

No. 686,059.

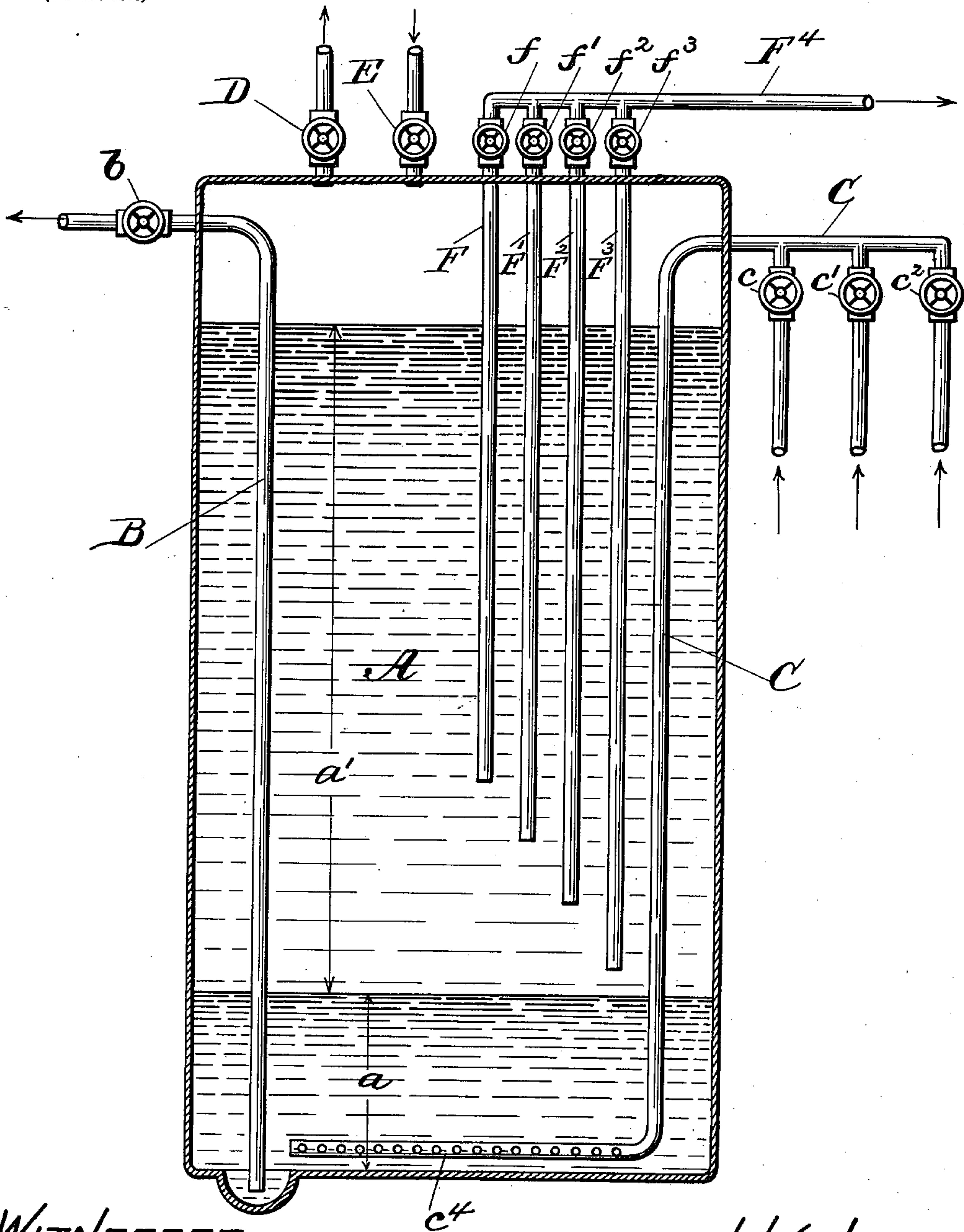
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W. D. HARTSHORNE & E. MAERTENS.

PROCESS OF SEPARATING VOLATILE SOLVENT FROM VARIOUS EMULSIFIED SOLUTIONS.

(Application filed May 2, 1900.)

(No Model.)



WITNESSES.

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# UNITED STATES PATENT OFFICE.

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## PROCESS OF SEPARATING VOLATILE SOLVENT FROM VARIOUS EMULSIFIED SOLUTIONS.

SPECIFICATION forming part of Letters Patent No. 686,059, dated November 5, 1901.

Application filed May 2, 1900. Serial No. 15,265. (No specimens.)

*To all whom it may concern:*

Be it known that we, WILLIAM D. HARTSHORNE, residing at Methuen, in the county of Essex and State of Massachusetts, and EMILE MAERTENS, residing at Providence, in the county of Providence, State of Rhode Island, citizens of the United States of America, have invented new and useful Improvements in the Art of Separating Solvents from Oily, Fatty, Waxy, Soapy, or Emulsified Solutions of the Same, of which the following is a specification.

In the degreasing of wool or other animal fibers with fat solvents (by processes such as are described in United States Patents Nos. 545,899, 545,900, 615,030, 630,293, 630,294, 630,295, and 630,296) and the subsequent recovery and separation of the extracted matter and solvent from each other a preliminary treatment or preliminary treatments of all or of a part of the solvent containing extracted matter in solution before distillation is not only desirable at times, but necessary for technical and economic reasons.

In the treatment of raw wool with a fat solvent not only does the solvent dissolve the fatty matters present in the wool, but some of the alkaline salts or natural soaps, mucus, and water existing in the wool are carried away by the solvent to some extent also, and according to the nature of the solvent used partly in solution and partly in suspension, and this is specially so for hydrocarbon solutions when no drying of the wool before its extraction has taken place. In order to facilitate the distilling operation for the separation and recovery of the solvent from the extracted fat and in order also not to deteriorate the quality or impair the value of the latter, it is advisable that the alkaline salts or natural soaps present in the solvent solution of fat should be decomposed, and in order to clarify the liquor from mucus and sediment more quickly the water or moisture present must be removed also. When the treatment or process is such that solvent fatty matter, alkaline salts, or natural soaps and water are brought together in any part of the apparatus an emulsion is formed, and upon sedimenta-

tion the liquids separate into layers and according to their density. One extreme layer consists mostly of solvent containing fat in solution, the other extreme layer consists mostly of aqueous solutions of alkaline salts or natural soaps, while the middle or intermediate layers are emulsions composed of some of all the bodies present and which are very hard to separate from each other without having recourse to a chemical reaction upon some of them.

The clarification of the solvent fat solution is carried out as follows: A concentrated and suitable salt solution is first prepared and run into a suitable tank, and then commercial sulfuric acid in sufficient quantity to make a solution of, say, from six to eight per cent. strength is added. The volume of this solution may be from eight to ten per cent. or more of the capacity of the treating-tank and should stand, preferably, not less than one foot in depth. The solvent solution of fat is forced under pressure through this acid-salt bath in a state of finely-divided spray, so as to cause all parts of it to come into intimate contact with the acid-salt solution. The dissolved and suspended alkaline compounds are immediately attacked by the acid and the entrained moisture is taken up by the salt solution. The rapid movement carries these compounds forward mixed with the attacking acid-salt solution; but upon shutting off the supply of the solution and completely closing all inlets and outlets to the tank they at once begin to settle, and in a short time the solution is clarified and practically freed from water, acid, and alkali and may be immediately distilled. This strength of acid-salt solution is ordinarily sufficient to treat thirty times its own volume of fat solvent solution.

A still more complete separation of entrained moisture and acid may be effected by passing the above settled or partly-settled solvent solution through a second or third concentrated solution of chlorid of sodium or other suitable salt and allowing to settle each time. The more completely this is done before distilling the better the quality and appearance of the grease.



Any compatible aqueous saline solution whose gravity is heavier than water with a sufficient acid reaction and affinity for water may be used for the purpose where the solvent is lighter than water. Very fine results may be produced by saline solutions of oxalic acid; but this is generally too expensive. Sulfate of sodium and sulfuric acid or sulfuric acid and chlorid of sodium are generally the most convenient and cheapest substances to use.

Where the solvent solution has come in contact with water in any quantity before the above treatment or when the fiber is freed from solvent by the action of water and pressure, the liquor so affected may be allowed to stratify, and the supernatant portion, which is principally a fat solution, is treated as above indicated, and those layers which show a decided emulsified effect are carefully separated and passed through the acid-salt solution above, and if the quantity of clear solution resulting is sufficient to warrant the trouble it may be decanted and distilled with the regular run. Generally this emulsified portion may be simply passed through the acid-salt solution mentioned above when this is nearly in a spent condition and the solvent simply boiled or distilled off before throwing away or taking care of the sludge or residuum. It is evident that for this latter operation, if more convenient, acid without any saline solution may be used to simply cause the breaking up of the emulsified compound of water, natural soap, fat, and solvent if the object is only to save the solvent and throw away the sludge or care for the latter in any suitable manner not requiring the product to be a pure form of grease.

If it is proposed to merely clarify the solvent fat solution, suitable saline solutions without acid will accomplish the purpose, and for some uses to which the grease may be put this may be desirable.

We do not limit ourselves to the precise strength, volume, or composition of the clarifying and decomposing solutions indicated or mentioned, nor to the precise manner in which their application has been described.

In the general description of the operation and the character of the solutions mentioned it has been assumed that the solvent used is a solvent lighter than water; but the principle of acid reaction and affinity for water in a solution of proper gravity may be applied to solvents of any density. It is evident, of course, that when the solvents used are heavier than water the saline or acid solutions are preferably passed or sprayed through the solvent instead of the solvent through them and that the bodies eliminated therefrom are then floated to the top of said solvent solution instead of being precipitated below it, and it is evident also that acid or saline solutions can be sprayed or passed downwardly through solvents lighter than them instead of said solvents being passed

through them upwardly and that solvent solutions heavier than the acid or saline solutions can be sprayed or passed downwardly through them also without departing from the spirit of the invention.

A general type of apparatus suitable to the process is represented by the annexed drawing, in which—

A represents a suitable tank, in which  $\alpha$  shows the approximate relative levels of the acid or saline solutions, and  $\alpha'$  the approximate relative levels of the solvent solution.

B is a draw-off pipe controlled by valve  $b$ .

C is an inlet-pipe by which the valved branch  $c$  admits acid or saline solutions. The valved branch  $c'$  admits steam and the valved branch  $c^2$  admits solvent solutions to tank A and which has a perforated branch  $c^4$  at the bottom of said tank for spraying purposes.

D is a valved outlet for gas and solvent vapors, communicating with a condenser.

E is a valved inlet for a compressed elastic fluid.

F F' F<sup>2</sup> F<sup>3</sup> are outlet-pipes for forcing the settled solvent solution to the still from different levels in the tank A by way of their corresponding valves  $f f' f^2 f^3$  and the pipe F<sup>4</sup>.

Sight-glasses and petcocks (not shown in the drawing) are placed at convenient points on the tank A for observation and sampling purposes before decantation or distillation.

What we claim, and desire to secure by United States Patent, is—

1. The process of decomposing the emulsion resulting from the cleaning of wool by volatile solvents which consists in subjecting said emulsion to the action of an acid whereby the acid combines with the alkaline salts of the emulsion, substantially as specified.

2. The process of decomposing the emulsion resulting from the cleaning of wool by volatile solvents which consists in subjecting said emulsion to the action of an acid whereby the acid combines with the alkaline salts of the emulsion, and then drawing off or decanting the solvent carrying the fatty matter from above the heavier mass from which it has been separated, substantially as specified.

3. The process of decomposing the emulsion resulting from the cleaning of wool by volatile solvents which consists in subjecting said emulsion to the action of an acid whereby the acid combines with the alkaline salts of the emulsion, and thus free the solvent carrying fatty matter, and then separating the solvent and fatty matter by distillation, substantially as specified.

4. The process of decomposing the emulsion resulting from the cleaning of wool by volatile solvents which consists in subjecting said emulsion to the action of an acid whereby the acid combines with the alkaline salts of the emulsion, then drawing off or decanting the solvent carrying the fatty matter, and then separating the solvent and fatty matter by distillation, substantially as specified.

5. In the art of separating volatile fat sol-



vents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying said solvent solutions by treating them before distillation with suitable saline solutions.

5 6. In the art of separating volatile fat solvents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying said solvent solutions by treating them before distillation with suitable acid saline solutions.

10 7. In the art of separating volatile fat solvent from oily, greasy, waxy or soapy solutions of the same, the method of clarifying said solvent solutions before distillation which consists in removing the water therefrom by the addition thereto of a suitable chemical.

15 8. In the art of separating volatile fat solvent from oily, greasy, waxy, or soapy solutions of the same, the method of clarifying said solvent solutions before distillation by the removal of water therefrom by the addition thereto of a chemical miscible with the water in said solutions.

20 9. In the art of separating volatile fat solvent from oily, greasy, waxy or soapy solutions of the same, the method of breaking up or decomposing an emulsified or turbid solvent solution of said fatty, oily, waxy or soapy compounds by a suitable acid saline solution.

25 10. In the art of separating volatile fat solvent from oily, greasy, waxy, or soapy solutions of the same, the method of breaking up or decomposing an emulsified or turbid solvent solution of said fatty, oily, waxy or soapy compounds by sulfuric acid.

30 11. In the art of separating volatile fat solvent from oily, greasy, waxy or soapy solutions of the same, the method of clarifying and purifying said solvent solutions which consists in adding thereto a suitable chemical or chemicals and then allowing the mixture to stand and stratify, substantially as described.

35 12. In the art of separating volatile fat solvent from oily, greasy, waxy or soapy solutions of the same, the method of clarifying and purifying said solvent solutions which consists in adding thereto a suitable chemical or chemicals and then allowing the mixture to stand and stratify in a completely-closed receptacle, substantially as described.

40 13. In the art of separating volatile fat sol-

vent from oily, greasy, waxy or soapy solutions of the same, the method which consists in mixing under pressure emulsified or turbid solvent solutions with a chemical or chemicals which will simultaneously remove the water therefrom and decompose the soap. 55

14. In the art of separating volatile fat solvent from oily, greasy, waxy or soapy solutions of the same, the method which consists in spraying emulsified or turbid solvent solutions with a chemical or chemicals (or vice versa) which will simultaneously remove the water therefrom and decompose the soap. 60

15. In the art of separating volatile fat solvent from oily, greasy, waxy, or soapy solutions of the same, the method which consists in mixing emulsified or turbid solvent solutions with a chemical or chemicals which will simultaneously remove the water therefrom and decompose the soap and then allowing the mixture to stratify. 65 70

16. In the art of separating volatile fat solvents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying said solvent solutions by treating them before distillation with a suitable sodium salt. 75

17. In the art of separating volatile fat solvents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying said solvent solutions by treating them before distillation with chlorid of sodium. 80

18. In the art of separating volatile fat solvents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying and purifying said solvent solutions by treating them before distillation with a suitable sodium salt and sulfuric acid. 85

19. In the art of separating volatile fat solvents from oily, greasy, waxy or soapy solutions of the same, the method of clarifying and purifying said solvent solutions by treating them before distillation with chlorid of sodium and sulfuric acid. 90

In testimony whereof we affix our signatures in presence of two witnesses. 95

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Witnesses:

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