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FREEZE DESICCATION - A NEW METHOD OF FOOD PRESERVATION

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INTRODUCTION

Most dehydrated foods, when compared with other kinds of processed foods, offer considerable logistic advantages for military feeding. Dried products are lightweight, easy and quick to prepare, and are stable when held without refrigeration over an extended length of time. As a result of intensive research performed in recent years, these products have reached a high quality standard. The main breakthrough in acceptance occurred when the food was vacuum freeze dried, a process by which the water vapor sublimes from the frozen product under low total pressure directly into the gaseous phase. By this method migration of solutes, shrinking, case hardening, loss of flavor volatiles, and other undesirable effects of air drying were greatly reduced.

Since the inception of this process about 15 years ago much pioneering development work has been performed at the Quartermaster Food and Container Institute in Chicago and its successor, the Food Laboratory of the U. S. Army Natick (Mass.) Laboratories.

In conventional freeze drying the water subliming from the food is condensed on refrigerated surfaces at sub-zero temperature. In 1956 work at the Western Regional Research Laboratory and in collaboration with the Biological Warfare Laboratories at Fort Detrick, Maryland, showed that frozen pellets of bacterial cultures could be vacuum freeze dried using silica gel (1). Recently we have investigated the use of solid desiccants similarly for drying foods. We termed this process "Freeze Desiccation". Preliminary studies indicated that the retention of quality of the food processed by freeze desiccation was high and that the equipment required was less intricate and less expensive than that needed for the conventional method. The new process offered certain additional advantages which are discussed later in this paper. Although our experiments were performed on a laboratory scale and in batches, the results suggest that industrial application in the form of a continuous freeze-desiccation plant is a distinct possibility.

PROCEDURES

The differences between laboratory-scale freeze drying and freeze desiccation are indicated in Fig. 1. The food is prepared in the same way for both processes. In conventional freeze drying a condenser to trap the water vapor and heating plates are necessary to provide the heat for sublimation. In freeze desiccation, as performed in our laboratories, the frozen food was wrapped with a protective fabric and surrounded by a solid desiccant inside a drying chamber. Silica gel, which is granular, amorphous silicon dioxide, was found to be suitable for this purpose. Water vapor penetrates its submicroscopic pores and is held by surface absorption and capillary condensation. The action of silica gel on water vapor is reversible by heat. The desiccant will totally desorb at 350°F, and is then capable of adsorbing to its full capacity again. In repeated cycles there is some loss of sorption capacity of the gel due to occlusion of small amounts of volatile organic materials from the foods and their alteration by the heat applied to dry the gel. For some purposes other desiccants are sometimes useful. For example, when low moisture levels in the dried food were desired, a mixture of silica gel with molecular sieves was found to be better than silica gel alone. The molecular sieves are crystalline complex silicates containing aluminum and one or more other metallic elements.

The desiccation chamber, packed with the frozen food samples as described above, was evacuated to about 0.3 mm Hg, at which point the valve to the vacuum pump was closed. The chamber was then placed in a refrigerated room (5°C). The most suitable wall temperature of the chamber was found to be between 0°C and 10°C. The moisture of the food is sublimed from the solid into the vapor phase, and is absorbed by the desiccant at a rate sufficient to keep the frozen food from melting. During the early stages of dehydration, the heat evolved due to the adsorption of water by the desiccant is utilized as the primary energy source for sublimation. As drying progresses, the heat conducted to the product from the chamber walls becomes increasingly important. Thus the size and shape of the chamber are also important. When the ice is completely sublimed, the walls of the chamber may be brought up to room temperature to hasten desorption of bound water and attainment of the ultimate moisture level.

STRASSER, KAPSALIS* and GIFFEE

STUDIES OF THE FREEZE-DESICCATION PROCESS UNDER LABORATORY CONDITIONS

1. Relationships Between The Weight of The Food And The Weight of The Desiccant.

Preliminary studies to determine the relationship between the initial weight of the product and the weight of the desiccant needed for drying were conducted with high protein foods (precooked beef and raw codfish). Differences in the initial moisture content of the frozen samples were considered to be negligible. Experiments with beef promptly revealed that the ratio of food to desiccant regulates the degree to which the product is dried (Fig 2). The sharp increase in the amount of desiccant necessary to obtain a low moisture content in the meat reveals the limitations of a one-step batch-type freeze-desiccation process. It should be noted that a smaller amount of molecular sieve than of silica gel is necessary to obtain the same final moisture content. The difference is caused by the strong affinity between molecular sieves and water in the low moisture region.

2. Influence of The Location of The Desiccant.

In order to investigate the influence of the heat transport from the desiccant to the product, two experiments were performed. In one experiment codfish samples (discs 16mm diameter, 6mm thickness) were packed in silica gel and the moisture content was determined after different drying times. The weight ratio of the frozen fish to silica gel was 1:5. The chamber wall temperature was kept at +10°C. In the other experiment, the sample and the desiccant were located in connecting chambers. The weight ratio and the chamber wall temperature were the same as in the first experiment. The drying times for samples in contact with the desiccant were about 50% less than those for those not in contact because of the better conditions for heat and mass transfer (Fig. 3). The experiments also showed that the final moisture content was independent of the location of the food relative to that of the desiccant. The final moisture content depends upon the weight ratio of frozen sample to dry desiccant, final chamber temperature, and the sorption characteristics of both the food and the desiccant.

3. Correlation Between The Desorption Isotherm of The Food And The Adsorption Isotherm of The Desiccant.

At the end of the freeze-desiccation process, an equilibrium water vapor pressure has been established in the chamber. This equilibrium can be best understood if the adsorption isotherm of the desiccant and the desorption isotherm of the food at the final drying temperature are known. For isotherm measurements the instrumentation shown in Fig. 4 was used. Precise water vapor pressures were generated in the system by means of a thermostatically controlled water bath. This method of measuring isotherms is superior to the usual technique of using saturated salt solutions since closely spaced points over the entire relative humidity (R.H.) range can be obtained and controlled. A front view of the apparatus is shown in Fig. 5.

The moisture sorption isotherms of raw codfish and silica gel are plotted in Fig. 6. On the basis of these isotherms, one can calculate the amount of desiccant necessary to reach a certain desired moisture content in the food. The following equation was derived:

$$R = \frac{m_d \times (1 + \frac{m_i}{100})}{m_i - m_e}$$

R = ratio of weight of wet food to weight of dry desiccant

m_d = final equilibrium moisture content of desiccant (% dry basis)

m_i = initial moisture content of food (% dry basis)

m_e = desired final moisture content of food at end of drying process (% dry basis)

The sorption characteristics of molecular sieves are compared with the sorption behavior of silica gel in Fig. 7. Below about 30% relative humidity, molecular sieves of the 4A type have a considerably higher affinity for water vapor. Above 30% R.H., silica gel is the more hygroscopic. Therefore, substitution of a molecular sieve for the silica gel below about 30% R.H. would be suitable when low final moisture contents are desired, especially in a continuous freeze-desiccation apparatus. Such a plant would be almost mandatory for large scale production.

It is conceivable that eventually a continuous freeze-desiccation process may become competitive with the freeze-drying process now in use. It might even be considerably less expensive, since high-cost refrigeration equipment would be displaced by low-cost desiccants, which can be used repeatedly following simple heat reactivation. The solution of all problems concerning the feasibility of a continuous system must await the information that can only be supplied by pilot-plant studies.

ADVANTAGES

The freeze-desiccation process has several advantages. No sophisticated freeze-drying equipment with refrigerated condenser and heating plates is necessary. A simple laboratory desiccator or any other sealable container may be used for the drying

4-

chamber. Vacuum pumping is necessary only at the beginning of the process. Therefore, a single vacuum source can be used for a whole series of chambers which are simply sealed off after evacuation and transferred to a cooler.

The advantages of predetermining a uniform terminal moisture content in the freeze-desiccated foods are manifold. In fact, this feature is as yet one of the unachieved goals for the conventional freeze-drying method. It has been known for several years that some foods undergo oxidative deterioration more rapidly when excessively dry than when they contain the so-called monolayer amounts of water (4, 3). Still higher levels of moisture promote discoloration and off-flavor development through amino-carbonyl and other types of reactions. Retention of desirable texture also requires optimal amounts of water in the stored product, and it was recently demonstrated (2) that this optimal moisture range can be accurately defined by suitable instrumentation.

There is a need by the military for a number of compressed food bars for both tactical and logistic purposes. Freeze-dried foods are particularly suitable for compression because of the voids remaining after removal of water. A certain amount of residual moisture in the dried product contributes a desirable degree of plasticity for compression purposes while an overly dry product tends to crumble. Without the advantage of freeze desiccation, products are often overdried and an extra step is introduced wherein the products are moistened before compression. This constitutes an added cost.

Freeze desiccation is a very gentle process; the mild heat of adsorption released by the desiccant is supplied to the food by conduction without perceivable damage to the product. The high survival rates achieved with bacterial cells by Graham et al. (1), is evidence that even subtle damage is unlikely.

SUMMARY

Freeze desiccation of foods is based on the use of a solid desiccant for the adsorption of water vapor which sublimes from the frozen food at a reduced pressure. Desorption isotherms of the food and adsorption isotherms of the desiccant were used for the calculation of the quantities of desiccant necessary to obtain different pre-determined residual moisture contents of the food. An equation and a curve were developed for this purpose. The new method compares favorably with conventional freeze dehydration. It offers the specific advantages of accurate control of the end point residual moisture in the food. For moisture contents above about 5%, the use of silica gel as a desiccant was found to be very suitable. For moisture contents below this limit, substitution of molecular sieves for the silica gel in a two-stage desiccation process was necessary in order to desorb the more firmly bound water.

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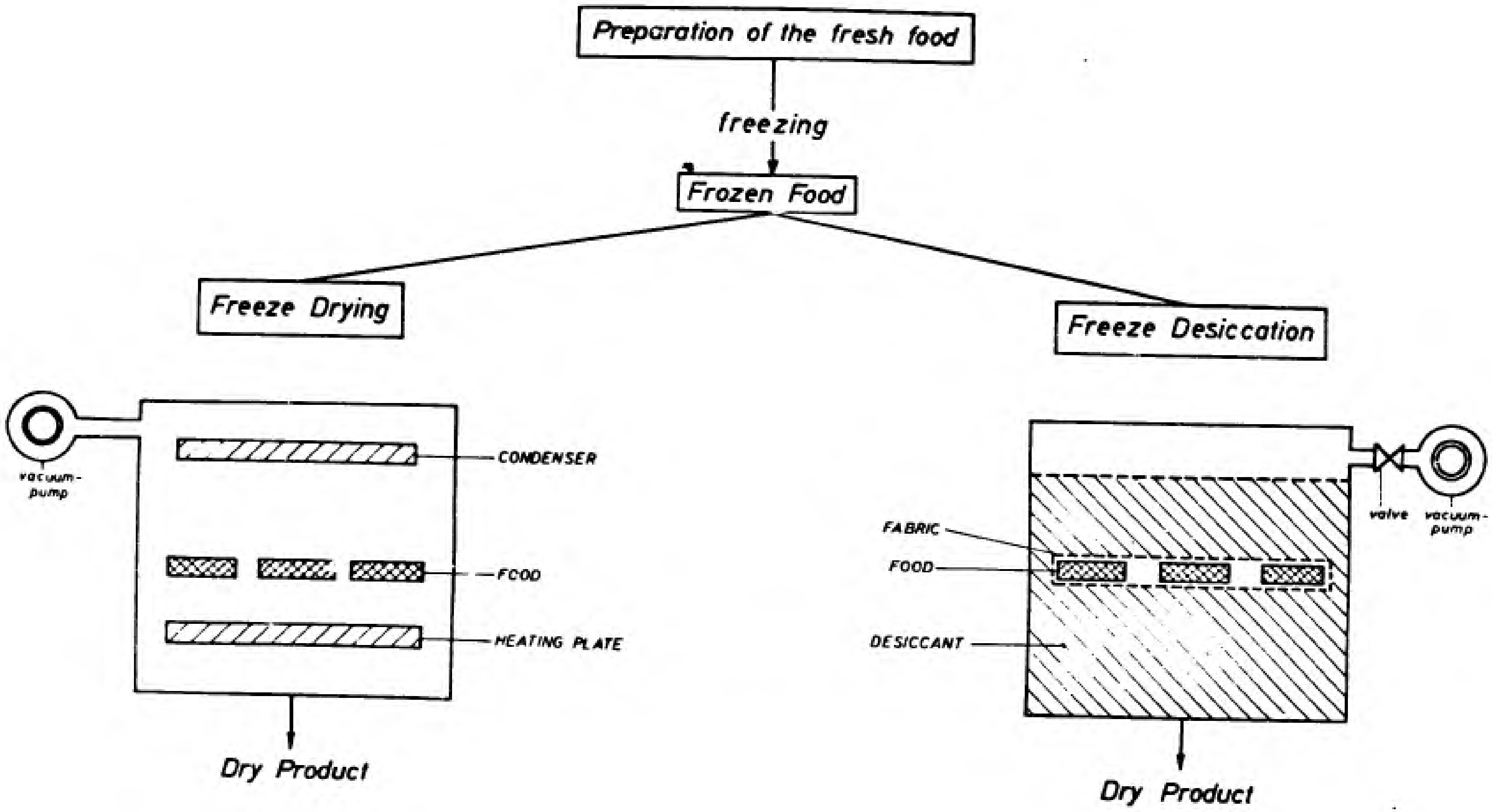

Fig. 1. Comparison of commonly used freeze drying with freeze desiccation

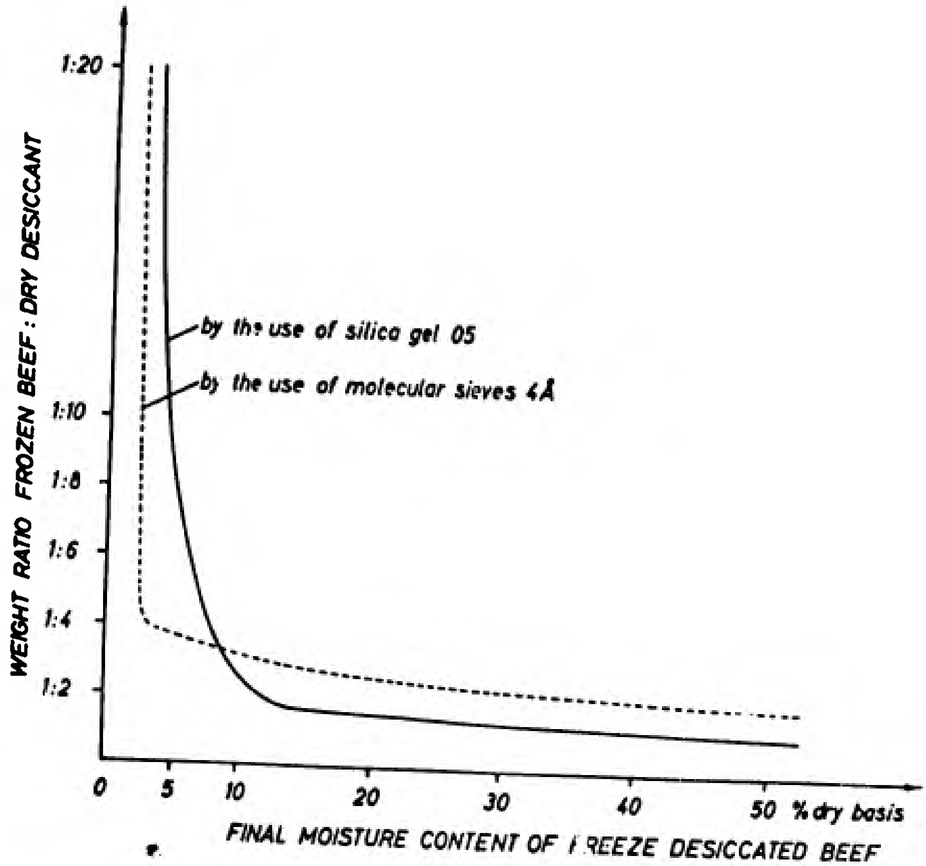

Fig. 2. Relationship between food to desiccant weight ratio and final moisture content in beef.

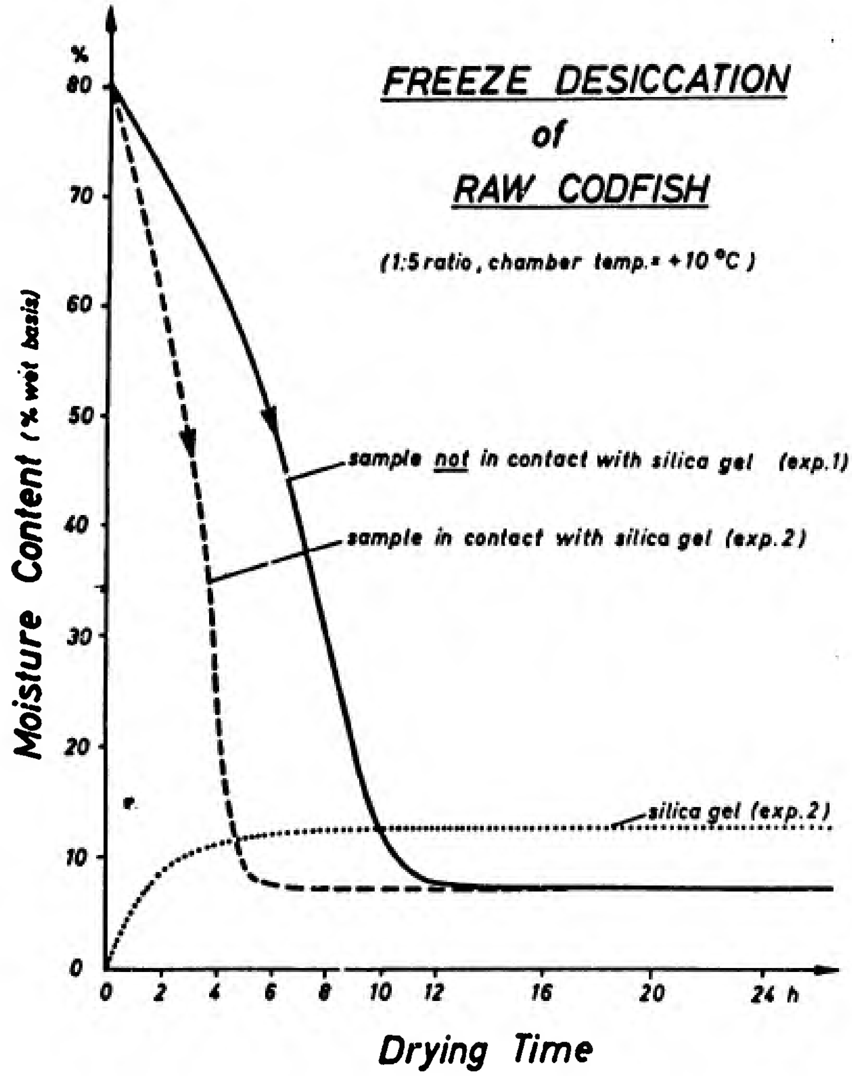

Fig. 3. Comparison of different methods of freeze desiccation. The dotted line indicates the moisture uptake of silica gel during experiment 2.

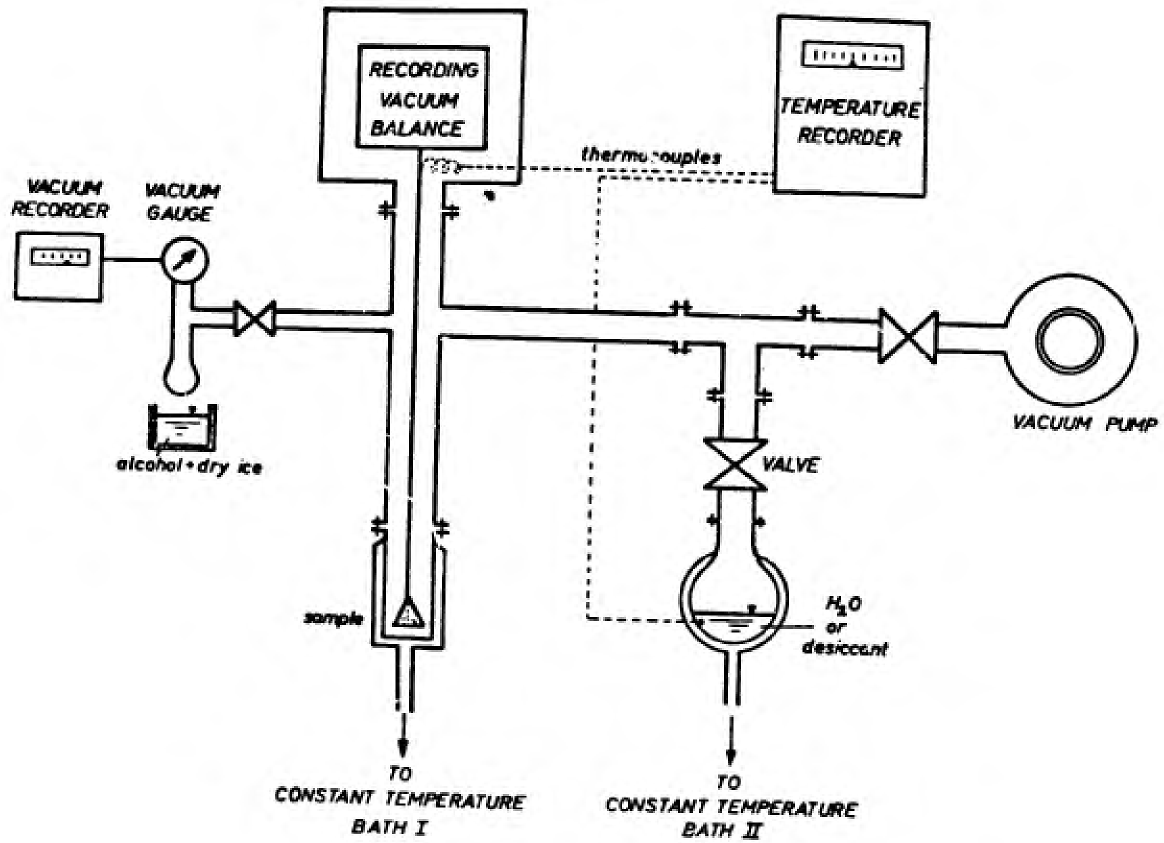

Fig. 4. Apparatus for the measurement of sorption isotherms or freeze-dehydration rates. The bath with alcohol and dry ice is used for the determination of the partial pressure of "non condensible gases" in the system.

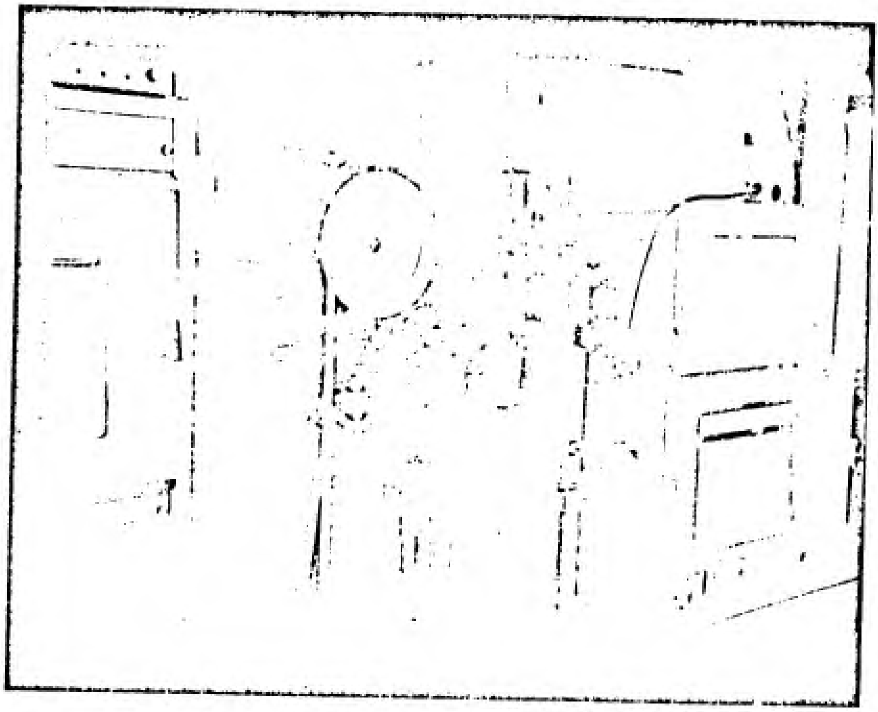

Fig. 5. Apparatus for determining sorption isobars.

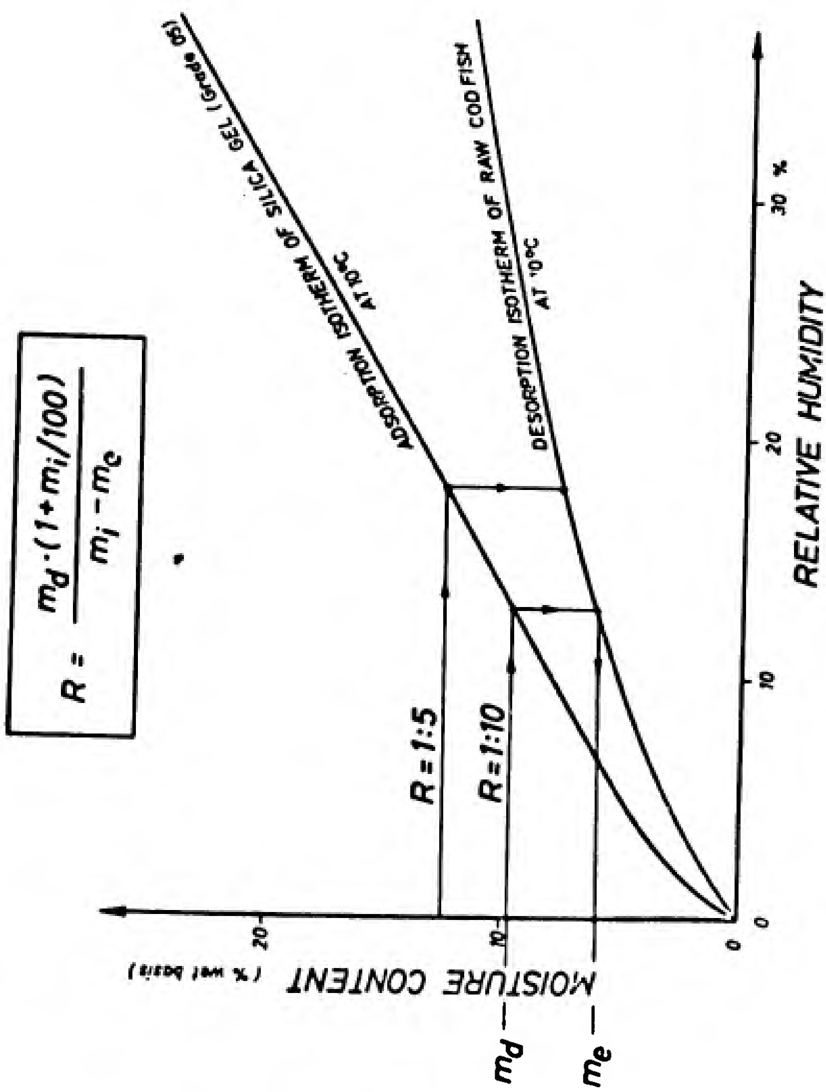

Fig. 6. Moisture sorption isotherms of raw codfish and silica gel.

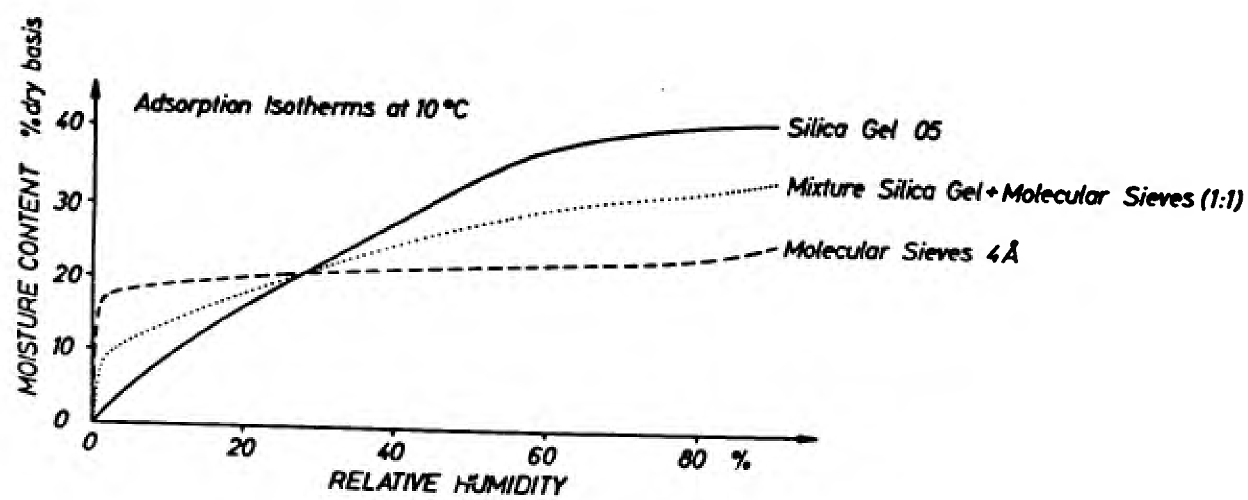

Fig. 7. Moisture sorption isotherms of silica gel and molecular sieves