DETERMINATION OF MOISTURE IN FOOD

3.1 Introduction

Moisture determination is one of the most important and most widely used measurements in the processing and testing of foods. Since the amount of dry matter in a food is inversely related to the amount of moisture it contains. Therefore it is of direct economic importance to the processor and the consumer. Stability and quality of foods is also affected by moisture content, hence it also has a role in the safety of foods. Grains that contain too much water is subject to rapid deterioration from mold growth, heating, insect damage and sprouting. The rate of browning of dehydrated vegetables and fruits, or oxygen absorption by egg powders increases with an increase in moisture content. High moisture content of ghee leads to hydrolytic rancidity.

Moisture determination is important in many industrial problems, i.e. in the evaluation of materials' balance or of processing losses. Moisture content (and sometimes its distribution) must be known for optimum processing of foods. Milling of cereals, mixing of dough to optimum consistency, and for producing bread with the best grain, texture and freshness retention. Moisture content must be known in determining the nutritive value of food, in expressing results of analytical determinations on a uniform basis, and in meeting compositional standards or laws.

3.2 Some Basic Considerations

Water may occur in foods in at least three forms.

1. A certain amount may be present as free water in the intergranular spaces and within the pores of the material.
2. Such water retains its usual physical properties and serves as dispersing agent for the colloidal substances and as a solvent for the crystalline compounds.
3. Part of the water is absorbed on the surface of the macromolecular colloids (starches, pectins, cellulose and protein). The water is closely associated with the absorbing macromolecules by forces of absorption (vander waals or hydrogen bond).
4. Finally some of the water is in a bound form in combination with various substances, i.e. water of hydration.

The rapid and accurate determination of water in foods possesses many problems. Many workers have stressed the complexity of analytical procedures for the determination of water in foods. In practice, the guiding principle has been to prefer the method that gives the highest moisture values, with negligible decomposition volatilization of compounds.

3.3 Methods for Determination of Moisture in Foods

Methods for determination of moisture in food can be divided into four different classes.

1. Drying methods
2. Distillation procedure
3. Chemical assays
4. Physical procedures

3.3.1 Drying methods

The procedures for determination of the moisture content specified in food standards generally involve thermal drying methods. The material is heated under carefully specified temperature and the loss of weight is taken as a measure of the moisture content of the sample. The value obtained for moisture depends on type of oven, temperature and length of drying. Therefore, the methods provide same time approximate rather than accurate moisture values. The rate at which moisture can be removed from the surface of a solid phase is a function water vapour pressure and of the drying temperature. Practical consideration dictates, however, selecting temperatures at which the decomposition of organic compounds is minimised, and yet the time required for quantitative drying at the selected temperature not unduly prolonged.

Advantages

Drying methods, however, are simple, relatively rapid, and permit the simultaneously analyzes of large number of samples.

Disadvantages

1. Heating of a moist organic substances causes, in addition, volatilization of material and formation of gaseous product by irreversible thermal decomposition of organic component.
2. Further weight changes resulting from oxidation phenomenon (i.e. oxidation of oils) occur.
3. Improperly maintained dessicator and dessicants can cause erratic results from pick

up of moisture during cooling.
4. Formation of a crust that is impervious to evaporation of moisture from the centre of a dried sample.

Factors affecting the precision of moisture measurements by drying methods

The accuracy of moisture determinations is affected by

1. Drying temperature
2. Relative humidity of the drying chamber
3. Air movement in the drying chamber
4. Vacuum in the chamber
5. Depth and particle size of the samples
6. Drying oven construction
7. Number and position of samples in oven
8. Diameter and type of container (material of container)

The rate of evaporation is higher in aluminium than in glass or porcelain dishes, high in vacuum than in simple ovens, and high in shallow than in deep dishes. Solid materials must be pulverized under conditions that minimize compositional changes. In drying liquids, it is essential to spread the material over a large surface. The liquid is preferably evaporated first on a water bath, and then drying is completed in an oven.

To reduce crust formation the sample is moistened with water and thoroughly mixed with sand or asbestos. To increase the area of drying semisolid material is spread with the help of glass rod. Sample weight is generally limited to 3 to 5 g. Standard aluminium dishes are recommended for cereals (55 mm diameter and 15 mm height). The drying temperature used in moisture determination ranges from 70 to 155^{o}C (depending on the tested material). The average time for drying is from below 1 hr to 6 hr or more. Certain sugars (especially fructose) are sensitive to decomposition at elevated temperatures like in honey and fruit syrups. Fructose solutions decompose at temperature above 70^{o}C and glucose is relatively stable at 98^{o}C. The drying of fructose, glucose or sucrose solutions is faster and the tendency to decompose is less if the pH is below 7.

Foods can be dried for moisture determination either for a selected period of time or until two successive weighing show a negligible loss in weight (generally 1 mg for a 5 g sample, at 20 min interval). Drying time is inversely related to drying temperatures. In foods susceptible to decomposition, drying temperatures can be reduced by using vacuum ovens.

3.3.2 Distillation methods

There are two types of distillation procedures.

- In one type, water is distilled from an immiscible liquid of high boiling point. The sample suspended in a mineral oil having a flash point much above the boiling point of water is heated to a predetermined temperature in a suitable apparatus. The water that distills off is condensed and is collected in a suitable measuring cylinder.
- In the second type, the mixture of water and an immiscible solvent (i.e. xylene, toluene, tetrachloroethane) distills off and is collected in a suitable measuring apparatus in which water separates and its volume can be measured.

Liquids with a high specific gravity (tetrachloroethylene, carbon tetrachloride) eliminate fire hazard and reduce the danger of overheating or charring, as sample floats on top of the liquid.

Distillation methods cause less decomposition in some foods than drying at elevated temperatures. However, chemical reactions produced by heat are reduced but not eliminated.

Disadvantages

Many difficulties may be encountered in the determination of moisture by the distillation method. These include

1. Relatively low precision of the receiving measuring device.
2. Difficulties in reading the meniscus.
3. Adherence of moisture droplets to the glass.
4. Overboiling (especially with xylol).
5. Solubility of water in the distillation liquid.
6. Incomplete evaporation of water and underestimation of moisture contents.
7. Distillation of water soluble components.
8. Foods in powder form (cereals, flours, starches) tend to bump during the distillation through overheating on the bottom of the flask.

The main objection to distillation procedures is that they are not adaptable to routine testing. Some of the disadvantages can be overcome by the following interventions:

1. Adherence of water to the walls of the condenser tubes or sides of the receiving tubes can be generally remedied by using thoroughly cleaned glassware.
2. Use of small amount of wetting agent will also improve meniscus reading.
3. Incomplete recovery of water due to the formation of an emulsion can sometimes be remedied by adding small amounts of amyl alcohol or isobutyl alcohol.
4. To improve the moisture distillation, wide mouthed boiling flasks can be used.
5. Dispersing the tested material on diatomaceous earth or on filter – cell is useful with many viscous foods rich in sugar or protein.
6. Bumping of powder foods can be overcome through the introduction of a small amount of dry short fiber asbestos.
7. Adverse effects of heat can be reduced still further by selecting organic solvents with a boiling point below that of water, such as benzene. Such a choice, however, lengthens the distillation time.
8. For accurate results, standard apparatus and careful attention to specified procedures are essential.

3.3.3 Chemical methods

Karl Fischer Reagent Titration

The time needed for dehydrated foods stimulated the search for more rapid and accurate methods for determining moisture. The Karl Fischer reagent has proved to be quite adaptable for this purpose.

Advantages

- It is a method of choice for determination of water in many low moisture foods such as dried fruits and vegetables, candies, chocolate, roasted coffee, oils and fats.
- Superiority of the method was demonstrated in determining moisture in sugar rich foods (honey) or foods rich both in reducing sugars and proteins.
- The procedure has been applied also to foods with intermediate moisture foods (bakery doughs, baked products, fat rich cake mixes) and to foods with high levels of volatile oils.
- The Karl Fischer reagent has proved to be quite adaptable for moisture determination by chemical method.
- The Karl Fischer method for moisture determination is based on the reaction which involves the reduction of iodine by sulphur dioxide in the presence of water.

$$2\ H_2O + SO_2 + I_2 \text{ ---------- } H_2SO_4 + 2\ HI$$

As shown by the above reaction for each mole of water one mole of iodine is required.

- Methanol is used to dissolve iodine and pyridine is used to dissolve sulphur dioxide.
- Numerous variations have been proposed for the preparation of the Fischer reagent.
- The sample in which water is to be determined is dispersed in an appropriate solvent (i.e. methanol, mixture of methanol-sulphur dioxide-pyridine etc.).
- The solution is then titrated with a solution of iodine in methanol.
- The excess of iodine that cannot react with water is in free form.
- Adding to the system a few drops of methylene blue gives a green end point.
- Interfering substances are ascorbic acid (oxidation), aldehydes, ketones (release water), mercaptans, diacylperoxide, thioacids and hydrazines – fading end point.
- The determination of moisture is carried out in a non aqueous system.
- Fluids are delivered but with an automatic pipet or syringe.
- Viscous fluids or pastes are generally homogenized with a solvent.
- Solids are either homogenized with solvent or titrated as suspensions.
- Granular products must be pulverized.

Disadvantages

- The main difficulty in using the Karl Fischer method arises from the lack of complete water extraction.
- Formaldehyde is found to be a more rapid and versatile extractant of water from foods than methanol.
- Modification of the extraction procedure is exemplified by a method for the water determination in dairy products, where in xylene or carbon tetrachloride is employed in mixed solvent systems with alcohol.

3.3.4 Physical methods

- Infrared determination – based on measuring the absorption at wavelengths characteristic of the molecular vibration in water.
- The most useful wavelengths are 3.0 & 6.1 μm.
- Gas chromatographic method: based on extracting the moisture with an organic solvent and determining water in the extract by gas chromatography
- Nuclear magnetic resonance
- Electric method: Densitometric method
- Refractometric method
- Polarimetric method