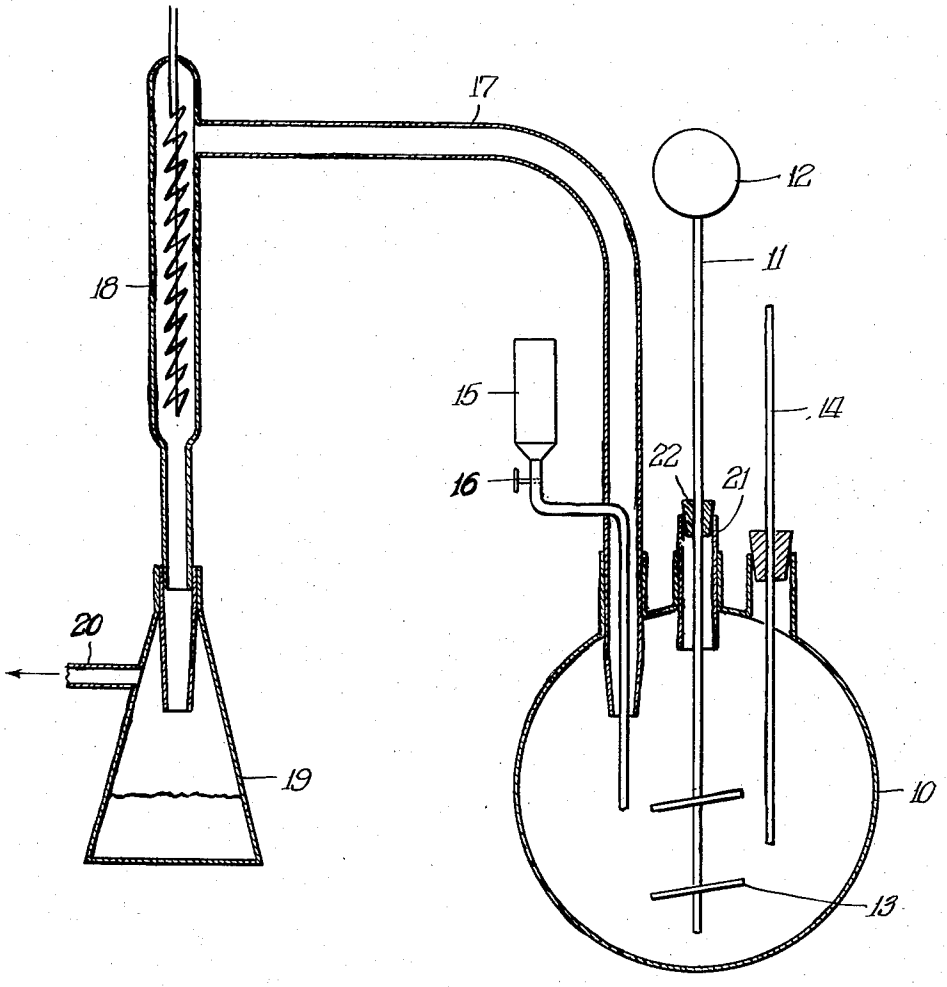
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DRYING OF MATERIALS

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DRYING OF MATERIALS

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This invention relates to the drying of materials, and is particularly concerned with a process for drying organic material which are deleteriously affected by high temperatures.

In removing moisture from organic materials it is essential in many cases to do so under conditions which preserve as many of the physiologically active constituents as possible and which avoid other objectionable changes. The art has long desired a more efficient way of drying without causing destruction or rearrangement in the materials themselves, such as denaturation of protein constituents or inactivation of physiologically active substances like the active factors present in animal glandular materials.

It has been customary in the past to dry the organic materials, for example, by treating at low temperatures with air which has been previously dried, or by subjecting the materials to heat under a vacuum, or by drying them in a current of warm air. These methods have required long drying times and the products obtained have disadvantages. For example, when the material is spread out on trays and dried in a vacuum oven, the necessary heat usually destroys at least a part of the wanted characteristics of the material. Furthermore, the color of the material, in the case of organic substances, usually darkens greatly and objectionable bacterial action frequently takes place. Also, in the usual drying operations considerable effort is required to scrape the dense, dried product off the trays at the end of the drying operation.

It is an important object of our invention to produce dried materials which exhibit as little change as possible, other than loss of moisture, in the starting constituents. A further object of our invention is to provide processes for drying heat-labile materials in a very rapid and efficient manner. Another object of our invention is to preserve the color of the starting material and to prevent darkening thereof. Many other objects and advantages will be made apparent as the description proceeds.

By the process of our invention, the material to be dried is immersed in a liquid heat transfer medium, such as xylene or kerosene, and preferably is well dispersed throughout the body of this liquid medium. Then while so held in the medium the temperature and pressure are regulated to distill off the moisture from the material. We prefer to use a liquid heat transfer medium having a boiling point above the boiling point of water, and in this case the temperature employed may be regulated so as to distill off moisture but not to distill excessive amounts of the liquid medium. Then the medium may be separated from the material after drying. The invention can, however, be practiced using a heat transfer medium having a boiling point below the boiling point of water, in which case it is desirable to add further quantities of medium during the drying period. We find a marked advantage in the use of a vacuum where heat sensitive organic materials are being dried.

One type of apparatus which may be used in practicing the invention is illustrated schematically in the accompanying drawing.

In the drawing, the apparatus includes a flask 10, suitably of glass, which may be 3-necked as shown. A shaft 11 fitted with agitator blades 13 enters flask 10 through a glass tube 21 fitted with a stopper 22 which provides a hermetic seal for the flask. An electric motor, or other suitable means 12, is provided to activate shaft 11. A funnel 15 serves to feed material into flask 10 and may be closed off by the stopcock 16. A tube 17, fitted with a hermetic seal to flask 10, leads to the condenser 18. The receiving flask 19, also hermetically fitted to condenser 18 has tube 20 leading to a vacuum line or to a means for reducing the pressure in the system. Heat or refrigeration may be imparted in any suitable way, as by placing the flask in a bath of water having controlled temperatures.

The following specific examples will serve more clearly to illustrate the process of our invention as carried out using the type of apparatus illustrated:

Example 1

30 grams of posterior lobes of pituitary glands are minced and emulsified with 100 cc. of a heat transfer medium consisting of xylene. 500 cc. of xylene are place in distillation flask 10 and the stirring device is started. The emulsion of minced glandular material and xylene is then allowed to flow into flask 10 by way of funnel 15 while stirring is continued. After all of the xylene has been added, stopcock 16 is closed and tube 20 is connected with the vacuum line, whereupon the pressure in the system is reduced to 12 mm. of mercury. At this pressure, distillation is carried on at 25° C. for one hour. At the end of this time no more moisture is observed to distill over and the residual material in the flask is filtered to recover the solid residue. The residue is air-washed by maintaining a suction in the filter flask for a time to remove the odor of xylene. The powdery residue is a light pinkish

color and assays 110% when compared with standard posterior pituitary powder prepared according to the U. S. Pharmacopoeia method.

Example 2

500 cc. of fresh ox bile are thoroughly mixed with 1000 cc. of deodorized kerosene and the resulting emulsion is placed in distilling flask 10 and agitation started. A pressure of 9 mm. of mercury is applied and distillation proceeds under agitation at 20° C. for two hours. At the end of this time, the process is stopped and the contents of the flask are filtered through paper. The solid residue on the filter is washed with hexane and is recovered as a light yellowish-green product which is easily powdered and is completely soluble in water, indicating that no denaturation has taken place.

A great variety of materials may be dried by the process of our invention and we find it especially advantageous for drying organic and biological substances which are unstable toward heat and which are ordinarily very difficult to dry. For example, we find it especially suitable for drying glandular material such as pancreas, pituitaries, ovaries, stomachs and the like and for drying organic liquids such as bile, plasma, aqueous solutions of organic materials and the like. The process is useful for drying solids and liquids of widely varying character and we do not wish to limit the invention to any kind of material to be dried.

Where the material to be dried is desired in the form of a dry powder, the material such as plant or animal organic substances is ground before being added to the liquid medium. In general, the more finely the material is ground the better for the drying procedure.

The liquid heat transfer medium employed may be any suitable liquid other than water and where the medium is to be separated from the dry material, should preferably be a liquid which is easily separated from the material and which will not leave an objectional odor. Where material such as animal glands or other material containing delicate destructible factors is dried, care must be taken not to select a medium which will by its chemical constitution be injurious to such factors.

Though the invention can be practiced using a water-miscible medium, it is much to be preferred that the liquid medium used be water-immiscible. Especially when drying organic materials like bile, glands and the like, we recommend the use of water-immiscible organic liquids, such as toluene, xylene, paraffin hydrocarbons, such as kerosene, Skelly Solve, and the like. Alcohols, ketones, halogenated hydrocarbons, and esters may also be used. The solvents such as toluene, xylene, kerosene, ketones, etc. are all volatile liquids and by "volatile" we mean they can be vaporized without chemical breakdown using heat and an ordinary vacuum such as 10 mm. of mercury.

When the material being dried contains fat desired to be removed, fat solvents may be used as the liquid heat transfer medium. By the term "fat solvents" we mean to include those liquids in which fats are soluble but do not mean to include fats themselves. These solvents dissolve fatty constituents and are removed by filtration after the distillation process. Thus the fat has been removed without practicing any more steps than are employed in the regular procedure of drying. We can, however, evaporate off the solvent and recover the fatty constituents with the other solids. Alternatively, we can separate the solvent from the solid residue and evaporate the solvent to recover separately the materials which have been dissolved therein.

The liquid medium desirably should have a boiling point above that of water at the pressure at which the operations are performed. However, as shown above, a medium having a lower boiling point than that of water may also be used. In carrying out the process in an apparatus such as shown, such lower boiling solvent is supplied, as needed, through the funnel 15 until the distillation of moisture is complete. When a high boiling solvent is used, the moisture will have been removed before the solvent begins to distill over, and the appearance of solvent in the condenser or receiver indicates the end of the drying process. Any solvent which distills off may be recovered and used again.

Especially when drying heat sensitive organic materials we prefer to maintain a high vacuum on the mixture during drying. We usually employ as high vacuum as is practicable with the equipment used. Using such a vacuum we are able to produce distillation of moisture from the material at room temperatures, or at temperatures only slightly above room temperature.

The temperature employed should be at least as high as necessary to distill water from the mixture and in the case of heat sensitive materials should be lower than the temperature at which the materials are deleteriously affected. In most cases the temperature should not be so high as to cause distillation of substantial amounts of liquid heat transfer medium, but in some cases it is desirable to raise the temperature so that after distillation of the moisture the liquid medium is also separated from the dried material by distillation.

The agitation of the material in the liquid medium during drying is an important feature of the invention. In the specific examples given, the agitation has been continuous throughout the drying period, but the purpose may be served, though less effectively, by intermittent agitation at frequent intervals.

By practicing the present invention, the time of drying is greatly reduced, less heat is required resulting in avoidance of the destruction of heat-labile materials, and also other unwanted chemical changes such as oxidation are avoided. The final dried products have an extremely low moisture content and are easily handled. Also, there is the further advantage that the products dried in the liquid medium by this method are less hygroscopic and upon standing after drying do not readily absorb moisture from the air.

The process is also applicable to the removal of water which is present in the form of ice crystals. For example, frozen glandular material may be ground and dispersed in a liquid heat transfer medium at a temperature below the melting point of the ice present. This mixture may be subjected to a reduced pressure at a temperature of about −5° C. and the water present is removed without liquefaction in accordance with the process herein described.

It is believed that many of the advantages resulting from our process are due to having the drying material widely dispersed in the liquid medium, thus exposing tremenduously large surfaces of the material to contact with a heat transfer medium, making it easier for the moisture contained to readily absorb heat for vapori-

zation. However, we do not wish to bind ourselves to any theory of explanation.

Although in the foregoing description we have referred to the removal of water from the material being dried, the process is applicable also to the removal of other substances of this nature, such as acetone, alcohol or the like.

It is to be understood that the foregoing detailed description and specific examples have been given for explanation only. The apparatus illustrated is only one of many widely varying types which may be employed. It will be understood that many changes and modifications may be made in the practice of the processes set forth without departing from the spirit of the invention. The foregoing detailed description has been given for clearness of understanding only and no unnecessary limitations are to be understood therefrom.

We claim:

1. A process for treating a heat labile fat-containing material to dry and defat the same comprising dispersing said material in a liquid water-immiscible fat solvent having a boiling point higher than that of water, subjecting said material while so dispersed to a pressure less than atmospheric and a temperature high enough to distill water from said material at said pressure, subjecting said material and solvent to mechanical agitation while maintaining said pressure and temperature until moisture contained in the material is driven off, and separating the solvent together with fat contained therein from the remaining dried material.

2. A process as in claim 1 where said material is animal grandular material.

3. A process for drying a heat labile material in the frozen state comprising dispersing said material, while frozen, in a non-aqueous liquid heat transfer medium, subjecting said material while so dispersed to a pressure less than atmospheric and a temperature below the melting point of the frozen moisture in said material, subjecting said dispersed material to agitation while withdrawing the resulting vapors, and separating the heat transfer medium from the dried material.

4. In a process for drying a heat labile material the step of applying heat and reduced pressure to said material while it is frozen and dispersed in a non-aqueous liquid heat transfer medium and at a temperature below the melting point of moisture in the material under the reduced pressure applied.

5. A process as in claim 1 and including the step of evaporating said separated solvent to separate the fat therefrom.

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