Application Story

Application Description

0. general information

Division:	Industries
Branche:	Food
Name of application:	**Freeze drying SIHI^{dry}**
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1. Introduction

Freeze-drying, or lyophilisation, is like "suspended animation" for food. You can store a freeze-dried meal for years and years, and then, when you're finally ready to eat it, you can completely revitalize it with a little hot water. Even after all those years, the taste and texture will be pretty much the same.

Today freeze drying is regarded as the "gold standard" of drying methods, where the preservation of biological activity, flavour, aroma, and/or chemistry are important. It is used widely in the pharmaceutical, food, and food supplement industries. One well-known example is coffee. However, freeze drying, is a costly process, and few instant coffees today are freeze dried. Most are spray dried, and then agglomerated into granules. The description of instant coffees as "roasted" refers to the roasting of the coffee beans, not to the drying method. Freeze drying is widely used to preserve sensitive pharmaceutical proteins and similar molecules, such as enzymes.

Why Freeze-dry?

The basic idea of freeze-drying is to completely remove water from some material, such as food, while leaving the basic structure and composition of the material intact. There are two reasons why to do this with food :

- **Removing water keeps food from spoiling for a long period of time.** Food spoils when micro-organisms, such as bacteria, feed on the matter and decompose it. Bacteria may release chemicals that cause disease, or they may just release chemicals that make food taste bad. Additionally, naturally occurring enzymes in food can react with oxygen to cause spoiling and ripening.
  Like people, micro-organisms need water to survive, so if you remove water from food, it won't spoil. Enzymes also need water to react with food, so dehydrating food will also stop ripening.

- **Freeze-drying significantly reduces the total weight of the food.** Most food is largely made up of water (many fruits are more than 80 to 90 % water). Removing this water makes the food a lot lighter, which it's easier to transport. The military and camping supply companies freeze-dry foods to make them easier for one person to carry. NASA has also freeze-dried foods for the cramped quarters onboard spacecraft.

People also use freeze-drying to preserve other sorts of materials, such as biological samples. Freeze-dried roses are growing in popularity as wedding decorations. The freeze-drying process has also been used to restore water-damaged materials, such as rare and valuable manuscripts.

It's pretty simple to dry food, drugs and just about any other biological material. Set it out in a hot, arid area, and the liquid water inside will evaporate. The heat gives the water molecules enough energy to "break free" of the liquid and become gas particles. Then you seal it in a container, and it stays dry.
This is how manufacturers make dehydrated meals like powdered soup and baking mixes.

There are two big problems with this approach. First, it’s difficult to remove water completely using evaporation because most of the water isn’t directly exposed to air. Generally, dehydrating food in this way only removes 90 to 95 % of the water, which will certainly slow down bacteria and enzyme activity, but won’t stop it completely.
Secondly, the heat involved in the evaporation process significantly changes the shape, texture and composition of the material, in the same way that heat in an oven changes food. Heat energy facilitates chemical reactions in food that change its overall form, taste, smell or appearance. This is the fundamental purpose of cooking. These changes can be good if they make the food taste better (or taste good in a different way), but if you’re drying something so you can revitalize it later, the process compromises quality somewhat.

The basic idea of freeze-drying is to “lock in” the composition and structure of the material by drying it without applying the heat necessary for the evaporation process. Instead, the freeze-drying process converts solid water – ice – directly into water vapour, skipping the liquid phase entirely.

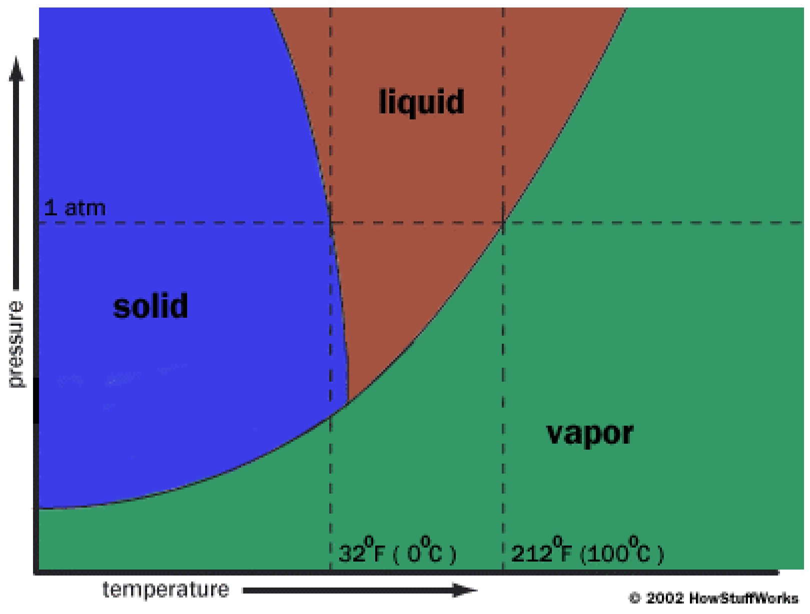
2. Glossary (Terms, Expressions)

ATM	atmospheres
K	Kelvin (293 K is room temperature)
Sublimation	The shift from a solid directly into a gas

3. The process

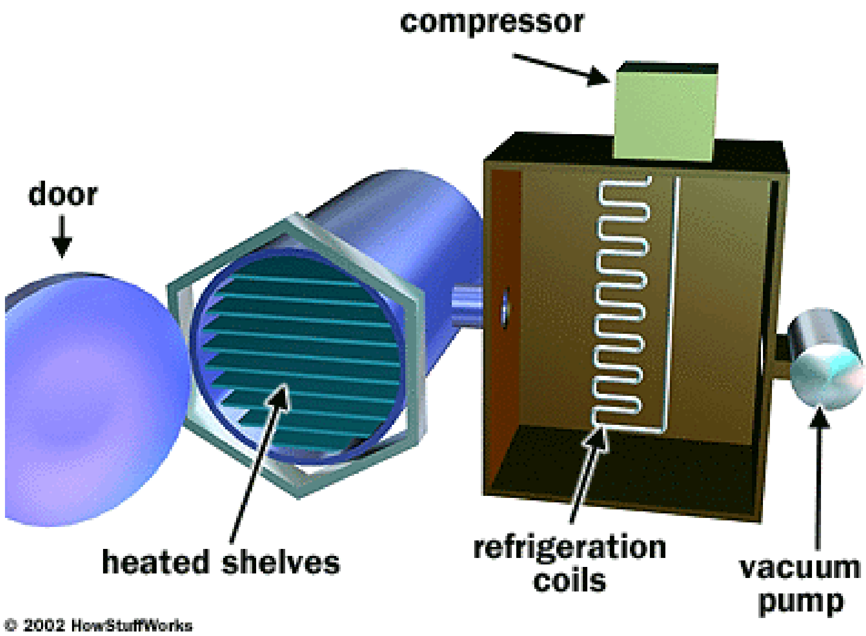
The fundamental principle in freeze-drying is **sublimation**, the shift from a solid directly into a gas. Just like evaporation, sublimation occurs when a molecule gains enough energy to break free from the molecules around it. Water will sublime from a solid (ice) to a gas (vapour) when the molecules have enough energy to break free but the conditions aren't right for a liquid to form.

There are two major factors that determine what phase (solid, liquid or gas) a substance will take: temperature and atmospheric pressure. For a substance to take any particular phase, the temperature and pressure must be within a certain range. Without these conditions, that phase of the substance can't exist. The chart below shows the necessary pressure and temperature values of different phases of water.



You can see from the chart that water can take a liquid form at sea level (where pressure is equal to 1 ATM) if the temperature is in between the sea level freezing point (0 ℃) and the sea level boiling point (100 ℃). But if you ca n increase the temperature above 0℃ while keeping the atmospheric pressure below .06 ATM, the water is warm enough to thaw, but there isn't enough pressure for a liquid to form. It becomes a gas.

This is exactly what a freeze-drying machine does. A typical machine consists of a freeze-drying chamber with several shelves attached to heating units, a freezing coil connected to a refrigeration compressor, and a vacuum pump.



A simplified freeze-drying machine

With most machines, you place the material to be preserved onto the shelves when it is still unfrozen. When you seal the chamber and begin the process, the machine runs the compressors to lower the temperature in the chamber. The material is frozen solid, which separates the water from everything around it, on a molecular level, even though the water is still present.

Next, the machine turns on the vacuum pump to force air out the chamber, lowering the atmospheric pressure below .06 ATM. The heating units apply a small amount of heat to the shelves, causing the ice to change phase. Since the pressure is so low, the ice turns directly into water vapour. The water vapour flows out of the freeze-drying chamber, past the freezing coil. The water vapour condenses onto the freezing coil in solid ice form, in the same way water condenses as frost on a cold day.

This continues for many hours (even days) while the material gradually dries out. The process takes so long because overheating the material can significantly change the composition and structure. Additionally, accelerating the sublimation process could produce more water vapour in a period of time then the pumping system can remove from the chamber. This could re-hydrate the material somewhat, degrading its quality. Considering that 1 gm of ice generates around 10000 l of water vapour at a pressure of 0.1 mbar.

Once the material is dried sufficiently, it's sealed in a moisture-free package, often with an oxygen absorbing material. As long as the package is secure, the material can sit on a shelf for years and years without degrading, until it's restored to its original form with a bit of water (a very small amount of moisture remains, so the material will eventually spoil). If everything works correctly, the material will go through the entire process almost completely unscathed!

5. Hazards and process Media

Most pharmaceuticals and biological products contain varying amounts of solvents and acid which were not entirely removed from previous extraction processes. These solvents and acids may or may not condense in the refrigerated condenser. Highly volatile alcohols and some of the light acids particularly acidic acid, find their way to the rotary pump thus causing severe pump contamination and corrosion. Although the gas ballast usually can assist in clearing some of the alcohols it is ineffective for acids. In such instances when pump contamination is experienced, regular preventative oil changes, cleaning and maintenance is the only way to ensure reasonable reliability. Alternatively, if such preventative measures are very frequent and costly, the pumps could be replaced by dry pumps or roots/dry pump combinations.

6. Main requirements for the vacuum unit

Considerations	Pump requirements	SIHI^{dry} Solution
Contamination & disposal	Dry pump	- Totally oil free
Reliability	Robust & maintenance friendly	- Non contacting/wearing parts (other than bearings) - No mechanical seals - Predicted failure analysis - Fast local service & spares
Noise	< 70 db(A)	- Non contacting screws - No motor fan - Wide clearances - No gear box
Defined vacuum process in order to meet the Qualification validation requirement	Pump has to be under 100% control	- Proven and accepted operating protocol - Serial interface
Liquid Carry-over	Liquid handling with no damage	- Torque Control - Simple cleaning operations
Corrosion	Avoidance of condensation within the pump	- Accurate temperature control within maximum and minimum limits - Closed–loop cooling in order to maintain a stable cooling. - Pre-inlet cooling/gas dilution - Automatic start & stop protocols
Dust	Dust ingress acceptability	- Large clearances - Liquid flushing & draining - Simple manual cleaning - Integrated over-load monitoring through angular difference

8. Proven solution

Guidlines

The size of refrigerated condensing area of the condenser is closely related to the dryer shelf area, and usually up to three times larger. With an optimum ice deposit thickness of 10 to 12 mm³ and more consecutive drying cycles can be operated before defrosting the condenser. Theoretically freeze drying represents ideal heat and mass transfer cycles whereby the sublimation rate in the dryer is equal to the condensation rate in the condenser, particularly if both are accommodated within a single chamber. If such an arrangement could be made absolutely leak free, the process would become self-sustaining once an initial evacuation to below the operating pressure is carried out. However, in practice the plant requires continuous vacuum pumping to overcome leaks, losses and product related constraints. The sizing of the vacuum pump can also be related back to the shelf area and a pump capacity of 50m³/h per 1 m² or 10 l product (optimum product depth 10 mm) generally gives very satisfactory results. For applications where an external pre-frozen product is handled, rapid chamber evacuation becomes necessary to avoid excessive temperature rises and or thawing and the pump size must then be determined for the required chamber pump down time.

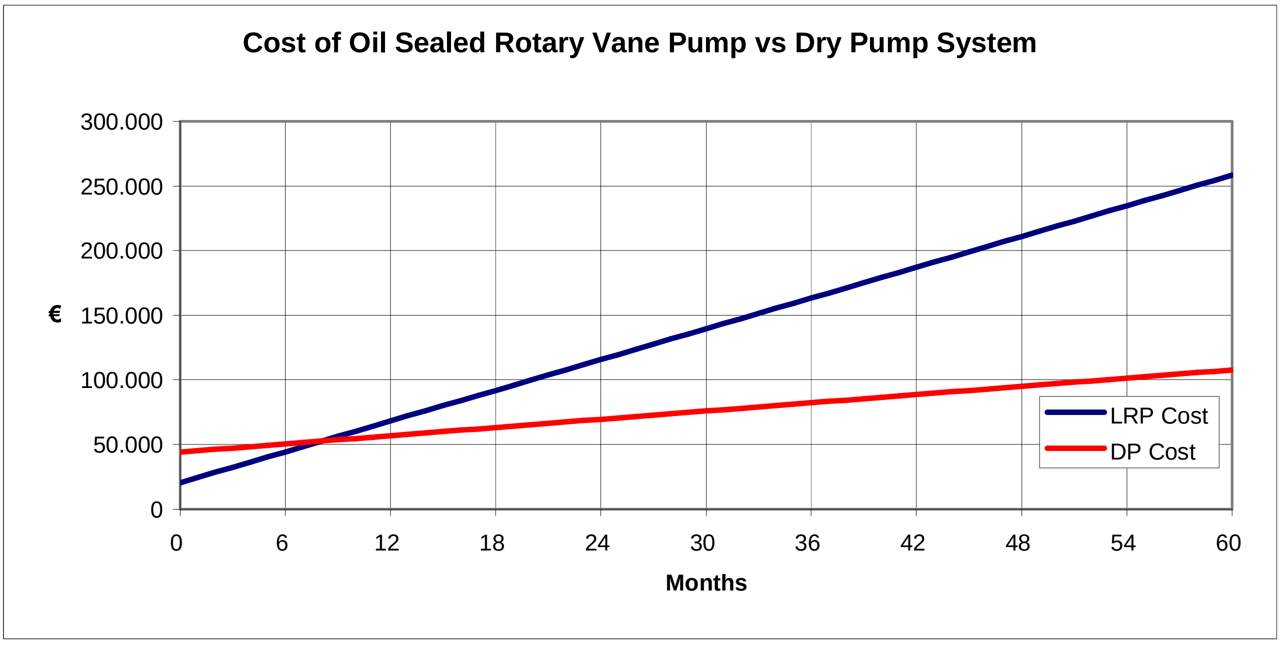
Usually two stage oil sealed rotary pumps for laboratory and pilot freeze dryer and roots/rotary, vapour booster/rotary and vapour diffusion/roots/rotary pump combinations for the production plants provide satisfactory backing and secondary drying pressures. Food freeze dryers operating at higher pressures generally employ large two stage rotary pumps or roots/single stage rotary pump combinations only.

For applications where severe pump combination by acid or solvent vapours was experienced in the past the roots/dry pump combinations should show marked improvements in plant performance, maintenance and reliability.

9. References / Photograph

10. Example of Cost of ownership

Cost of Ownership of a Oil sealed rotary vane pump versus a SIHI^{dry} system



11. Used formular and units

Pressure units : **Normal used 0℃ and sea level.**

P = 1013 mbar = 760 mmHg = 760 Torr = 1 atm.

Calculation of the Gas quantity Q

$$Q = P.V = [m/M].R.T$$

P = Pressure in mbar

V = Volume in (l)

m = Mass in (g)

M = Molmass in (g/Mol)

R = General gas constant (R = 83,14 mbar.l/Mol.K)

T = Absolute temperature in (K) [K = Kelvin]

Application Story

Calculation of the fore vacuum pump-down time.

$$t_a = V/S_{eff} \, . \, \ln(P_o/P_e)$$

t_a = pump-down time in (s)

V = volume in (l)

S_{eff} = pumping speed in (l/s)

P_o = initial pressure (mbar)

P_e = final pressure (mbar)

<u>Remark :</u>

With a given (constant) pumping speed, this equation offers rough values for the pump-down time in the range of (1000 – 1 mbar). Out-gassing effects, which maybe especially be effective in the range of < 1 mbar, have not been considered.

The pumping speed is given in l/s. Values given in m³/h can be calculated into l/s by dividing though 3,6.

NB. : Generally, the pumping speed of a vacuum pump depends on pressure. Conductance losses due to tubes or other conducting elements have to be considered.

Calculation of gas flow (mbar l/s)

$$\Delta Q/\Delta t = \Delta(p.V)/\, \Delta t = (\Delta m/\Delta t)/M.R.T$$

$\Delta Q/\Delta t = \Delta(p.V)/\, \Delta t$ = gas flow in mbar l/s

p = pressure (mbar)

V = volume in (l)

$\Delta m/\Delta t$ = mass flow in kg/h

M = molecular mass in (g/mol)

R = general gas constant R = 83,14 mbar.l/mol.K

T = absolute temperature (K)

Remark :

Derived from the general state equation for ideal gases, this formula allows the calculation of the gas flow as a function of mass flow and the characteristic molecular mass of each gas. The mass flow is given in kh/h. A temperature value of 293 K (room temperature) is present.