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FREEZE DRYING SYSTEM

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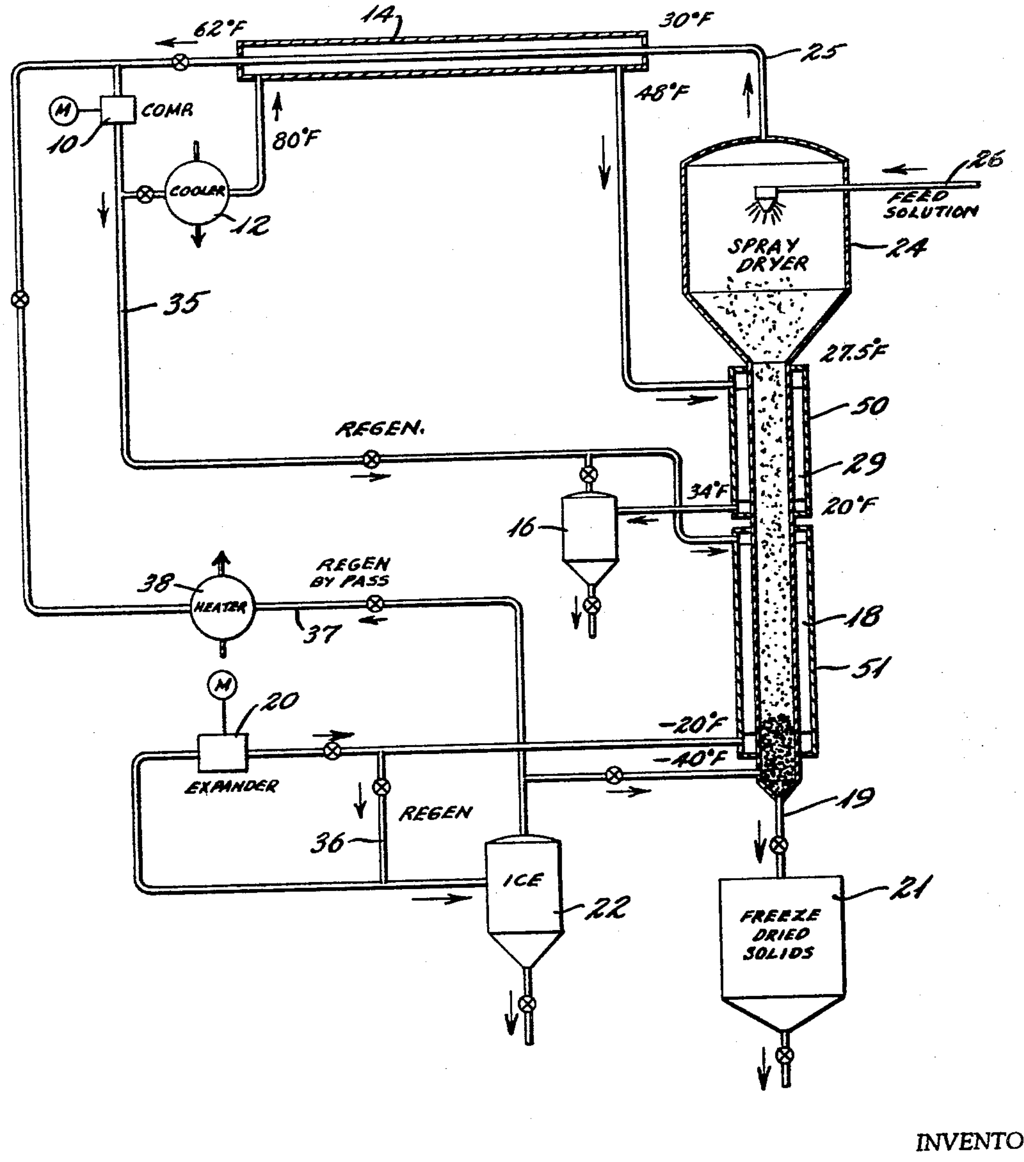

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FREEZE DRYING SYSTEM

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ABSTRACT OF THE DISCLOSURE

A freeze drying system characterized by direct freeze drying contact between liquid feed and a chilled carrier gas at sub-atmospheric pressure. The carrier gas recirculates through a cycle in which the carrier gas and the vapors evolved by the freeze drying are compressed, then progressively chilled to a sub-freezing temperature level. The evolved vapors condense during the course of chilling, either being removed as a liquid condensate or deposited in frozen state in the system. The chilled compressed carrier gas is expanded to further reduce the temperature level, then is employed to chill the compressed carrier gas by indirect heat exchange therewith and to freeze dry the liquid feed by direct contact therewith. Periodically, the feed is halted, and the system is regenerated by flowing relatively hot gas directly from the compresser through the ice laden portions of the system to melt frozen condensate.

The present invention relates to a freeze-drying system of high efficiency with low power requirements.

Conventionally freeze-drying procedures are affected by subjecting the starting material to high vacuum conditions and to temperatures sufficiently low to freeze the material. Sublimation reduces the water content of the frozen material to the desired freeze-dried level. The evolved water vapor is compressed and thereafter condensed, usually to solid ice, in a refrigerated chamber.

Heat transfer to the frozen solid is poor largely because the thermal conductivity of the gas is negligible at the relatively low vacuum which is maintained in the freeze dry chamber, and because all heat necessary for sublimation of the ice must be conducted through the frozen solid. In consequence, such freeze dryers ordinarily operates at a small fraction even of the equilibrium vapor pressure of ice, the absolute pressure frequently being as low as tenths or hundreths of a millimeter Hg. Maintenance of freeze drying conditions and removal of the evolved water vapor involves high compression ratios (in the thousands).

Characteristically conventional freeze drying features require considerable power, time and labor, making for relatively high cost operation. The principal industrial applications of freeze drying, therefore, are relatively high priced materials, e.g., penicillin.

The principal object of the present invention is to provide an efficient low cost freeze drying technique.

Further objects and the advantages of the present invention will become apparent from the description which follows.

Briefly stated the present invention involves freeze drying a pre-chilled liquid feed material by contact with a cold recirculating inert gas. The gas and evolved water vapor is warmed to about ambient temperature, then compressed to 1.25–3.0 times the absolute pressure of the freeze drying step. Thereafter the compressed gas is chilled progressively to a suitably low subfreezing temperature. During the course of chilling, the evolved water vapor content in the recirculating inert gas is condensed (principally to ice). The chilled, now cold, gas is then expanded essentially adiabatically in an expander engine to the operating pressure of the freeze drying step. The

expansion serves to recover an appreciable fraction of the power originally required to compress the gas and to lower the gas temperature still further. The temperature differential achieved by low temperature expansion provides the necessary temperature differential to operate the entire freeze drying system.

The expanded cold gas is warmed to about 10°–30° F. by heat exchange against the higher pressure gas being progressively chilled thereby; thereafter the still cold gas directly contacts the material to be freeze dried, removing its water content therefrom as water vapor. Finally the gas and water vapor is again passed in heat exchange with the freshly compressed gas, thereby being warmed to the about ambient temperature of compression.

Employment of the above described inert gas cycle improves thermal conductivity of the gas phase, significantly increasing the sublimation rate of ice. The effluent gas and water vapor leaves the freeze dry chamber at a temperature near the freezing point with a water vapor partial pressure of several millimeters, approaching even the theoretical of 4.58 millimeter Hg. In addition, a surprisingly low compression ratio, e.g., 1.5, is all that is required to operate the freeze drying system. Moreover, as much as half of the work of compression is recoverable in the expander engine. Overall the power requirements of the present system are almost negligible compared to other freeze drying procedures.

Potentially the present procedure is applicable to all concentration problems where prolonged exposure to temperature levels at which the solvent (it need not be water) in liquid phase has deleterious effect on the concentrated product. In food products, dehydration in the continued presence of water (liquid phase) frequently alters flavor or taste; in essence or perfumes so drying affects the aroma; and in vitamins and pharmaceuticals drying at liquid phase temperatures frequently results in some product decomposition with loss of potency.

For further understanding of the present invention reference is now made to the attached drawing wherein is shown, diagrammatically, a flow sheet embodying a mode of the present procedure and which is described below in terms of a specific example of a preferred embodiment of practice thereof. For clarity the exemplary temperature levels have been illustrated on the drawing.

Recirculating inert gas, suitably air (62° F.) is compressed from 0.5 atmospheres absolute to 0.75 atmospheres absolute in compressor 10 (at the rate of 12,100 lbs. per hour). A major portion of the heat of compression is removed (by heat exchange against cooling water) in cooler 12, e.g., cooled from 110° F. to 80° F. The recirculating compressed air will be about only 35% saturated (with water vapor) under these conditions. It is cooled to 48° by exchange against the effluent from the spray dryer in exchanger **14** and then cooled further in the upper section **50** of exchanger **18** to 34° F. where it is removed, passed through settling tank **16** to remove liquid water and then returned to exchanger **18** for further cooling in lower section **51** to −20° F. During this stage of the cooling, ice will build up rapidly in heat exchanger **18** since the bulk of the water vapor initially present in the recirculating compressed gas is removed in this section **51** of heat exchanger **18** during the course of chilling the gas from about 34° F. to the subfreezing level (−20° F.). In the present exemplary instance 10.9 lbs. per hour of water are removed from settler **16**, and 61.8 lbs. per hour of ice build up in lower section **51** of heat exchanger **18**.

The chilled gas leaving heat exchanger **18** is expanded under load in expander engine **20** to approximately 0.5 atmospheres (absolute). The work recovered in expander engine **20** is employed to supply some of the load of the compressor **10**. The snow formed during the essentially adiabatic expansion in expander engine **20** is collected in settling tank **22** (2.3 lbs. per hour) and the expanded cold air (now about −40° F.) passed back into heat exchanger **18** wherein the cold gas is in countercurrent heat exchanger relation to the higher pressure air being chilled. As shown in the drawing, heat exchanger **18** is constructed so that the cold gas passes also in countercurrent direct contact with the freeze dried solids. The freeze dried solids are withdrawn via outlet **19** to collection chamber **21**. From collection chamber **21** the freeze dried solids can be removed intermittently without destroying the partial vacuum (½ atm.) inside heat exchanger **18**.

The cold gas stream passing through heat exchanger **18** is warmed by indirect heat exchange against the higher pressure gas stream and then by direct contact with the freeze dried solids causes the ice to sublime into the gas stream. A small amount of heat is also transferred directly to the solids to assist in the sublimation of the ice. Ultimately, the gas stream leaves heat exchanger **18** and enters spray chamber **24** warmed from −40° F. to 27.5° F.

Desirably, the liquid feed solution (e.g. 100 lbs. per hour of coffee extract, 75% water, 25% solids) entering spray chamber **24** via line **26** has been pre-chilled, e.g., to about 40° F. The liquid feed spraying into chamber **24** is quickly cooled by evaporation and by direct heat transfer from the low pressure gas stream passing through chamber **24**. The air and evolved water vapor leaves spray chamber **24** via line **25** at about 30° F. The frozen solids pass down through heat exchanger **18** in direct contact with the colder gas ultimately leaving the system at the rate of 25 lbs. per hour via outlet **19** in freeze dried state at about the temperature of the incoming cold gas (i.e., −40° F.).

The gas 12,100 lbs. per hour air, 76.9 lbs. per hour of water vapor leaving spray chamber **24** via gas outlet **25** passes through heat exchanger **14** wherein it is warmed to about 62° F. by heat exchange against compressed gas, then to compressor **10** to repeat the gas cycle.

As has already been indicated the present system provides for rapidly freeze drying a solution with relatively high efficiency. Typically, the gas leaving the spray chamber **14**, e.g., air, 90% saturated with water vapor, contains about 0.01 mol fraction of water vapor therein, a partial pressure of several millimeters Hg, a value which approaches the theoretical 4.58 millimeter Hg.

A distinct advantage of the present system is its relative lack of sensitivity to minor unbalances. Thus, for example, in small scale units where appreciable heat may leak into the system the refrigeration obtained by expansion of the chilled gas in expander engine **20** will create a smaller temperature differential (than 20° F.) between the high and the low pressure gas streams. However, since the minimum temperature differential theoretically required for operation of the present freeze dry system is only 6° F., the heat leak will not seriously affect operation. As a practical matter, the affect of heat leakage is to change the operational period before regeneration is required to remove ice deposited in the high pressure side of heat exchanger **18**.

Mention has already been made of that much of the water vapor taken up in the gas cycle will be deposited out as ice on the high pressure side of heat exchanger **18**. As ice builds up there the outlet pressure and temperature of the gas being chilled are affected until freeze dry operations must cease, if only because ultimately ice deposition would block gas flow altogether. Practice of the present invention includes halting freeze dry operation periodically for regeneration purposes. Regeneration is affected by shutting off the liquid or slurry feed line **26**, then sending the hot compressed gases via by-pass lines **35** directly from compressor **10** to the lower section of the heat exchanger **18** (by passing the cooler **12**, heat exchanger **14** and the upper section of heat exchanger **18**). Within a few minutes, e.g., 10 minutes, the hot gas will melt all of the ice in exchanger **18** and the snow collected in settler **22** so it can be drawn off via line **36** to settler **22** as liquid water. The cold gas from settler **22** is heated and recycled to the compressor via line **37**, heater **38**. The system may be started up by once again sending the circulating gas through cooler **12** and heat exchangers **14**, **18** and expander engine **20** and then when temperatures have reached the desired level once again introducing the feed material from inlet line **26**. Practice of the current invention on an industrial scale specifically contemplates having five or six units in parallel (not shown) so that continuous operation can be obtained with one of the units always on regeneration. Also a vacuum pump (not shown) is needed to create the desired sub-atmospheric pressure levels in the system.

The heat transfer surfaces provided in heat exchangers **14** and **18** should be of the extended surface type with finning virtually essential in section **51** on the high pressure side of heat exchanger **18**. Fins provide a sufficiently large surface area for substantial deposition of snow or ice formed during cooling of the high pressure stream before the surface becomes so insulated by the ice as to reduce heat transfer rates to uneconomic limits. If small fin tubes are employed, lower section **51** of exchanger **18** will become plugged in a relatively short time and automatically shut down the system by reducing the pressure differential on the expander engine **20**. By way of example, fin tubes about 4 inches in diameter can operate for about an hour before ice plugging will halt operation. It should also be recognized that the exchange surface provided can be in excess of that normally required for noncondensing service since, as the surface becomes coated with solid it becomes insulated and the condensation automatically progresses to the following area of surface.

Ordinarily the recirculating carrier gas will be air but when the material to be spray dried is susceptible to air oxidation, employment of a more inert gas may be advisable. Such gases include, for example, nitrogen, the noble gases (helium, neon, argon, krypton, xenon), or common gaseous refrigerants such as the well known Freons. Each have their special advantages. The noble gases have a lower specific heat than the Freons or nitrogen, but in the case of helium this is partially offset by the increased diffusivity therein of water vapor. On the other hand the higher molecular weights of Freons give more efficient operation in a centrifugal compressor, requiring less stages, and this feature is the major advantage of the Freons, assuming of course that they are nonreactive with the freeze dried product. Nitrogen is the least expensive gas, but is intermediate in its other properties between helium and the Freons. The prime requirement for the carrier gas is, of course, that it be inert to the freeze dried product.

In specific application the procedure of the present invention may be employed in the freeze drying of food products, which can be made to retain their natural flavor thereby, e.g., coffee, milk, orange juice, other citrus fruits. The present procedure is contemplated also for the drying of fruits, vegetables and eggs.

The present procedure is not limited to freeze drying of aqueous solutions only. Other volatile solvent solutions may be dried in the same manner, even with solvents which do not solidify to ice at the desired minimum operating temperatures. Where freezing is involved, the heat exchanger **18** is appropriately divided as illustrated to remove liquid condensate (e.g. water) from its upper section **51** at just above the freezing point. If the solvent does not freeze at all, the heat exchanger **18** need not be divided and periodic regeneration unnecessary. The drying system could then be a continuous operation.

As has already been indicated, a principal advantage of the present procedure is its low power requirements.

Operating between ¾ atm. and ½ atm. expander engine **20** recovers about 40% of the power input to the process, and in consequence only about 0.8 kwh. is required per pound of water removed from the feed solution. Operation with the low pressure side at atmospheric pressure and the high pressure side at 1.5 atmospheres absolute, about doubles the power requirement. On the other hand, operating at lower pressures, i.e., 0.25 atmosphere at the low pressure side and 0.375 atmosphere on the high pressure side reduces the power requirement to about half the above value. Accordingly, even with equipment available on an off-the-shelf basis, freeze drying according to practice of this invention costs far less than conventional spray drying. If equipment can be designed to operate at sufficiently reduced pressures operational costs may be reduced virtually to that of boiling water in a single effect evaporator.

It will be obvious to those skilled in the art that various changes may be made without departing from the spirit of the invention and therefore the invention is not limited to what is shown in the drawings and described in the specification, but only as specified in the appended claims.

What is claimed is:

1. A regenerative freeze dry process which comprises:

(1) recirculating a carrier gas through a cooling-warming cycle wherein compressed gas is progressively chilled in the cooling part of the cycle from about ambient temperature to a sub-freezing temperature in heat exchanger means by heat exchange against expanded gas, then expanded to further cool the gas, the expanded gas being progressively warmed in the warming part of the cycle by heat exchange against the compressed gas to about ambient temperature, then compressed for recycle;

(2) introducing liquid feed directly into the expanded gas, said gas being at a sub-freezing temperature level, the feed being freeze dried by direct contact with the cold expanded gas, the vapor evolved from the feed thereby being carried along in the expanded gas through the warming part of the cycle, compression and the cooling part of the cycle to ultimate condensation during the course of said progressive chilling of the compressed gas;

(3) periodically halting introduction of the feed and warming the sub-freezing portion of the heat exchanger means to melt condensate ice deposited therein and removing the liquid condensate.

2. The process of claim **1** wherein liquid condensate is removed from the compressed gas being cooled at a temperature level close to the freezing point thereof, just prior to further chilling of the compressed gas to sub-freezing temperatures.

3. The process of claim **1** wherein the warming-cooling heat exchange is effected in stages, the first of the stages chilling the compressed gas not below the freezing point of the liquid in the feed and wherein the feed is introduced between stages.

4. The process of claim **1** wherein the compression ratio is about 1.5.

5. The process of claim **1** wherein the feed material is pre-chilled close to the freezing point of the liquid in the feed.

6. The process of claim **1** wherein the heat of compression of the compressed gas is removed by an external source of cooling and wherein the regenerative warming is effected by passing hot compressed gases directly to the normally sub-freezing portion of the heat exchanger.

7. The process of claim **1** wherein the feed is sprayed into the expanded gas.

8. The process of claim **1** wherein water is the liquid in the feed.

9. The process of claim **1** wherein air is the carrier gas.

10. A drying process which comprises:

(1) recirculating a carrier gas through a cooling-warming cycle wherein compressed gas is progressively chilled in the cooling part of the cycle from about ambient temperature to a low temperature by heat exchange against expanded gas, then expanded to further cool the gas, the expanded gas being progressively warmed in the warming part of the cycle by heat exchange against the compressed gas to about ambient temperature, then compressed for recycle;

(2) introducing liquid feed directly into the expanded gas, said gas being at a low temperature level, the feed being dried by direct contact with the cold expanded gas, the vapor evolved from the feed thereby being carried along in the expanded gas through the warming part of the cycle, compression and the cooling part of the cycle to ultimate condensation during the course of said progressive chilling of the compressed gas; and

(3) removing the vapor condensate.

11. The process of claim **10** wherein the compression ratio is in the range of about 1.25–3.00.

12. The process of claim **1** wherein expansion is effected under load.

13. The process of claim **1** wherein the pressure level of both compressed and expanded gases is sub-atmospheric.

14. The process of claim **3** wherein liquid condensate is removed from the compressed gases in the second of the stages of said warming-chilling heat exchange just prior to the chilling of the compressed gas from just above freezing to sub-freezing temperature levels.

15. The process of claim **1** wherein directly after expansion thereof the expanded gas is partially warmed to a still subfreezing temperature level prior to introduction of the liquid feed thereinto.

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