

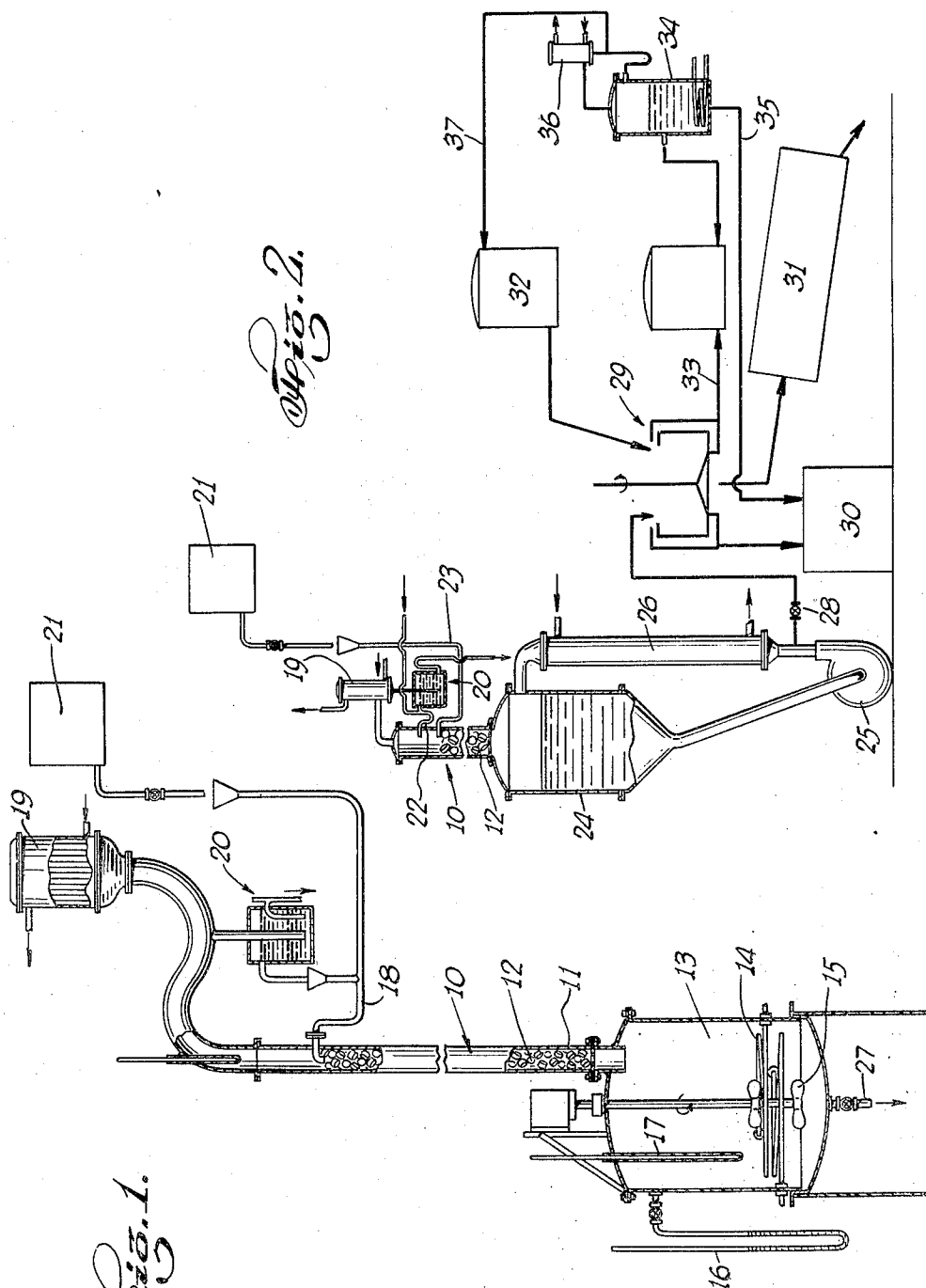
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METHOD OF EVAPORATING SOLVENTS FROM SOLUTIONS

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METHOD OF EVAPORATING SOLVENTS
FROM SOLUTIONS

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This invention relates to a method of removing volatile solvent from a solution and particularly to the method including the counter-current movement of the solution and the vapor of a liquid that is immiscible with the said solution.

Various methods of removing solvent from solutions have been known heretofore. One method, for instance, involves the distillation of water with a miscible liquid, either with or without fractionation. Another method utilizes an immiscible liquid without counter-current movement of its vapor and the solution.

We have now discovered special advantages in the counter-current movement of the solution and the said vapor.

Our invention comprises passing a solution that is to be concentrated or desolvenated (treated to remove some or all of the solvent) in a direction opposite to that of a stream of vapor of the immiscible liquid and in good contact therewith, at an elevated temperature so that a mixture of vapor of the solvent and of the said liquid is separated. In the preferred embodiment, the invention comprises the use of an apparatus of the type of a rectifying column providing the desired closeness of contact of the solution which descends through the apparatus and the ascending vapor. In this embodiment, the resulting vapor mixture is withdrawn from the upper part of the apparatus, whereas the concentrated solution or desolvenated product issues from the lower part of the apparatus.

The invention, stated briefly above, will be illustrated by description in connection with the attached drawing, to which reference is made.

Fig. 1 shows diagrammatically a side view of equipment suitable for the practice of the invention, the view being partly in section.

Fig. 2 shows, on a smaller scale, a similar view of a modified form of equipment together with a flow sheet illustrating the handling of the product subsequent to the desolvenation by evaporation.

There are shown an apparatus suitable for effecting intimate contact between the descending liquid and the ascending vapor as, for instance, the rectifying column 10, including the shell or pipe 11 and packing 12, the packing suitably consisting of lumps of coke or Carborundum or Raschig rings of iron or other material that is not readily corroded by the materials which are to be passed through the column.

At its lower end the column 10 is in communication with an evaporator 13 provided with suit-

able heating elements such as coils 14 for steam or other heating fluid and equipped with a motor driven agitator 15. The evaporator may be provided also with a pressure indicator such as the gage 16 and a thermometer well 17.

The equipment shown is particularly adapted to the dewatering of solutions of sodium hydroxide, with kerosene as the immiscible liquid. For this reason the method and equipment will be discussed in detail as utilized with these materials.

The sodium hydroxide solution and the kerosene are fed to the evaporator system, suitably continuously and at a position substantially above the bottom and preferably near the top of the apparatus 10. For instance, the solution and kerosene may be supplied at a position just above the uppermost of the filling material 12, as, for instance, through a common feed line 18, so that there is intermixture of the two liquids before they enter the column. Such premixing, however, is not necessary for all purposes.

Once the system is in operation there is present in the evaporator a slurry of kerosene or the higher boiling fractions thereof and dewatered sodium hydroxide, which slurry covers the heating elements 14 in the bottom of the evaporator. These heating elements are maintained at a temperature, as by the use of very high pressure steam or a heated liquid of high boiling point, such, for example, as diphenyl or a high boiling fraction of lubricating oil, so that the kerosene is caused to boil and the resulting vapor is passed upwardly by the force of the boiling through the apparatus 10, in counter-current direction to the descending sodium hydroxide solution. Refluxing occurs and there is good contact of the kerosene vapor with the said solution. Since the temperature of the vapor of kerosene is necessarily high, its vapor pressure and the vapor pressure of the water of the sodium hydroxide solution added together give a total that exceeds slightly the external pressure, ordinarily approximately the atmospheric pressure, so that boiling ensues.

It will be understood that, since the combined partial pressures of the immiscible liquid and the aqueous solution coact to offset the external pressure, the so-called boiling occurs at a temperature below that of boiling of either the said solution or the immiscible liquid separately.

The resulting mixture of vapors of water and kerosene is withdrawn from the top of the apparatus 10, say to a condenser 19 cooled by water.

The condensate is then passed to a separator

or decanter 20, in which the condensate is allowed to separate by difference of specific gravity into a lower aqueous and an upper non-aqueous layer. The lower layer is removed ordinarily to a waste line. The material in the upper layer is returned to the distilling system, suitably continuously to the upper part of the rectifying column.

The sodium hydroxide solution also is fed advantageously in a continuous stream to the upper part of the column, as from the storage 21.

Since sodium hydroxide solution, from electrolytic cells or other equipment in which sodium hydroxide is customarily manufactured, may readily be made of approximately 50 per cent strength, the sodium hydroxide solution supplied to the reservoir 21 is ordinarily of that strength. However, stronger or weaker solutions may be used.

Obviously, various dimensions of the apparatus 10 and of other parts of the equipment may be used. In the preferred practice of the invention, there is an excess of evaporating capacity in the system so that the sodium hydroxide which issues from the bottom of the apparatus 10 is practically anhydrous and forms a flowable slurry with the refluxing kerosene at that point. In a typical operation, the sodium hydroxide, which is collected in the evaporator 13 and there maintained in suspension in the kerosene, contains about 0.1 to 0.8 per cent water and usually not more than 0.3 per cent.

A feature of the method described is the fact that there is prevented coalescence of the particles of recovered sodium hydroxide, so that the formation of massive material is avoided, the particles of sodium hydroxide in the evaporator 13 remaining small and discrete. The method is particularly desirable, for this reason, as applied to the desolvenating of solutions of solids adapted to coalesce when particles of the solid are heated to a temperature somewhat above the boiling point of water, say 300° to 500° F.

The equipment shown in Fig. 2 contains many elements in common with those of Fig. 1, including the column 10, filling 12, condenser 19, separator or decanter 20, and the reservoir 21 for sodium hydroxide solution from which water is to be removed. The recovered kerosene is returned from the decanter to the system through the line 22 and the sodium hydroxide solution is fed through line 23.

The collecting tank 24 is disposed beneath the column. The slurry of dehydrated sodium hydroxide and kerosene in this tank is kept in continuous agitation by means of the pump 25, which forces the solution upwardly through the calandria 26 including parallel steam heating pipes and back to the tank 24.

The flow sheet, constituting the part to the right of Fig. 2, shows diagrammatically the sequence of steps in the further treatment of the slurry such as may be withdrawn from the evaporator 13 or the collecting tank 24 of Figs. 1 and 2 respectively.

The slurry may be removed through the outlet 27 (Fig. 1), either by gravity flow or pump, or through the valve 28 (Fig. 2), to the centrifugal separator 29 in which the solid sodium hydroxide is retained and the major portion of the kerosene is removed. The kerosene which is removed centrifugally is passed to a storage tank 30 for kerosene.

The sodium hydroxide from the centrifuge may be discharged directly to an evaporator or

drier 31 in which kerosene adhering to the sodium hydroxide after centrifuging is removed by evaporation at an elevated temperature, the temperature being maintained well below the point of fusion or softening of the sodium hydroxide.

Instead of centrifugal separation, the slurry may be filtered or subjected to settling and decantation, to separate the sodium hydroxide from the liquid. The sodium hydroxide so separated is further treated as described in connection with the centrifuged material.

For some purposes the removal of adhering kerosene from the centrifuged sodium hydroxide is facilitated by washing, in the centrifuge, with a small amount of a very volatile solvent for kerosene that is a non-solvent for sodium hydroxide. For this solvent wash there may be used, for example, ethylene dichloride, carbon tetrachloride, ligroin or the like, the wash solvent being supplied to advantage from a tank 32.

When the wash solvent has been used, the sodium hydroxide in the centrifuge is freed from residual volatile material, as in apparatus 31 as described previously except that only moderate temperatures of drying are necessary.

When the wash solvent is used, the mixed liquids removed by the washing is separated by difference of boiling point, into kerosene and wash solvent. Thus, the discharged liquid from the centrifuge may be passed through a pipe into storage tank 33 and then to evaporator 34 from which the kerosene issues at the bottom and may be passed through line 35 to the kerosene storage 30. The wash solvent, after passing through the condenser 36, may be returned through line 37 to the wash solvent storage 32.

Using the continuous evaporation system described in connection with either Fig. 1 or Fig. 2, there is the advantage recited above of non-coalescence of the sodium hydroxide particles which are recovered from the dewatered solution. Also there is a very large increase in the ratio of water to kerosene in the condensate which goes to the separator 20, as compared to the ratio obtained when the counter-current contacting of the kerosene vapor and sodium hydroxide solution is omitted. For instance, with the continuous system described, we have separated from the sodium hydroxide solution as much as 1.3 pounds of water for each pound of kerosene distilled, whereas when sodium hydroxide solution and kerosene are charged to the vessel 13 and distilled without any rectification or use of counter-current contact, we evaporate only about 0.66 pound of water for each pound of kerosene. The reason for this gain and attendant saving in evaporation costs, is not entirely clear, inasmuch as rectification is considered commonly not to have any such effect when the liquids being distilled are immiscible with each other.

In place of the sodium hydroxide solution there may be used other solutions that are to be desolvenated, as, for example, aqueous solutions of potassium hydroxide, zinc chloride, magnesium chloride, calcium chloride, or an alum or their hydrates in undissolved form. In place of kerosene as the immiscible liquid whose vapor is contacted counter-currently with the solution, there may be used some other immiscible material, as, for instance, the fraction of coal tar distillate beginning with pseudocumene and extending through hexamethylbenzene, phenyl propyl ether, diphenyl, chlorinated diphenyl, and/or chlorinated naphthalene, provided the material selected

is a liquid at the temperatures prevailing in all parts of the apparatus 10. For best results, the immiscible liquid selected should have a boiling point considerably higher than the boiling point of the solvent which is to be removed from the solution. With water as a solvent, for example, the immiscible material selected should have preferably a boiling point of about 300° to 500° F.

Particularly satisfactory results have been obtained when the immiscible liquid is a mixture of materials of graded boiling points, such as the mixture occurring in commercial kerosene of boiling range about 356° to 482° F. or the coal tar fraction referred to above. With such a liquid supplied to the top of the apparatus 10, the boiling point increases towards the lower part of the apparatus.

In addition to the use of the method in the removal of water from aqueous solutions, the method may be applied also to the desolvenating of non-aqueous solutions. A solution that may be substituted for the aqueous solutions described is an aqueous alcoholic solution of a substantially non-volatile substance as, for instance, glycerine, glycol, potassium acetate or the like. Using such a solution there is selected, as the immiscible liquid, gasoline, toluene, or other liquid that is insoluble in an alcoholic solution of the ratio of water to alcohol that prevails in the solution being concentrated. Another solution that may be desolvenated is a glycerine solution of a soap, kerosene or other high boiling immiscible liquid being used to supply the vapor for counter-current contact with the solution.

The method described may be modified for some purposes by operating the evaporating system, including evaporator vessel, calandria (if used), apparatus 10, and the condenser 19 or 21 under reduced pressure. Thus, there may be used a pressure corresponding 2 to 15 inches of mercury or a vacuum of 15 to 28 inches.

It will be understood that the term "immiscible" is used herein to mean insoluble or soluble to a small extent only in the solution being desolvenated.

The term "substantially non-volatile" means giving little or no vapor pressure at the temperature of distillation. With a solution of a substantially non-volatile material, no appreciable fractionation takes place, therefore, between the solute and solvent.

It will be understood, also, that the details given are for the purpose of illustration, not restriction, and that variations within the spirit of the invention are intended to be included within the scope of the appended claims.

What is claimed is:

1. In removing water in a continuous process by evaporation from an aqueous solution of a non-volatile material that is a solid at ordinary

temperatures and at temperatures of approximately 300° to 500° F. but undergoes coalescence causing adherence of its contacting particles when heated in the presence of water vapor to a temperature between approximately 300° and 500° F., the method which comprises passing the solution of the said material downwardly through a fractionating column adapted to effect intimate contact between a liquid and a vapor, simultaneously passing upwardly through the column the vapor of a liquid that is immiscible with the said solution, maintaining the solution and vapor thus brought into contact at the temperature of boiling of the mixture so that boiling and fractionating ensue, condensing the said vapors in part and returning the condensate so formed down the fractionating column so that the immiscible liquid is in intimate contact with the aqueous solution in the column, withdrawing from the upper part of the column the remaining fractionated mixture of vapors of water and the immiscible liquid, condensing the withdrawn mixture of vapors, separating the resulting condensate into an aqueous and a non-aqueous layer, returning the non-aqueous layer to the fractionating column, withdrawing from the lower part of the column a slurry containing the immiscible liquid and the non-volatile solid material in substantially completely dewatered condition, and separating the said dewatered material from the slurry.

2. The method described in claim 1, the said non-aqueous layer being returned to the fractionating column at a position near the top of the column.

3. The method of claim 1, including maintaining the elevated temperature in the rectifying column by heating a supply of the immiscible liquid to boiling and passing the vapor of the said liquid upwardly through the column against the descending solution to be dewatered, maintaining the temperature within the bottom of the column substantially at the temperature of boiling of the immiscible liquid there present, and withdrawing the said substantially non-volatile material from the lower part of the column as a practically anhydrous mixture with a portion of the said liquid.

4. The method of claim 1, the solid adapted to coalesce being sodium hydroxide, the immiscible liquid being kerosene, and the sodium hydroxide being collected in the kerosene below the column in practically anhydrous condition.

5. The method of claim 1, which includes maintaining a reduced pressure in the rectifying column.

6. The method of claim 1, the solid adapted to coalesce being an alkali metal hydroxide.

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