No. 630,293. Patented Aug. 1, 1899.

E. MAERTENS.

PROCESS OF AND APPARATUS FOR CLEANING WOOL.

(Application filed June 24, 1899.)

(No Model.)

FIG. 1.

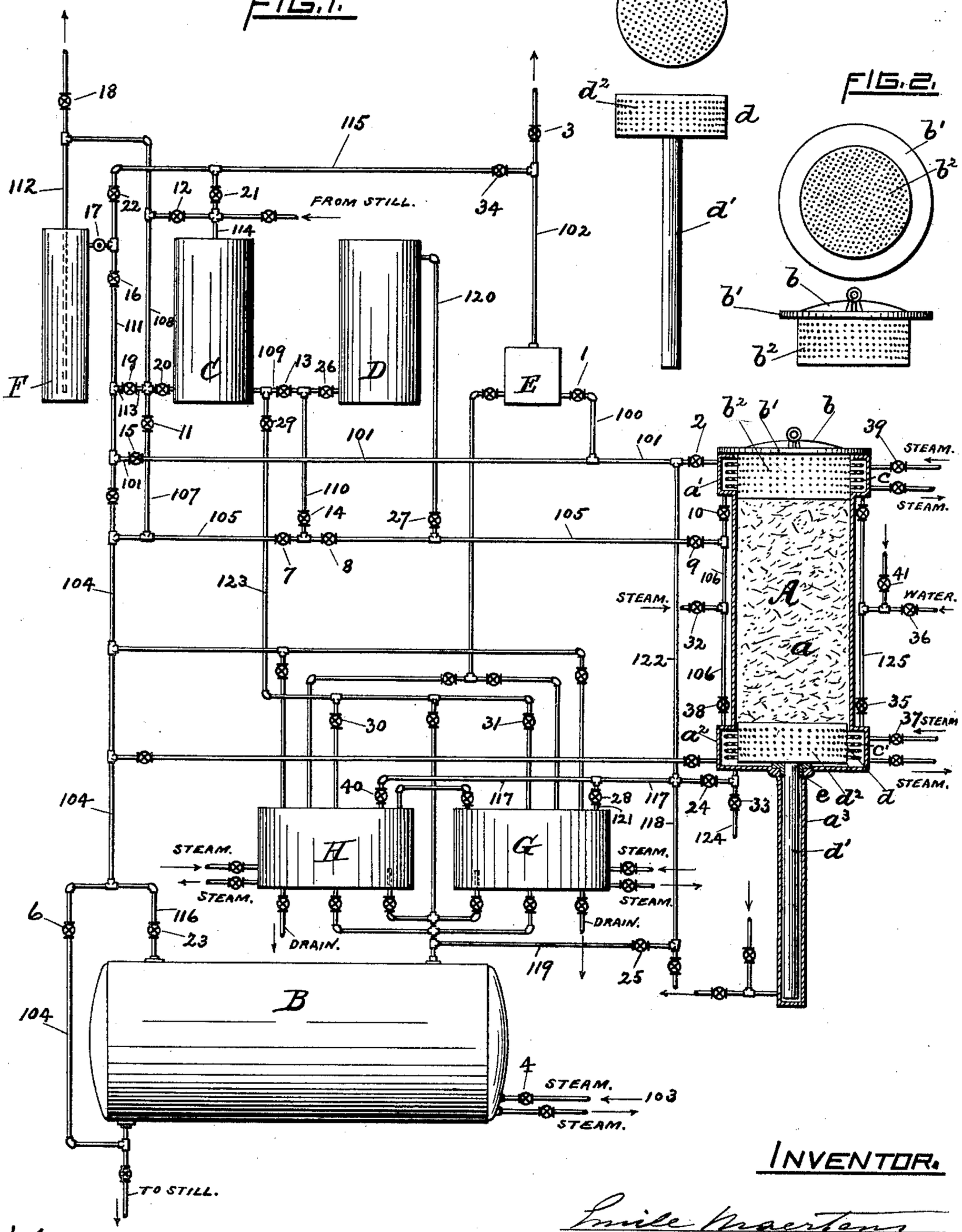

FIG. 3.

FIG. 2.

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PROCESS OF AND APPARATUS FOR CLEANING WOOL.

SPECIFICATION forming part of Letters Patent No. 630,293, dated August 1, 1899.

Application filed June 24, 1899. Serial No. 721,730. (No model.)

To all whom it may concern:

Be it known that I, EMILE MAERTENS, a citizen of the United States, residing at Providence, in the county of Providence and State of Rhode Island, have invented certain new and useful improvements in the art of cleaning wool and other animal fibers with volatile solvents, the object being to obtain the wool or fiber in the best workable condition and to recover the by-products, such as wool-fat and the potash, of which the following is a specification.

The invention relates to a process of and apparatus for the extraction of oily, greasy, resinous, and other substances from wool and other material and is especially designed for use in establishments where the output does not warrant the necessary expenditure for the more elaborate processes and apparatus described in my United States Patents Nos. 545,899, 545,900, and 615,030.

Referring to the accompanying drawings, Figure 1 is a general elevation of the apparatus with the digester shown in section. Fig. 2 is a detail of the digester-cover, and Fig. 3 is a detail of the hydraulic plunger with its perforated extension or platen.

A is the digester, wherein the material to be extracted is placed, either while contained in a cage or basket or loose, and is supposed to be provided with suitable doors or covers for loading and unloading the material and closing the apparatus. It can also be of the special construction shown herein, which consists of the straight cylindrical part a, carrying at its top an enlarged annular chamber a', adapted for the reception of a cover b and of heating-coils c, and at its bottom a somewhat similar enlarged annular chamber a^2, adapted for the reception of heating-coils c' and of a hydraulic ram d, having a water-tight packing at e. The cover b consists of the solid cap part b' and of the foraminous hollow extension b^2.

The ram d, which is shown in detail in Fig. 3, consists of the plunger d', adapted to slide in the extension a^3 of chamber a^2, and of the foraminous hollow extension or platen d^2, which is fastened to it.

B is a reservoir provided with suitable heating-coils for the solvent used in the process and when in use generally contains solvent having a certain amount of extracted matter in solution.

C is a condenser or cooler of any suitable construction for the cooling of liquids or the condensation of vapors.

D is a heater of any suitable construction for the heating or superheating of gases or vapors.

E is a vacuum-pump adapted for use also as an air or gas circulating pump, and it is connected by suitable valved piping with all the other elements of the apparatus.

F is an oil condenser or extractor used for the retention or condensation of the solvent vapors contained in the air which is allowed to escape into the atmosphere and may be provided with heating means for the volatilization of the solvent retained by the oil in said condenser.

G and H are drums or reservoirs provided with heating-coils and are adapted for the reception of liquids, for their separation, volatilization, or evaporation.

The various elements of the apparatus are provided wherever necessary with suitable valved inlet and outlet pipes, as well as with gages, drain-cocks, thermometers, sight-glasses, &c.

The method for operating the apparatus is as follows, the material to be extracted or treated being supposedly raw wool: The digester A having been charged with the wool to be extracted (in space a) and hermetically sealed, by preference, and in order to insure a perfect penetration of the solvent to be used to all parts of the mass to be treated a vacuum is created in the digester A by putting the latter in communication with the pump E through valve 1, pipes 100 and 101, and valve 2. The air exhausted is discharged into the atmosphere through pipe 102 and valve 3. When a suitable vacuum has been obtained, the pump E is stopped, and the valves previously opened are again closed. Steam having been turned onto the coils in reservoir B by opening valve 4 on pipe 103, the pressure soon rises in this tank through the tension of the vapors of the solvent volatilized by the heat of the coils, and when the internal pressure becomes sufficient valve 6 on pipe 104, valves 7, 8, and 9 on pipe 105, and valve 10 on pipe 106 are opened, or if the solvent is judged to be too

hot it is first put through the cooler or condenser C via pipe 107, valve 11, pipe 108, and valve 12, and from the condenser enters pipe 105 by way of valve 13 on pipe 109 and valve 14 on pipe 110. When driving the solvent into pipe 105 by way of the condenser C, valve 7 on pipe 105 must of course be closed. The solvent containing extracted wool-fat in solution from preceding operations is now forced from the bottom of the tank B through the pipes and valves just mentioned into the top of the digester A, which is filled therewith. Should a vacuum not have been made in the digester prior to the admission of the solvent, the latter is preferably allowed to enter at the bottom of the digester by valve 38, the air displaced by the solvent being allowed to escape by opening valves 2 and 15 on pipe 101 and valve 16 on pipe 111 and enter the oil-condenser F through check-valve 17, where in passing through the oil contained therein it is stripped of the solvent vapors with which it is charged before escaping into the atmosphere through valve 18 on pipe 112, or it can first be passed through the condenser C and from there to the condenser F by way of valves 19 and 20 on pipe 113, valve 21 on pipe 114, pipe 115 and valve 22, and check-valve 17 on pipe 111. The digester A being now full of solvent containing wool-fat in solution valve 6 on pipe 104 is closed, valve 23 on pipe 116 is opened, and valve 7 on pipe 105 is closed. The solvent vapors generated in reservoir B now rise through pipes 116, 104, 107, valve 11, pipe 108, and valve 12 and enter the condenser through pipe 114, from which they emerge in liquid form through pipe 109, and from there the pure solvent enters the top of digester A through valve 13, pipe 110, valve 14, pipe 105, and valves 8, 9, and 10, valve 38 being closed. Valve 24 on pipe 117 and valve 25 on pipe 119 are now opened and the charged solvent contained in digester A is allowed to return to tank B through pipe 117, 118, and 119, care being taken by throttling valves 24 or 25 not to let the charged solvent from the bottom of digester A into tank B any faster than the pure solvent from condenser C enters digester A at its top, and thus a complete continued immersion of the material being extracted is insured. If desired, the solvent from the digester can be run into drums H or G by opening valves 28 or 40 before being returned to tank B. When the whole of the material contained in digester A has been sufficiently rinsed with the pure solvent coming from condenser C, valve 11 on pipe 107 is closed, as well as valves 12 and 13 on the inlet and the outlet to the condenser C and valve 8 on pipe 105, whereas valve 7 on pipe 105, valve 26 on pipe 109, and valve 27 on pipe 120 are opened, valve 14 on pipe 110 not having been closed. Meanwhile all the liquid solvent contained in the digester A having been drained into tank B valve 25 on pipe 119 is closed. The vapors generated in B now pass into the heater D, where they are superheated, and from there through valve 10 into the top of the digester, or they can be sent direct from pipe 104 into pipe 105 through valves 7, 8, 9, and 10 and be superheated in the chamber a' by the coils c. These superheated vapors, which are under pressure, somewhat compress the whole mass of wool and at the same time heat it up to the point of vaporization of the residual solvent which it contains, and upon opening valve 28 on pipe 121 leading into drum G a quantity of liquid solvent is discharged into said drum, whereas vaporized solvent ascends pipe 122 into pipe 101 and from there enters the condenser by way of valve 15, pipe 113, valve 19, pipe 108, valve 12, and pipe 114 and when condensed is delivered into drum H or G via valve 29 on pipe 123 and valves 30 and 31. This operation is continued until substantially all the residual solvent has been removed from the material. I have found by experience that the wool is liable to be injured if in the treatment thereof the temperature is allowed to go above 60° centigrade. The exact temperature will vary with different wools as well as with the nature of the solvent employed. The temperature which I prefer to employ in driving off the residual solvent from the wool is from 50° to 55° centigrade. Valves 4, 23, 9, and 28 are now closed, steam is turned on at valve 32 on pipe 106 for a few seconds, and this completely removes the last traces of solvent and deodorizes the material, which is now dry and degreased and in condition for removal from the digester in any suitable manner after atmospheric air has been circulated through the material in order to cool it off. This is done by closing all valves previously opened, starting up the vacuum-pump, and opening valve 3 on pipe 102, valve 1 on pipe 100, valve 2 on pipe 101, and valve 33 on pipe 124. This latter valve 33 admits atmospheric air, which is drawn through the mass and discharged into the atmosphere either through valve 3 or by way of the oil-condenser F via valves 34 and 22 on pipe 115 and valve 18 on pipe 112 or by way of condenser C and the oil-condenser F, as previously described.

The removal of residual solvent by the aid of superheated solvent vapors alone is a tedious and dangerous operation, which, unless carried on with great care and judgment, is very liable to injure the material being treated when this is wool, and is not to be recommended even if carried on by very experienced and careful operators.

The removal of the residual solvent is much accelerated, the quality of the work is much improved, and the danger of injury to the material very materially removed by the use of a vacuum-pump or of steam in conjunction with solvent vapors or by a combination of two or of all three of the elements mentioned.

When steam is used with or without the vacuum-pump, a small jet of it is admitted

to pipe 106 by valve 32, where it mixes with the solvent vapors before they enter the digester, the water resulting from the condensation of the steam being separated from the solvent in drums H or G.

When the vacuum-pump is used with solvent vapors or with steam or with both solvent vapors and steam for the removal of the residual solvent, valve 28 on pipe 121 is closed, and all other valves being open or shut, as required, the vapors are circulated from the bottom of the digester through valve 24 on pipe 117, through pipes 122 101 100, and through valve 1, and are discharged by the pump E into condenser C via pipe 102, valve 34, pipe 115, and valve 21 on pipe 114, the liquefied vapors finding their way into drums H or G via pipe 109, valve 29, pipe 120, and valves 30 or 31.

In lieu of solvent vapors water vapors are sometimes used for the removal of the residual solvent, and when this is the case valve 24 on pipe 117 is closed and the bottom chamber a^2 of the digester is filled or partially filled with water by opening valves 35 and 36 on pipe 125. Steam is turned on to coils c' of chamber a^2 by opening valve 37. Valve 2 on pipe 101 and valve 1 on pipe 100 being opened and the vacuum-pump started, under the influence of the vacuum produced and the heat transmitted by coils c' the water in chamber a^2 is vaporized and drawn through the contents of the digester, heating it up and vaporizing the residual solvent, which, along with the water-vapors, is delivered by the pump E into condenser C and from there into drums H or G, as previously described.

In lieu of water-vapors, of steam, of solvent vapors, or a combination of two or three of the above with or without a vacuum, the residual solvent is sometimes removed by air which is circulated over and over again through the material, preferably in conjunction with steam. When this is practiced, air is allowed to enter the system by valve 33 on pipe 124 or by any other inlet or inlets suitably situated for the purpose, and the pump E is started. If desired, the air may be indirectly heated prior to its introduction into the digester or may be directly heated by mixing steam therewith. The air is drawn through the material and emerges from the digester through valve 2 on pipe 101, enters the pump through valve 1 on pipe 100, leaves the pump through pipe 102, enters the condenser through valve 34 on pipe 115 and valve 21 on pipe 114, passes through the condenser, leaving behind solvent and water vapors, which are condensed and run to tanks H or G, via valve 29 on pipe 123 and valves 30 or 31, and through pipe 109 and valves 13 and 26 enters the heater D, where it absorbs heat, emerges from the latter through pipe 120, enters pipe 105 through valve 27 and pipe 106 through valve 9. In pipe 106 it is preferably mixed with steam injected through valve 32 and reënters the bottom chamber a^2 of digester A through valve 38. This air is thus circulated in a cycle, recooled, reheated, and remixed with steam until all of the residual solvent contained in the material held by the digester has been removed, and it is then discharged into the atmosphere by way of the oil-condenser, as previously described.

In order to accelerate the removal of the residual solvent and before the circulation of steam, air, or vapors through the material, the latter can be squeezed for the expression of the bulk of the residual solvent. This is accomplished by making use of the ram d, (or of any equivalent device or means,) which on being raised by the medium actuating it compresses the material between the hollow foraminous platen d^2 and the hollow foraminous extension b^2 of the cover b, the liquid expressed escaping through the perforations of said platen and cover into chambers a' and a^2 and emerging from there, through valves 10 and 38 on pipe 106 and valves 24 and 28 on pipes 117 and 121, into drum G. When the liquid has been expressed as described, the ram is again allowed to drop, and the removal of the still-adhering solvent is carried on as previously described.

It is obvious that after treating the material and removing the cover b the digester can be emptied by the action of the plunger d', which, when raised, carries with it the hollow foraminous platen d^2 and all that is supported by the latter to the top of the digester A, where it is cared for in any suitable manner.

When the material being extracted is going to be subjected to a subsequent wet treatment in the digester itself, it is convenient to remove the residual solvent by means of the liquid or solution with which the material is to be treated or with water, provided these are immiscible with the solvent used in the extraction, and the operation is then as follows: Assuming that the liquid to be used is water and that the material is to be partially prepared for carbonizing or entirely carbonized in the digester A and that the bulk of the residual solvent has or has not been removed by compression, as described above, water of a suitable temperature is allowed to enter digester A via valve 36, pipe 125, valve 35, and chamber a^2 (it can, if need be, be heated by coils c' in said chamber) and to rise slowly in the part a, containing the extracted material. The adhering solvent is thereby nearly all removed and floated on top of the water or solution used. When the floating solvent reaches chamber b^2, the inlet-flow of water is reduced or stopped, steam is turned on the coil c by opening valve 39, and the floating solvent distilled off either *in vacuo*, the vapors going through valves 2 and 1 and the pump on their way to the condenser, or direct to the condenser via valve 2, pipe 101, valve 15, pipes 104 and 113, valve 19, pipe 108, and valve 12, or the solvent can be floated off slowly into drums H or G via valve 2, pipes 122 117, and valves 28 or 40. When the sol-

vent which has floated to the top has been removed, valve 2 is closed and the material is compressed by moving plunger *d'* upward, as previously described. This liberates further traces of solvent and also the natural soaps or potash salts contained in the wool and which had dissolved in the surrounding liquid. The floating solvent is floated off or distilled off, as previously described, and the liquid containing the potash salts in solution is run into drums H or G by way of valves 10 and 38 on pipe 106 and valve 24, pipe 117, and valves 28 or 40 for the removal of traces of solvent, or it is run direct to a storage-tank by way of valve 33 on pipe 124 for reuse or until its concentration warrants its vaporization for the recovery of the potash salts or other extracted material held in solution. The material can now after a slight steaming be removed from the digester and carried to the washers to be rinsed for the removal of extraneous impurities, or it can be further rinsed in the digester itself by repeated immersions, compressions, and changes of water, and this is the case when it is desired to prepare the material for carbonizing or when it is desired to carbonize the material in the digester itself or to remove it from the digester cleansed or cleansed and dried.

In order to prepare the material for carbonizing after it has been sufficiently rinsed, it is finally compressed and the water allowed to drain out of the digester through valve 33 on pipe 124. The plunger *d'* is then allowed to drop, and after closing valve 33 the carbonizing solution is introduced through valves 41 and 35 on pipe 125 and chamber a^2 into the part *a* of digester A, when the material contained therein is completely immersed by the solution, the flow is stopped, and the material is allowed to steep for a suitable period. The solution is then allowed to return to its storage-tank through valve 33 on pipe 124, and the plunger *d'* is made to compress the material for the removal of the bulk of the adhering carbonizing solution. The material can now be removed to be dried, carbonized, neutralized, and redried, or it can be dried by passing steam and hot air through it in the manner previously described and when dry carbonized by eliminating the steam from the circulation and increasing the temperature of the circulating air in the heater D and in coils *c* and *c'*. When the carbonization is complete, the material can be neutralized in the same manner that it was prepared for the carbonization by merely changing the solution used. It can then be rinsed with water and after being pressed for the removal of the bulk of adhering moisture dried with hot air and steam, as previously described, when it will be found in a finished and clean condition ready to be thrown out of the digester by operating the plunger *d'*. The liquids accumulated in drums H and G are separated by decantation or evaporation, or by both, the drums being suitably connected with the condenser C and tank B, as well as with the drain and with storage-tanks for this purpose.

If desired, the material can be treated with water in the digester after the residual solvent has been removed by superheated solvent vapors, water-vapors, air or steam, or both, or a combination of any of the means mentioned. If this water treatment is done thoroughly in the case of some wools, so as to remove all potash soaps present, and the wool is then partially dried, it can then in many cases be freely carded, and an expensive process can thus be saved.

When the solvent in tank B becomes too much saturated with extracted fat, a part of it is run to a still, where the fat is separated from the solvent. The solvent is then returned to tank B by way of the condenser C direct or by way of condenser C and drums H and G in case the solvent is mixed with water and needs separating in said drums.

It is evident from the description and drawings that, if desired, the liquids, gases, or vapors used in the process can be passed through the digester in either direction when it is deemed advisable or necessary to so do. It is evident that the vacuum-pump can be dispensed with, if necessary, in some of the the operations described and that the suction or vacuum created by the condensation of the vapors in the condenser will to a great extent accomplish the same object. It is further evident that when the solvent used in the process is heavier than the extracted matter its flow through the digester should be upward, whereas when the solvent is lighter than the extracted matter its flow should be downward and that in case of the removal of the residual solvent with a liquid the flow of the liquid should be downward if the solvent is heavier than the liquid used for its removal and that the flow of said liquid should be upward through the digester when the solvent is lighter than the liquid used for its removal.

The method hereinbefore described of removing residual solvent by the circulation of air in a closed circuit is made the subject-matter of my application Serial No. 721,731, filed concurrently herewith.

The described method of removing residual solvent by aqueous vapor is claimed in my application Serial No. 721,732.

The described method of removing residual solvent by the inflow of water is claimed in my application Serial No. 721,733.

Having described my invention, what I claim is—

1. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below 100° centigrade, substantially as described.

2. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in first removing

the excess of solvent therefrom by pressure and then subjecting it to the action of superheated solvent vapors at a temperature below 100° centigrade, substantially as described.

3. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below the point at which the fiber would be injured by heat, substantially as described.

4. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below the point at which the fiber would be injured by the heat, and then drying the same, substantially as described.

5. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in first removing the excess of solvent therefrom by pressure and then subjecting it to the action of superheated solvent vapors at a temperature below 100° centigrade in conjunction with steam or aqueous vapor, substantially as described.

6. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below the point at which the fiber would be injured by heat in conjunction with steam or aqueous vapor, substantially as described.

7. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in first removing the excess of solvent therefrom by pressure and then subjecting it to the action of superheated solvent vapors at a temperature below 100° centigrade in conjunction with a vacuum, substantially as described.

8. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below the point at which the fiber would be injured by the heat in conjunction with a vacuum, substantially as described.

9. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in first removing the excess of solvent therefrom by pressure and then subjecting it to the action of superheated solvent vapors at a temperature below 100° centigrade in conjunction with steam or aqueous vapor and a vacuum, substantially as described.

10. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a temperature below the point at which the fiber would be injured by the heat in conjunction with steam or aqueous vapor and a vacuum, substantially as described.

11. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a suitable temperature and in conjunction with or without steam, aqueous vapors or a vacuum and then deodorizing it, substantially as described.

12. The process of removing residual solvent from wool after the grease has been extracted therefrom which consists in subjecting it to the action of superheated solvent vapors at a suitable temperature and in conjunction with or without steam, aqueous vapors or a vacuum, then deodorizing it and then airing or cooling it, substantially as described.

13. The process of cleaning wool in a digester which consists in first removing the fat therefrom by a volatile solvent, eliminating the residual solvent therefrom with superheated solvent vapors at a temperature not exceeding 100° centigrade, and then depotashing the same, substantially as described.

14. The process of cleaning wool in a digester which consists in first removing the fat therefrom by a volatile solvent, eliminating the residual solvent therefrom with superheated solvent vapors at a temperature not exceeding 100° centigrade, depotashing it and then rinsing it, substantially as described.

15. The process of cleaning wool in a digester which consists in first removing the fat therefrom by volatile solvents, eliminating the residual solvent therefrom with superheated solvent vapors at a temperature not exceeding 100° centigrade, depotashing it, rinsing it, and then drying it, substantially as described.

16. The process of cleaning and treating wool in a digester which consists in first removing the fat therefrom, by volatile solvents, eliminating the residual solvent therefrom, depotashing and rinsing it and then subjecting it to the action of a carbonizing solution, substantially as described.

17. The process of cleaning and treating wool in a digester which consists in first removing the fat therefrom by volatile solvents, eliminating the residual solvent therefrom, depotashing and rinsing it, subjecting it to the action of a carbonizing solution and then drying and carbonizing it, substantially as described.

18. The process of cleaning and treating wool in a digester which consists in first removing the fat therefrom by volatile solvents, eliminating the residual solvent therefrom, depotashing and rinsing it, subjecting it to the action of a carbonizing solution, drying and carbonizing it and then neutralizing the carbonizing agent, substantially as described.

19. The process of cleaning and treating wool in a digester which consists in first removing the fat therefrom by volatile solvents, eliminating the residual solvent therefrom, depotashing and rinsing it, subjecting it to the action of a carbonizing solution, drying

and carbonizing it, neutralizing the carbonizing agent and finally rinsing and drying it, substantially as described.

20. An apparatus for cleaning wool consisting of a digester, a solvent-tank, a settling or separating tank, a surface condenser, a heater or superheater and a vacuum or circulating pump, substantially as described.

21. An apparatus for cleaning wool consisting of a digester, a solvent-tank, a settling or separating tank, a surface condenser, an oil-condenser, and a vacuum or circulating pump, substantially as described.

22. A digester for use in cleaning wool by means of volatile solvents provided with a hydraulic piston adapted for the expression of liquids from the material under treatment, and a cover with a cage attached thereto having its bottom and sides perforated, substantially as described.

23. A digester for use in cleaning wool by means of volatile solvents provided with a hydraulic piston having a foraminous platen attached thereto and adapted for the expression of liquids from the material under treatment, and a cover with a cage attached thereto having its bottom and sides perforated, substantially as described.

24. A digester for use in wool-cleaning, having an enlarged chamber at its top provided with coils, substantially as described.

25. A digester for use in wool-cleaning having an enlarged chamber at its top provided with coils, and a similar chamber at its bottom, provided with coils, substantially as described.

26. A digester having an enlarged chamber at its bottom provided with coils and a foraminous platen adapted to compress the material under treatment, substantially as described.

27. A digester having an enlarged upper portion forming a chamber, heating-coils therein, and a foraminous false bottom adapted to be operated to compress the material under treatment, substantially as described.

28. A digester having an enlarged chamber at its bottom provided with coils, a perforated false bottom or platen adapted to compress the material under treatment, and a cover provided with a depending foraminous chamber, substantially as described.

29. A digester with enlarged upper and lower portions forming chambers exterior to the main body of the digester, coils in said chambers, and means for compressing the material under treatment, substantially as described.

30. The process of removing residual solvent from wool which consists in subjecting it to the action of superheated solvent vapors at a temperature not exceeding 60° centigrade, substantially as described.

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Witnesses:
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