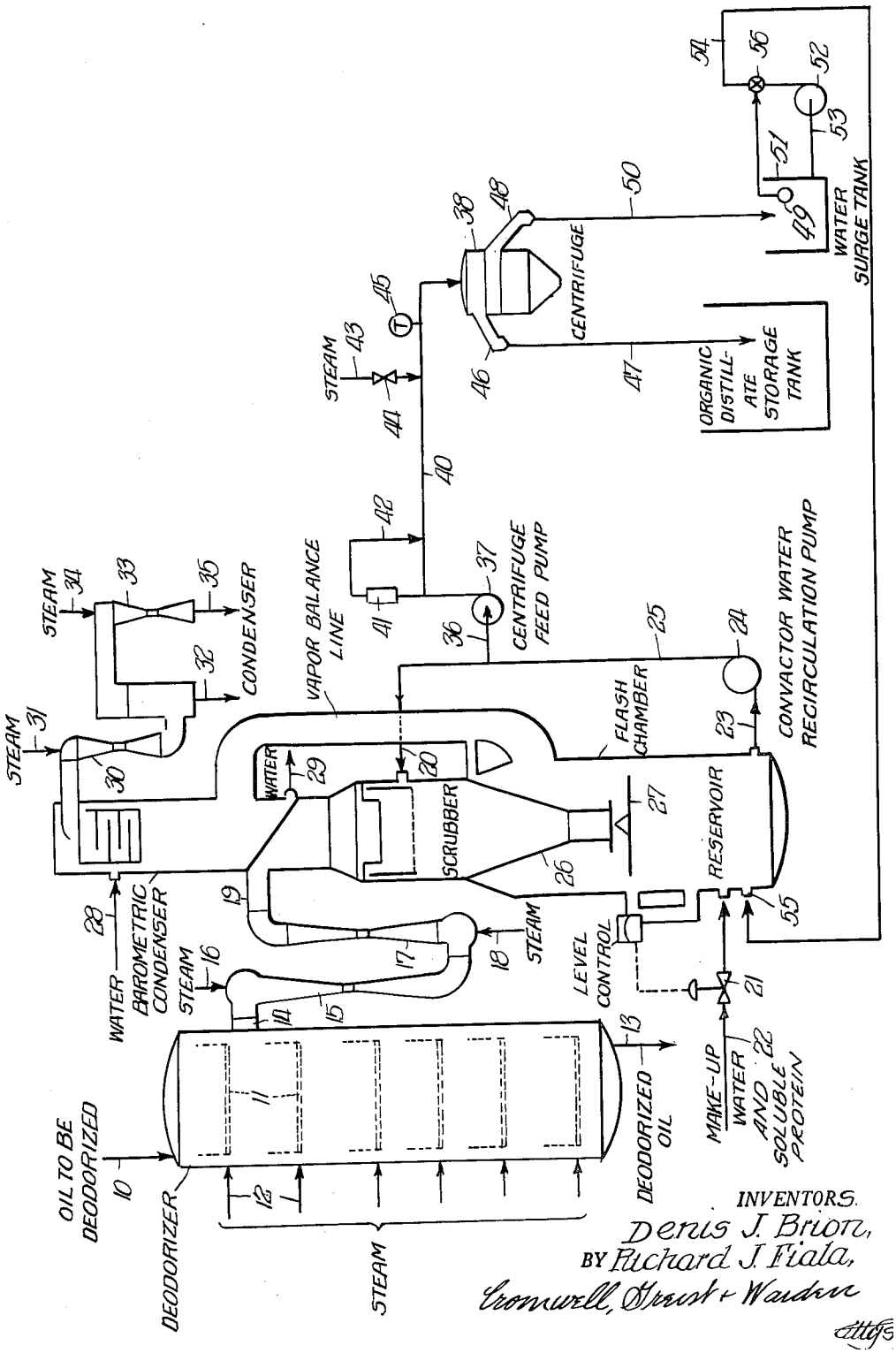
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PROCESS FOR DEODORIZING FATS AND OILS BY DISTILLATION WITH STEAM UNDER VACUUM CONDITIONS

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PROCESS FOR DEODORIZING FATS AND OILS BY DISTILLATION WITH STEAM UNDER VACUUM CONDITIONS

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This invention relates, generally, to an improved method of controlling the content of oil and fat deodorizer distillates in condensate water and to the recovery of such distillates therefrom. The invention relates more particularly to the recovery of vegetable oil distillates (e.g., soybean oil distillates) which form emulsions in the condensate water used in removing and recovering such distillates from deodorizer vapors. The use of water-soluble proteins, notably corn steep water, to break such emulsions and effect the recovery of the distillates therefrom constitutes an important feature of the invention.

Large quantities of vegetable oils are deodorized during the refining thereof in the vegetable oil industry. Such oils include soybean oil, corn oil, peanut oil, and cottonseed oil. In general, the deodorization of such oils is accomplished by introducing steam into them while they are maintained under vacuum. The high vacuum, steam deodorization of vegetable oils may be carried out either as a batch operation or in a continuous manner, the trend in the industry being toward installation of continuous deodorizers which operate under very high vacuums.

The vapors which come off during deodorization (i.e., treatment with steam and under vacuum) whether a batch or continuous process, contain so-called vegetable oil distillates (VOD) which are valuable by-products and, which if not recovered, often pose problems from the standpoint of waste disposal and stream polution. While the vegetable oil distillates are relatively easily condensed, they have posed a problem in the industry in that they form oil and water emulsions which become highly viscous and difficult to handle when the concentrations of the distillate in water exceeds about 2%. This problem of removal and recovery of vegetable oil distillates from condensate water or scrubber water is discussed by R. J. Fiala in a paper entitled "A Commercial Process for the Recovery of Deodorizer Distillates," appearing in the September, 1959 issue of the Journal of the American Oil Chemists' Society and in his Patent 3,061,622, dated October 30, 1962. In that article a commercial type of vapor scrubber was referred to which has found good acceptance in the industry for scrubbing the vapors from vacuum deodorizers so as to remove 99+% of the vegetable oil distillates therefrom. This vapor scrubber is known by the trade name "Convactor" and is manufactured by Croll-Reynolds Co., Inc., of New York, New York. This particular apparatus includes a scrubber section wherein effluent deodorizer vapors are intimately mixed with cool scrubber water with complete condensation occurring, followed by flashing of water, equivalent to the steam content of the deodorizer vapors, and subsequent delivery of the "clean" steam into a barometric condenser. The scrubber water has condensed therein the organic materials evolved during deodorization, referred to as vegetable oil distillates and comprised for the most part of sterols, tocopherols, free fatty acids (55–65%) and neutral oils. Other vapor scrubbing apparatus of known type may also be used in lieu of the so-called "Convactors."

While the "Convactors" or other distillate scrubbing and condensing equipment provides for efficient removal of the vegetable oil distillates from the deodorizer vapors, heretofore it has been not practical to operate such apparatus except at very low concentrations of the distillates

in the condensate or scrubber water. As long as the concentrations of the vegetable oil distillates in the condensate water remains low, e.g., not over about 1%, the emulsions which forms are highly fluid, pumpable liquids very much resembling ordinary milk in appearance and fluidity, but containing a small amount of curd-like flocs of the organic material. However, as the concentration of the organic material increases toward 2%, the curd-like flocs become thicker and the emulsion becomes less fluid and increasingly less pumpable. Finally, when the concentration of the distillates or oil organic material rises above about 2%, the floc becomes so thick that the emulsion no longer exhibits Newtonian flow but instead it resembles a material like mayonnaise which does not exhibit ordinary fluidity and cannot be pumped and circulated in the usual manner that water and similar fluids can.

If the emulsion is allowed to approach or reach this mayonnaise-like stage, then operation of the "Convactor" or other scrubbing equipment has to cease and the contents cleaned out before operation can be resumed with fresh water.

In the above mentioned technical paper of R. I. Fiala, a practical method of controlling the concentration of vegetable oil distillates or organic material in the "Convactor" circulating water is described which has served to avoid excessive build up of the distillate concentration in the scrubber water and to keep it at a safe operating level. Briefly, the Fiala method involves bleeding off a stream of the scrubber water from the circulating system of the "Convactor" and after heating the stream to a temperature sufficient to break the emulsion, passing it into a separator such as a centrifuge. The vegetable oil distillate separates out as the non-aqueous or oil phase and is collected while the clean water phase that separated out as the aqueous phase is returned back to the "Convactor" system. However, since it is not safe to permit the vegetable oil distillate content to build up in excess of about 1.5% by weight in the scrubber or "Convactor" water, the success of the Fiala method depends on diverting a large enough quantity of the Convactor water to the centrifuge to keep the organic level below about 1%.

It was discovered in accordance with the present invention that substantial improvements could be obtained in the treatment procedure described in the Fiala paper by incorporating in the scrubber or condensate water of the "Convactor" or similar apparatus a small amount of a water-soluble protein material, corn steep water being a preferred such material. Thus, by maintaining a concentration of from 0.1 to 0.2% water soluble protein in the scrubber or condensate water of the Convactor or other vapor scrubber apparatus the build-up of vegetable oil distillates in the scrubber water can be permitted to rise substantially above the prior safe level of 1.5% to 2%, even up to as much as 25% before the pumpable fluidity is interfered with. Furthermore, with this minor content of water soluble protein being maintained a true emulsion does not form and the vegetable oil distillate is readily separated and recovered from the condensing water.

One of the principal advantages of the present invention is that the concentrations of the vegetable oil distillates in the scrubber water can be satisfactorily maintained at or below a safe level simply by subjecting only a small stream of the condensate water to the separation treatment of this invention for say only six hours out of twenty four hours of continuous operation. Of course, with separating equipment of larger capacity this time could be further reduced. Conversely, by operating smaller equipment for longer periods the safe level can also be readily maintained.

While the present invention is particularly useful in connection with controlling the vegetable oil distillate

content and build-up in condenser or scrubber water and recovering the same therefrom, it has application to the control and recovery of deodorizer distillates of other oils and fats, including animal oils and fats.

The object of the present invention, generally stated, is the provision of an improved method of controlling the content and build-up of oil and fat deodorizer distillates in condensate or scrubber waters, and the recovery of the same therefrom.

An important object of the invention is the provision of such a method of control and recovery which is more efficient than prior methods in that: (1) higher levels of concentration of the oil and fat deodorizer distillates may be allowed to accumulate in the condensate or scrubber waters without endangering the operation of the scrubber apparatus because of an increase in the viscosity of the scrubber water beyond the pumpable stage; (2) smaller volumes of the scrubber water need to be treated for shorter periods; and (3) the cost of treating material (i.e., water soluble protein) is so small as to be almost negligible; and (4) heating costs are reduced.

For a more complete understanding of the nature and scope of the invention reference may now be had to the following detailed description thereof taken in connection with the accompanying drawing wherein, the single figure constitutes a flow diagram in connection with which one working example or embodiment of the invention will be described.

Referring to the flow diagram, the left-hand portion thereof illustrates a known type of deodorizer which operates continuously under a high vacuum, such as a Girdler semi-continuous deodorizer. Oil to be deodorized is delievered into the top portion of the deodorizer through a line **10** and is subjected to steam treatment under varying temperature conditions as it flows into and through a series of troughs **11** into each of which live steam is delivered through a series of pipes **12**. The deodorized oil is taken off at the bottom of the deodorizer through a line **13**.

The distillates and steam mixture constituting the effluent from the deodorizer is discharged therefrom through a connection **14** and moves through a first stage booster jet **15** in the form of a venturi operated by steam injected through the line **16**. The vapors then move through a second stage booster jet **17** which is operated by steam injected thereinto through a line **18**. The steam injected into the booster jets **15** and **17** aid in maintaining the high vacuum under which the deodorizer is operated and in this manner the effluent vapors of the deodorizer including incondensables, organic condensables and steam are delivered into the distillate condenser through an inlet **19**.

The distillate condenser illustrated is of the "Convactor" type described above and, by way of example, consists of a 33-foot black iron tower which is designed for a dual purpose. The first purpose of the tower is to remove the non-volatile organic material from the deodorizer effluent vapors and the second purpose is to condensate and discharge the jet exhaust steam and deodorizer blowing steam. The tower is divided into two specific sections, an upper barometric condenser section and a lower vapor scrubber section. These two sections are joined by a vapor balance line which allows steam and high volatile organic compounds in the scrubber section to "flash" to the barometric condenser.

The deodorizer effluent vapors enter the upper portion of the scrubber condenser section and are deflected downwardly through a curtain of relatively cool scrubber condensing water delivered into the scrubber section through a line **20**. The condensing water is maintained in a closed-circuit system to provide for continuous recirculation thereof through the scrubber section of the tower. This section at the bottom thereof includes a reservoir containing a supply of condensing water the level of which is maintained by a level control operating a valve **21** disposed in a make-up water supply line **22** which feeds fresh water into the reservoir for level maintenance purposes. In recycling, the condensing water is taken out from the bottom of the reservoir through a line **23** by a pump **24** placed therein and delivered through a line **25** connected with the line **20** in communication with the upper portion of the scrubber section.

Upon contact of the deodorizer effluent vapors with the relatively cool condensing water, the organic material and steam is condensed resulting in a raising of the temperature of the condensing water by the transfer of latent heat. The heated condensing water then falls through a water collecting cone **26** constituting the bottom portion of the upper section of the scrubber and in communication at the base thereof with a flash chamber. A flash plate **27** is located at the base of the cone **26** in the flash chamber to provide for efficient flashing of the more volatile materials from the condensing water. The flash chamber is in communication with the upper barometric condenser section through the vapor balance line is thus at a slightly reduced pressure as the barometric condenser operates at a slightly reduced pressure relative to the scrubber section. The more volatile materials including the incondensables will flash from the condensing water until thermodynamic equilibrium has once more been attained. This procedure, therefore, leaves only the non-volatile organics in the once again cool condensing water with this water falling into the reservoir for recirculation through the tower. The heat picked up by the condensing water is immediately dissipated by rapid evaporation or flashing of the volatiles therefrom, this evaporation process being facilitated by a rather substantial surface area of condensing water which is exposed by flashing on the cone-shaped baffle or plate **27** as the condensing water falls into the flash chamber. Since a fairly high degree of superheat is present in the vapors entering the tower, the total weight of saturated steam flashed from the warm condensing water is greater than the weight of the incoming vapors. This excess flash is continuously made up by adding water to the tower through the line **22**.

The flash vapors are relatively free of organic contaminants and rise through the vapor balance line to be condensed in the barometric condenser. The flashed material is approximately equal to the sum of the deodorizer blowing steam plus the jet exhaust steam delivered through the lines **16** and **18**. Condensing water is supplied through line **28** to the barometric condenser section and is removed through line **29** as waste or for any suitable treatment. The vacuum in the barometric condenser section is drawn by the utilization of a booster jet **30** operated by steam delivered through a line **31** and any effluent vapors taken from the barometric condenser section of the tower are delivered into a smaller condenser connected to the discharge end of the jet **30** from which liquid discharge to a drain is handled through a line **32**. Vacuum is maintained on the smaller discharge condenser by a booster jet **33** operated by steam delivered thereinto through a line **34** with the subsequent steam and vapors being discharged in any suitable manner through a line **35**, normally to the atmosphere.

The net result of the foregoing operation is the removal of the organic distillates from the deodorizer vapors and concentration of these distillates in the condensing water in the scrubber section of the tower. These distillates, including the physical entrained solids, consist of varying amounts of fatty acids, glycerides, sterols, and tocopherols. As these materials are relatively immiscible with the water in which they are condensed, the accumulation in the condensing water takes the form of an oil-in-water emulsion. Several distinct phases occur in the condensing water as the concentration of organic distillates increases from zero through 1.5%. The following description of these phases is representative of distillates recovered from the deodorization of an acetic anhydride degummed soybean oil which is break-free but con-

tains about 0.2% to 0.3% free fatty acids, such as produced according to the method disclosed in Patent No. 2,782,216.

With the type of oil described, this being generally representative of various types of fatty oils, the condensing water becomes a turbid white, resembling milk, with between 0% and 0.15% organic distillates condensed therein. The fluidity characteristics, as measured by viscosity, are not greatly different from pure water during this phase. Between 0.15% and 1.0% concentration, the organic materials begin to associated, forming solid aggregates or flocs which float on the condensing water. This phase is characterized by a gradually increasing viscosity although the thickening does not materially affect the flow properties of the water. Within the concentration range of about 1.0% to 1.5%, the flocculent organic solids become dense causing the water viscosity to increase more than a 100%. In this phase the fluidity characteristics of the water are greatly reduced. With a concentration exceeding 1.5% a gelling action occurs and the water cannot be pumped with the result that the tower becomes inoperative.

As indicated above, the present invention provides a new and improved method of controlling the concentration of organic distillates in the condensing or condensate water so as to prevent the organic distillate content from resulting in an increase in viscosity to the point that the scrubber or condensing water cannot be readily recirculated. Basic to the improved method of the present invention is the addition and maintenance of a small per cent of a water soluble protein material in the scrubber or condensing water.

Referring again to the flow diagram, a small portion of the scrubber or condensing water which is being continuously recirculated through line **25** is periodically bled off through line **36** to the inlet connection of a pump **37** which feeds a centrifuge **38**. The pump **37** discharges through a line **40** to the feed inlet of the centrifuge **38**. Line **40** is provided with a rotameter, other flow measuring device **41** connected in a by-pass line **42**. A steam injection line **43** having a control valve **44** is also connected in line **40** upstream from an indicating thermometer **45**. The oil phase discharge connection **46** of the centrifuge **38** discharges through a line **47** into an organic distillate storage tank. If line **47** is exposed to cold temperatures it should be provided with an external heater so that the stream of organic distillates may be maintained fluid at all times.

Clean water discharging from the aqueous discharge connection **48** passes downwardly through line **50** into a water surge tank **51** provided with a liquid level control means such as the conventional float control indicated **49** operatively connected with flow control valve **56** in the return line **54**. A clean water recycle pump **52** has its inlet connection connected with the bottom of the tank **51** by way of a line **53**. The discharge connection of the pump **52** communicates by way of the line **54** to an inlet connection **55** in the bottom of the reservoir portion of the Convactor. The flow control valve **56** in line **54** is regulated by the float **49** or other liquid level sensing means in the tank **51** so as to maintain the water level in tank **51** between predetermined operating levels.

The following working example will more fully inform those skilled in the art of the nature of the invention and a preferred embodiment thereof.

Example

This example is a description of a satisfactory operating procedure for the plant described in the accompanying flow diagram wherein the deodorizer is being utilized on a round-the-clock basis to deodorize soybean oils which has already been degummed so as to be break-free, by treatment with acetic anhydride in accordance with the method disclosed in Patent No. 2,782,216.

The distillate condenser tower of the Convactor operates with the condensing water in the lower scrubber section in equilibrium with the barometric condenser portion at a temperature within the range of 85 to 105° F. The body of condensing or scrubber water (e.g., 15,000–16,000 lbs.) is recirculated through the line **25** at an average rate of about 1075 gallons per minute (g.p.m.). By means of prior addition and occasional make-up as periodic (e.g., weekly) tests indicate to be necessary, a concentration in the range of from 0.1 to 0.2% by weight of protein is maintained in the condensing or scrubbing water. The occasional addition of make-up protein can be conveniently made either at the water surge tank **51** or with make-up water through line **22**. A highly satisfactory source of water soluble protein for this purpose has been found to be corn steep water or corn steep liquor, a well known material in the corn wet milling industry and described, for example, in a paper by R. Winston Liggett and H. Koffler entitled, "Corn Steep Liquor in Microbiology," in Bacteriological Reviews, vol. 12, No. 4, December 1948. So-called heavy pure steep water contains about 12.5 to 15% protein and has been used with complete success in the practice of the present invention.

By maintaining a level of concentration of from 0.1 to 0.2% of the water soluble protein in the condensing or scrubber water, it is necessary to only operate the centrifuge **38** six hours out of every twenty-four hours of operation of the Convactor. In starting up the 6-hour treatment of the condensing or scrubber water, the centrifuge **38** is first brought up to full speed. In the case of a Sharples D2 centrifuge this requires about 10 minutes time. When the centrifuge **38** is up to speed then the centrifuge feed pump **37** is started and the flow of water through the line **40** is adjusted by means of observing the rotameter **41** until it equals approximately 10–15 g.p.m. Steam is added through line **43** until the steam temperature obtains a value between 150–160° F. as indicated by the thermometer **45**. Good separation of fatty material and water is readily obtained in the centrifuge **38**.

During operation, the centrifuge discharges the oil or fatty material consisting of the organic distillates through the connection **46** into the line **47** so that they accumulate in the storage tank provided therefor. The pump **52** operates to return the separated clean water to the Convactor as it accumulates in the tank **51**, the flow rate being controlled by means of the valve **56**. The heat content of the clean water is thus dissipated in the Convactor unit.

It will be seen that a number of variations can be made in the above described example, in practicing the present invention. The following specific variations or changes will be mentioned with the understanding that still others will be suggested to, and occur to, those skilled in the art:

(a) Instead of adding steam line **43**, the liquid steam going to the centrifuge may be heated by means of a heat exchanger disposed in line **40**.

(b) The temperature of the liquid steam going to the centrifuge may be adjusted within the operable range of 100 to 250° F. The narrower range of 150–160° F. is preferred in that heating costs are low and the temperature is sufficient to keep the vegetable oil distillate melted so that separation in the centrifuge is facilitated.

(c) A centrifuge of less capacity than that of centrifuge **38** may be used to control the concentration or build-up of organic distillates in the condenser or scrubber water within proper operating range by running it for longer periods, including continuous operation if necessary.

(d) Conversely, by using a centrifuge of increased capacity, proper control over the concentration and build-up of the organic distillates in the condensate or scrubber water may be maintained while operating for shorter periods of time.

(e) Instead of operating the centrifuge 38 for 6 hours out of every three shifts, it could be operated 12 hours continuously during every six shifts.

(f) While the preferred range of concentration of water soluble protein the condensate or scrubber water is from 0.1 to 0.2% as above mentioned, it is possible to operate satisfactorily with as little as 0.05%. It is also possible to operate the system with higher concentrations, such for example as 0.5% water soluble protein.

(g) Instead of using corn steep water as the source of water soluble proteins, other source materials therefore could be used including egg albumen, gelatin, and peptone. Also water soluble proteins can be inexpensively obtained from such materials as casein and soybean meal.

The foregoing and other modifications and variations may be adopted in practicing the invention without departing from the spirit and scope of the invention as indicated in the appended claims.

What is claimed as new is:

1. In the process of deodorizing fats and oils by distillation with steam and under vacuum wherein the organic distillates are condensed in a scrubber in recirculating water in which flocs form that are emulsions of said distillates in water, said flocs decreasing the fluidity of the condenser water substantially as the concentration of distillates increases toward a value on the order of 2%, the improvement for suppressing the effect of said flocs on the fluidity of the condenser water as the concentration of distillates approaches and exceeds said 2% value that comprises incorporating and maintaining in said condenser water a concentration of water-soluble protein in the range of 0.05% to 0.5% and permitting the concentration of said distillates in said recirculating water to build up to values several times greater than 2%.

2. The method of claim 1 wherein the water from said diverted portion is returned to the condenser water.

3. In the process of deodorizing soybean oil with steam and under vacuum wherein organic distillates from a deodorizer are condensed in a scrubber in recirculated condenser water in which said distillates form viscous emulsions at low concentrations in the order of about 2%, the improvement for controlling the formation of such emulsions and for recovering the distillates from said condenser water which comprises introducing and maintaining a concentration of water soluble protein in the range of from about 0.05% to 0.5% in the recirculated condenser water to permit a build-up of up to about 25% of condensed distillates in the recirculated condenser water heating at least a portion of recirculated condenser water and condensed distillates to facilitate separation of distillate material from the water, separating the distillate material from the water, and returning the separated water to the scrubber as condenser water.

4. The method of claim 3 wherein corn steep water is used as the source of water soluble proteins.

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