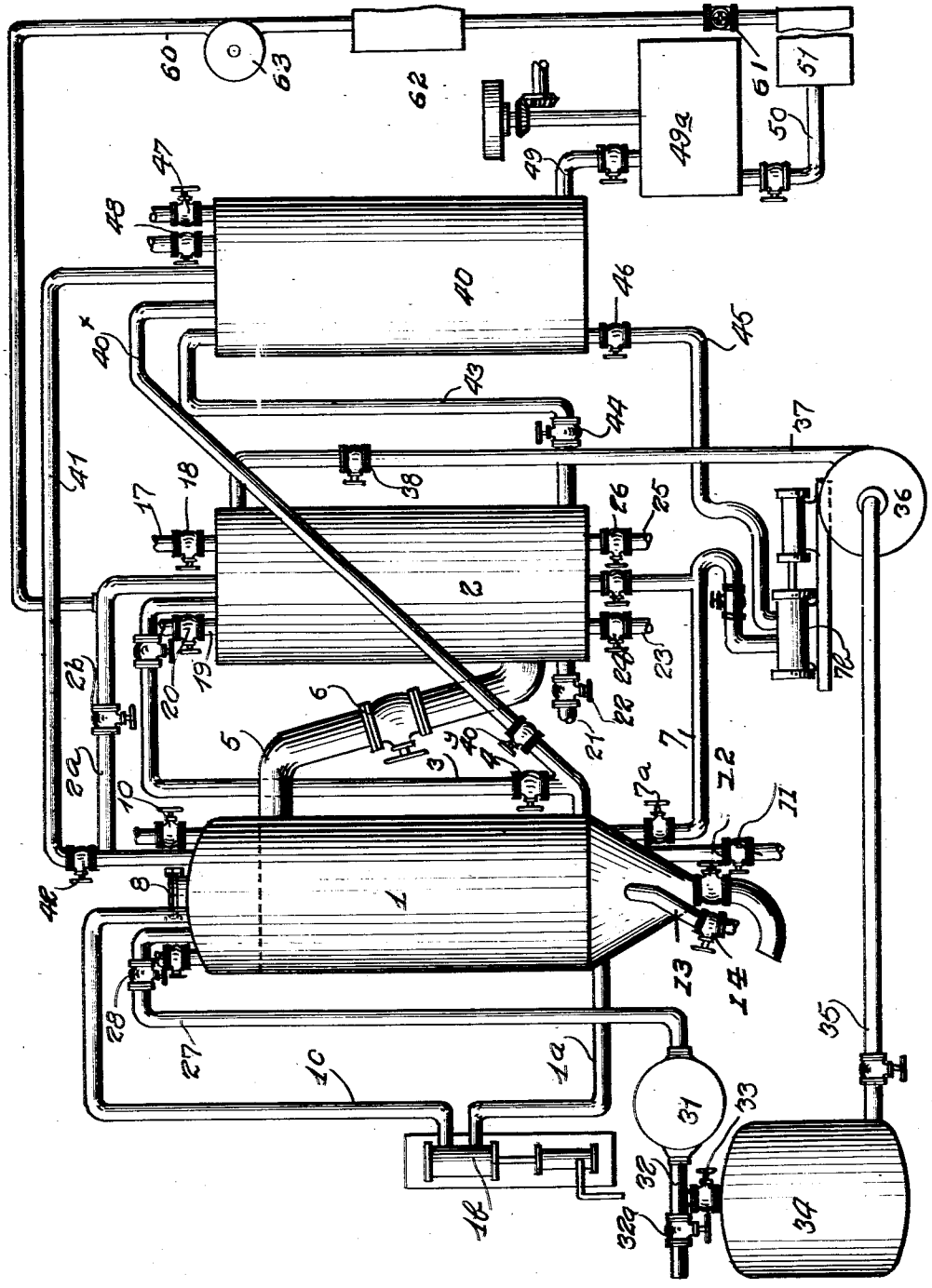

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APPARATUS AND PROCESS FOR MAKING PAPER PULP.

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This invention relates to improvements in an apparatus and process for making paper pulp.

Heretofore in the manufacture of pulp, when the cook is completed the liquor is blown off through the atmosphere and cools and the contained volatile extractives incident to the cooking step pass off and are wasted.

It is true in the soda and sulphate processes, the used liquor is saved, and part of it is subsequently used in the process, but before such use it is exposed to the air and is strengthened by the addition of raw liquor. The loss of heat when the liquor is blown off, and its consequent contact with the air as the liquor is now recovered, renders subsequent use of the liquor expensive, as it is deprived of the effects of the contained and intermixed volatile extractives given off in the previous cook.

One of the prime objects of this invention is to provide a process, and an apparatus for carrying out the process, which will insure of the retention of the solvent extractives, resulting from the treatment free of contact with the air, and thereby preserve most of the heat and the values in the liquors for future use. Then, in the next operation these liquors are enriched by the addition of strengthening agents, which produce highly important results and a greatly improved pulp.

A further object of the invention is the provision of an apparatus, and process, for materially lightening the color of the pulp, without employing intensive bleaching after the pulp is finished, thereby reducing the cost and time incident to the manufacture of the product.

Another object of the invention is to provide a step in the process of making pulp which acts to wash the pulp before it cools, and by the liquor used in treating the material.

Another object of the invention is to provide a process, wherein, in the treatment of the material being acted upon, the digesting liquor is initially mixed with an alkali and solvent extractives from material similar to that being treated, or solvent extractives from such treated material, to soften, attack and otherwise act on the constituents of the material under treatment, other than the contained cellulose, without weakening or dissolving said cellulose.

I have found by practical demonstration, and it is one of the objects of this discovery, that by treating material used for making pulp with digesting liquor charged with an alkali and the solvent extractives from material similar to the material being treated, and preserving this liquor free of the atmosphere, in its re-use, when strengthened with added alkali, or like reagent a chemical action or re-action is brought about, and when the material being treated is impregnated with this super-charged liquor a high grade pulp is produced.

A still further object of this invention is to provide a process, wherein the digesting liquor is initially charged with an alkali or like reagent and solvent extractives of plant material, and after use on the treated material the liquor is preserved without contact with the atmosphere, for re-use in a subsequent treatment of a charge of material in a liquor, stronger in solvent extractives than the initially charged liquor first used.

Other objects and advantages will appear from the specific description in the specification, and will be particularly pointed out in the claims.

In order to obtain a comprehensive understanding of the invention, reference is made to the accompanying drawing which illustrates diagrammatically one form of apparatus for carrying out the process.

It may be stated that the term "initially charged" as used in this application is intended to distinguish the liquor employed, from liquor in which an alkali or like reagent is used in wood treating processes, but is free of solvent extractives at the time said liquor is introduced to the material being treated. In other words, "initially charged" as herein used means that the liquor before actual treatment on the material commences, contains an alkali or like reagent and solvent extractives of plant substance. Solvent extractives as used throughout this application means substances removed or liberated from the plant material by means of the particular digesting operation and at the temperature employed therein. These extractives may be in the form of gases or vapour and may be in solution or absorption in the liquor. Thus, alkali is a solvent but not a solvent extractive as used herein. Thus a distinction between solvent extractives and chemical re-agents as used will be apparent.

In the drawing, 1 illustrates a digester to receive chips or other fibrous material, suitable for making pulp. Spaced from the tank 1, is a tank 2, in which the digesting liquor is stored and pre-heated before being introduced to digester 1. A pipe 3, connects the bottom of digester 1, with the top of the tank 2, a valve 4, being provided to regulate the flow of fluid or volatile extractives and the like, as the case may be, passing from tank to the digester. A pipe 2^a connects the top of the digester 1 and the top of the tank 2, a valve 2^b being provided to control the flow through the pipe. At times it may be found desirable to take off fluid or volatile extractives and the like from tank 2 to tank 1, or vice versa. This pipe is provided to meet this emergency.

A pipe 5, communicates with the digester 1, at or about the level of the liquor, when the chips are submerged, and communicates at its other end with the bottom of tank 2, a valve 6 being provided to control the flow through the pipe to tank 2.

Communicating with bottom of the digester 1 is a pipe 7, which also communicates with the bottom of tank 2, a valve 7^a in the pipe, controlling the flow of liquor between the digester and tank and vice versa. In this pipe is a double acting pump 7^b, to assist the flow of liquor through the pipe in either direction.

At the top of the digester 1, is a closure 8, for the introduction of the chips to be treated, and adjacent thereto is a relief valve 10. From the bottom of the digester 1, extends a drain pipe 11, provided with a valve 12, and adjacent thereto is a steam pipe 13, having a valve 14, for introducing steam to the liquor in the digester 1, when carrying out the process.

A pipe 1^a communicates with the bottom of the digester and connects with the suction side of a pump 1^b, and from the opposite or outlet side of the pump leads a pipe 1^c, which communicates at its opposite end with the top of the digester. This pump and pipe line is utilized to establish a circulation in the digester 1, to facilitate the action of the liquor on and in the chips during the treating operation, as will hereinafter appear.

From the top of tank 2, extends a steam pipe 17, having a valve 18, and adjacent thereto is a pressure release pipe 19, provided with a valve 20. The bottom of the tank 2 is provided with a pipe 21, and a valve 22, to charge the tank with liquor, and in said bottom is a draw-off pipe 23, provided with a valve 24, and a steam pipe 25, having a valve 26.

Extending from the top of the digester 1, is a pipe 27, having a valve 28, the pipe 27 leading to the suction side of a vacuum pump 31, and from the exhaust side of the pump leads a pipe 32, provided with valves 32^a, and 33. The pipe 32, communicates with a tank 34, and extending therefrom is a pipe line 35, leading to a pump 36. A pipe 37, leads from pump 36, to the top of tank 2, and in this pipe is a valve 38.

40 indicates a closed tank, from the top of which leads a pipe 40^x, connected at its opposite end to the bottom of the digester, and provided with a valve 40^y. Another pipe 43 extends from the top of tank 40 and leads to the bottom of tank 2, and is provided with a valve 44. From the bottom of tank 40 extends a pipe 45, which leads to the pump 7^b and is provided with a valve 46.

A pipe 41 communicates with the top of the tank 40, and is provided with a valve 42. This pipe connects with the top of the digester 1, through a part of pipe 2^a, and like pipe 2^a is used to pass solvent extractives and other fluids from the top of tank 40 to the top of the digester, and vice versa. This line is only used under circumstances requiring passage of extractives from the tanks, should occasion require.

The top of the tank 40 is provided with a relief pipe and valve 47, and a steam pipe and valve 48. From the bottom of the tank 40, leads a pipe 49, which extends to a centrifugal machine 49^a, and from this machine extends a pipe 50 which leads to an evaporator 51.

In operation, digesting liquor is introduced to tank 2, through pipe 21. This liquor is initially charged with an alkali, such for instance as sodium carbonate, lime, caustic potash, or the like, and solvent extractives of plant material, such for instance as the terpenes and other lighter and heavier oils. By introducing steam into tank 2, through pipe 25, the liquor is heated to a temperature of about 274° F., and a pressure of about 30 lbs. is produced. Through the closure 8, chips to be treated are introduced to the digester 1. Before charging the digester 1 with chips a sufficient quantity of alkali is introduced in the bottom of the digester. Then the chips are run into the digester and valve 7^a is partially opened to admit sufficient liquor from tank 2, to allow of saturation of the chips, and simultaneously valve 4 is partially opened to admit volatile extractives and alkali from the top of tank 2 to the liquor in the bottom of the digester, in an amount sufficient to maintain the desired temperature of the chips and the liquor, care being taken to regulate the heat so as to avoid raising the temperature above 150° F. Pump 1^b is started and the charge of liquor is pumped through pipe 1^c, to the top of the digester and continuously sprays the liquor on the chips while the digester is being charged. The pump circulates and sprays the liquor

in the digester, which penetrates and saturates the chips and raises the temperature of the entire mass. When the digester has been charged, closure 8 is put in place, but the operation of spraying the liquor is continued. Valve 33 is closed and valves 28, and 32[a] are opened, and vacuum pump 31 is started to exhaust the air from the digester 1, to the atmosphere, through pipes 27, and 32, and a vacuum of about 15 to 20 inches is created.

The vacuum withdraws the air from around the chips, and from the interior of said chips, to render the interior more susceptible to the penetration of the digesting liquor, even the innermost cells of the individual pieces. The vacuum is maintained for such length of time as may be found desirable, this depending upon the physical characteristics of the chips, and the nature and extent of treatment, the chips are to be subjected to.

During the time the vacuum is maintained, the pump 1[b] operates to continue the circulating action of the liquor. This circulation of the liquor in spray form, while the vacuum is on, insures the cellular structure of the chips being further saturated with the alkali and solvent extractives which produces in effect a bleaching action. While the vacuum is on valve 4 is also kept open for continued admission of the volatile extractives to the liquor in the bottom of the digester 1.

When the material becomes thoroughly saturated the vacuum pump is stopped and valves 28 and 32[a] are closed, and valve 4 is opened wide to admit a full charge of the alkali and solvent extractives to the digester 1, to bring up the temperature of the chips to or about 212° F. The increased charge of alkali and solvent extractives under this higher heat removes resins and inter-cellular binding materials, previously softened and dissolved by the alkali and extractives under low temperature conditions and a further bleaching action of the fibers is obtained. This treatment is continued for about thirty minutes, more or less, and then valve 4 is closed.

During the vacuum period, the digesting liquor in tank 2, is heated to the desired degree, and the corresponding pressure raised in the tank by introduction of steam into the bottom of the tank through pipe 25.

In like manner valve 40[y] may be opened if desired, to transfer volatile extractives stored in tank 40, to digester 1. Of course, during this time the cooking liquor in tank 40 is highly heated and considerable pressure results, which causes the solvent extractives above the liquor to readily pass through pipe 40[z] when valve 40[y] is opened. These extractives penetrate the plant material even to the inner cells of the latter, and the chips are heated by these extractives, to or about 200° F. this step in the process requiring approximately 15 minutes. Valve 4 having been closed, valve 7[a], is opened and liquor initially charged with the solvent extractives and alkali, in a super-heated condition in tank 2, is, by reason of the pressure in said tank forced over into the digester 1, and the chips are submerged, the level of the liquor in the digester being at about the level of pipe 5. When the chips are submerged in the digester, their temperature is about 250° F. This temperature and a pressure of about fifteen pounds is produced by admitting steam to the digester through pipe 13, and is maintained for about thirty minutes, more or less, to insure of the digesting liquor containing the solvent extractives and alkali, thoroughly impregnating the cells and fibers of the chips to act on the constituents associated with the cellulose, without in any way injurying the cellulose and the cellulosic material. To cause the most effective action of the liquor on the chips, operation of the pump 1[b] is continued, so that at this period in the process the super-heated liquor is circulated, which with the high heat and pressure, a uniform distribution of the liquid components in and around the chips is insured. The liquor treating step in my process is important, as the previous treatment of the plant material by the mixture of the solvent extractives and alkali materially assists the liquor step to accomplish the desired end, bearing in mind that, previous to this time the plant material was subjected to the action of the alkali and extractives, and liquor under low temperature conditions, which distributed the alkali and extractives in and throughout the cellular structure of the chips and caused a bleaching action of the fibers. The constituents to be removed have therefore, to a very large degree, been heated, softened, dissolved, and partly removed, and the chips are made ready for the more active treatment of the introduced digesting liquor.

The liquor serves to melt and dissolve resins and removes same from within the chips; removes tannins and organic matter, which if allowed to remain would cause a tanning effect upon the fibers of the chips and thereby deteriorate their value; and it also removes mineral salt from the chips.

The treatment of the plant material by the liquor, initially charged with solvent extractives from chips and an alkali, lightens the color of the chips being treated a phenomena which cannot well be explained, but actual demonstration has shown it to be a fact. It is thought that the first subjection of the chips to the combined action of solvent extractives and alkali at low tem-

perature, materially assists in the accomplishment of this highly important result. It is further believed that with this preliminary treatment, the subsequent treatment of the chips by the liquor, and the differences in temperature surrounding the same, from the time they are placed in the digester to be treated, until the treatment is completed, brings about certain unexplained actions and reactions on the material, without affecting the cellulose. The light color of the final product is one of the results flowing from my improved process and is of commercial importance as it saves the expense and delay incident to the usual intensive bleaching step. During the time the chips are treated in the digesting liquor, ebullition of the digesting liquor in the digester 1, and the liberation of the released constituents from the chips, cause certain of these constitutents to rise to the top. This layer or surface accumulation on the liquor contains resinous substances, terpenes and other by-products liberated from the chips, which would be detrimental to the pulp if permitted to come into contact with the atmosphere and thereafter remain in or on the liquor or material, and yet these by-products are valuable as extractives for future use, if preserved free of contact with the atmosphere. Hence, after the chips are treated with the digesting liquor in the digester 1, valve 6, in pipe 5, is opened, and pressure in the digester 1 blows the surface constituents accumulated on top of the liquor, and the volatile extractives above the liquor, over into tank 2, where same are preserved for use on a subsequent charge of chips in the digester. This suddenly reduces the pressure in tank 1, which causes an outward movement of the liquor containing the constituents from the chips, pressure at this time being below the pressure in tank 1. Valve 6, is closed, and the volatile and other constituents saved from the charge in the digester 1, are preserved in tank 2 without air in anywise coming into contact with said liquor, thus preventing oxidation or other deterioration of these important constituents. When valve 6 is closed, valve 7^a is opened, and pump 7^b is started, which with the contained pressure, forces the digesting liquor from the digester 1, through pipe 7 and into the bottom of tank 2, then valve 7^a is closed.

In this transfer of the liquor from one tank to the other, the liberated extractives and the resinous properties taken from the wood now contained in the liquor, are carried over without in anywise coming into contact with the atmosphere, and the original digesting liquor is thereby enriched, and is preserved for a subsequent treatment, the lighter properties of course rising to the top of the liquor in tank 2, and are employed for the preliminary treatment in the next run.

After the now super-charged liquor has been transferred to tank 2, and valve 7^a closed, valves 28, 33 and 38 are opened, and pumps 31 and 36 are started. Pump 31, pulls a vacuum of about 15 inches in the digester 1, which will draw moisture volatile extractives and remaining dissolved constituents from the innermost cells of the chips and transfer same to tank 34, from whence they are transferred into tank 2 by the pump 36, and are preserved to further enrich the already enriched liquor in tank 2, to be used in a subsequent treatment. This second vacuum treatment recovers extractives which only become volatile under pressure below atmospheric, such extractives being preserved and intermixed in the liquor for enriching same for future use.

Tank 40 is charged with cooking liquor, which may be soda, sulphate or the like, and the temperature and corresponding pressure is provided to super-heat the liquor, the desired temperature and pressure being raised according to the character of liquor being used. While the vacuum is held in the digester 1, valves 7^a and 46 are opened and steam is admitted to the top of the tank through pipe 48, which transfers the cooking liquor from tank 40 to the bottom of digester 1, and there valves 38 and 28 are closed, and the vacuum pump is stopped, and steam is then admitted to the digester through pipe 13. The temperature of the cooking liquor in the digester is raised to about 337° F. and a corresponding pressure of about 100 lbs. and the material is cooked from about four to seven hours, depending upon the result sought to be accomplished. Valves 7^a and 46 are of course closed as soon as the liquor has been transferred.

During the cooking step, the temperature and pressure in the digester is maintained, while the temperature and pressure in tank 40 is lowered, and periodically the valve 42 or 2^b are opened and the solvent volatile extractives in the digester are transferred to the top of tank 40 or tank 2 as desired and are thus saved, to enrich the liquor for future use.

During the period of the cook, the material and the liquor are agitated at intervals by introducing steam through the bottom of the digester. From time to time valve 44 may be opened and by reason of the pressure in tank 40 being higher than the pressure in tank 2, the solvent volatile extractives in tank 40 are transferred to the bottom of tank 2 to enrich the digesting liquor therein, for use in a subsequent digesting step.

After the cooking step is completed, valve 7^a and 46 are opened and pumps 7^b started and the pressure in the digester and the action of the pump transfers the liquor to

tank 40, entirely free of contact with the atmosphere, hence saving the values in the liquor and the added solvent extractives from the material for future use.

After the cooking liquor has been transferred to tank 40, valve 7^a is opened and pump 7^b is started and super-heated digesting liquor from tank 2 is transferred to the digester 1, and the pulp material is treated for approximately 30 minutes, the temperature of the liquor during this treatment being maintained at about 285° F. With a corresponding pressure. After this treatment the liquor is again transferred to tank 2, without in anywise coming in contact with the atmosphere. The pulp is now removed from the digester.

This treatment of the pulp material at this stage of the process acts to preliminarily wash the material, which assists in expediting the final washing of the pulp.

One of the salient features of the invention is the recovery of the cooking liquor without contact with the air to preserve the base liquor and its now intermingled and intermixed solvent extractives for cooking the next run of chips in the digester. All this cooking liquor is not preserved as its base has been weakened by the action incurred in the digester. Hence a part of the recovered liquor is drawn off to the centrifugal machine, and treated to separate the lighter from the heavier portions, the lighter portion of the liquor being preserved to recover any remaining values. The matter separated in the centrifugal machine is removed to incinerators and treated to further recover chemicals for reuse.

As stated, the used recovered liquor in tank 40 is weakened but the materials recovered from the previous use are preserved and are utilized by adding strengthening agents to bring it up to the desired strength for action on a charge in another reuse.

The employment of the enriched digesting liquor in tank 2, after the cooking step is completed acts to wash the pulp material, due no doubt to the fact that it is preserved free from the atmosphere and the collected volatile extractives are retained at the temperature employed. This may be said to be a preliminary washing which of course reduces the cost and time incident to the final or usual intensive washing always necessary in the manufacture of pulp.

Another important result obtained by my invention is, by the recovery of the volatile solvent extractives resulting from the cook, and transferring at least part of these to tank 2, the digesting liquor is greatly enriched for the next charge to be treated.

In order to save and utilize the volatile extractive envolved from the evaporator, and utilize same in my method, a pipe 60 is connected to the pipe 2^a, and the evaporator 51 and in the pipe is a valve 61. A condenser 62, and a pump 63, is arranged in the pipe line 60. When it is desired to collect the volatile extractives from the evaporator, the valve 61, is opened, pump 63 is started, and the extractives evolved from the contents of the evaporator are pumped through the condenser and into tank 2, to enrich the contents of the tank. In this way the extractives are preserved for future use in the process.

In the development of the art of making pulp I have coined the term "superheated water of super-heated liquor" to distinguish from mere cooking or boiling the material at atmospheric pressure. Therefore that the meaning of this term be understood in connection with the present invention, it may be said that "super-heated" as herein employed, defines a temperature and corresponding pressure above the normal boiling point of the liquid and of the solvent constituents of the material when mixed with water. Certain peculiar beneficial results in the treatment of pulp material flow from the use of super-heated liquor, and in the present case advantage is taken of these results by super-heating the liquor initially charged with an alkali and solvent extractives of the plant material. In other words, practical demonstrations have proven that by super-heating the initially charged liquor the combined temperature and pressure produces a condition, whether chemical or physical, I am unable to state, that effectually treats the material to remove certain constituents from the material which may be used for subsequent use, without impairing the quality of the cellulose.

What I claim is:

1. The process of producing fibre pulp consisting in removing air from the material to be treated, treating the air free material with liquor charged with volatile extractives of plant material, and subjecting the so treated material to a second digesting operation with a renewed and strengthened liquor containing solvent extractives.

2. The process of producing strong fibre pulp consisting in removing air from the material to be treated, treating the air free material with liquor charged with volatile solvent extractives of plant material, and subjecting the so treated material to a second digesting operation with a renewed and strengthened liquor containing solvent extractives and preventing the atmosphere from coming into contact with the material being treated or the liquors during the successive steps of the treatments.

3. The process of producing strong fibre pulp consisting in removing air from the material to be treated, treating the air free material with liquor charged with volatile solvent extractives of plant material to re-

move certain constituents, and subjecting the so treated material to a second digesting operation with a stronger liquor containing solvent extractives to remove constituents which have not been removed by the previous treatment.

4. The process as defined in claim 1 in which the air free material is impregnated with volatile extractives from either of said liquors.

5. The process as defined in claim 1 in which the air free material is impregnated with volatile extractives from said first mentioned liquor and in which excess liquor together with further volatile extractives are removed by means of a vacuum and transferred to a liquor container.

6. The process as defined in claim 1 in which the said second mentioned liquor containing solvent extractives not removed by the first mentioned liquor is strengthened by the addition of fresh pulp producing liquors.

7. The process as defined in claim 1 further characterized in that said liquors are independently removed together with available volatile extractives to a liquor container such materials being there made available for reuse.

8. The process of producing strong fibre pulp consisting in removing air from the material to be treated, treating the air free material with liquor charged with volatile solvent extractives of plant material, and subjecting the so treated material to a second digesting operation with a previously used liquor to which the desired strengthening material has been added.

9. The process as defined in claim 1, wherein the so treated material is subjected to a preliminary wash with used first mentioned digesting liquor.

10. In a two-stage pulp producing process the step which consists in admixing the volatile extractives from the two treating liquors and while in a super-heated condition applying said admixture to the material being treated.

11. In a two-stage pulp producing process the step which consists in enriching the liquor used in the first stage by the addition of gases or vapours evolved from the second stage liquor in the storage tank.

12. In a two stage pulp producing process the step which consists in transferring volatile extractives evolved in the digester during the second stage cook to one or both of the tanks in which the treating liquors are stored.

13. An apparatus for producing pulp consisting of a digester in combination with a tank for preliminary treating liquor, a second tank for secondary treating liquor, a pump for removing air from material in said digester, means for circulating liquor to or from said digester from or to each of said tanks, without contact with the atmosphere, and separate means for transferring vapours containing volatile solvent extractives from or to the top of said digester from or to each of said tanks.

14. An apparatus as defined in claim 13 having additional means for circulating volatile extractives from the top of either or both of said tanks to the bottom of the digester for the purpose described.

15. In a process of making pulp, the step which consists in recovering the vapors and volatile extractives from the evaporator and incorporating the recovered vapors and volatile extractives in the liquor in which the pulp is treated.

In testimony whereof I affix my signature.

FREDERICK K. FISH, JR.