# 農產品平衡相對濕度之測定與應用

## Ⅱ. 平衡相對濕度之測定方法和影響因素

# The Measurement and Application of the Equilibrium Relative Humidity of Agricultural Products

## II. Determination methods and effect factors


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**摘 要**

由於 ERH/EMC 的物性在農產品加工工程應用上十分重要，許多種測定此性質之方法已爲研究者加以研究。依其測定的對象，可分爲直接式和間接式兩種。本研究中探討其方法之特點和限制，並介紹電子相對濕度計應用之限制。由於 ERH/EMC 的性質受到許多因素所影響，本文亦就其主要之影響因子分別加以探討。

**ABSTRACT**

Because of the importance of ERH/EMC properties on the processing work, many methods have been employed to determine these properties. Major methods used most frequently to collect sorption data are direct and indirect methods. Their advantages and limitations are discussed in this research. The performance test of electric RH sensors is described. There are many factors affecting the ERH/EMC properties. The domainant factors are discussed in this study.


### I. Determination of the sorption isotherm

Many methods have been employed to measure EMC or ERH properties. Detailed reviews of this subject were presented by Gal (1981), Leung (1986), and Spiess et al. (1987).

Major methods used most frequently to collect sorption data are indirect (gravimetric) and direct (hygrometric/manometric) methods.

### a. Indirect method

Samples are placed in an environment of constant temperature and relative humidity. After a long period, the samples reach equilibrium with the environment. The moisture content of the samples is then measured.

The most popular humidity generators are various saturated salt solutions and sulfuric acid solutions at different concentrations.

A salt slurry is usually prepared by adding an excess amount of salt to a saturated solution until a slurry is formed. The excess slurry serves as the moisture sink to allow exchange of water with the sample without affecting the ERH value. Several kinds of salts are used to produce a range of RH values.

According to the state of air of the head space, this method can be divided into static, dynamic, and evacuated method. The air is mechanically moved in the dynamic method (Rosin and Easthouse, 1970). For the evacuated method, the desiccator or humidity chamber is evacuated to reduce the time required to reach the equilibrium state by reducing the resistance to transfer of water molecules between the sample and the salt slurry. Recently, a standard gravimetric method has been proposed (Wolf et al. 1985).

Although a chemical treatment has been utilized to retard mold development (Dunstan et al., 1972), it does not eliminate mold development at high relative humidities.

### b. Direct method

The manometric method provides the most direct measurement of the vapor pressure of the sample. With this technique, the chamber containing the sample is evacuated and allowed to equilibrate at a specific temperature. The pressure of water vapor in equilibrium with the sample is measured with an oil manometer or a differential pressure transducer.

With the hygrometric method, a sample of known moisture is allowed to be controlled in a limited volume environment. The air conditions (temperature and relative humidity or dew point temperature) are measured. This method can shorten the time required for obtaining the ERH data, but the technique has been limited in the past by poor resolution and stability of the measurement devices.

Henderson (1970) used a closed-loop dew point system to determine ERH data. In his method, a relatively small quantity of air was circulated in a closed system until it came into moisture and temperature equilibrium with a relatively large sample of hygroscopic material. The sample temperature and the dew point temperature of the circulating air established the ERH value. Due to the relatively small volume of air being circulated, there was no significant change in sample moisture content.

Pixton and Warburton (1971a) adopted the dew-point method to measure the ERH values of five cereal grains at different temperatures. A thermocouple in metal-to-metal contact with the silver mirror surface wes ultilized to obtain the dew-point temperature.

Vemuganti and Pfost (1980) used this technique to measure ERH data for 20 grains. The Aminco hygrosensor was selected to measure the RH value of the circulating air.

Recently, the same method was adopted by several experimenters. Flood and White (1984) measured the dew point temperature by dew point hygrometer to determine desorption data for popcorn. To prevent the heat, which came from the air pump, from affecting the equilibrium state, a four-meter coil of copper tubing was used to dissipate thermal energy before air was returned to the sample holder. The same technique was employed by White et al. (1985) to find Three sorption isotherms of corn cobs of different varieties and by Flood et al. (1986) to obtain the isotherm relation of pine shaving poultry litter. Sokhansanj et al. (1985) chose this method to collect ERH data for rapeaseed (Canola) using a LiCl dew cell to measure relative humidity.

Jayas et al. (1987) employed a similar technique to measure the ERH properties of Canola meal. The RH value was measured with a LiCl humidity sensor. The sensors were calibrated with two kinds of saturated salt solutions.

In spite of the convenience of the direct method, the accuracy of the measured value depends on the performance of the RH measurement. The performance characteristics, however, were not mentioned by above experimenters and no calibration curves were established and reported.

### c. Performance tests of electric hygrometers

As a direct method of collecting ERH data, electric hygrometers have their advantages in accuracy, speed, and convenience. However, some drawbacks exist. The hygrometers are expensive, require precise calibration, experience aging problems, may be affected by contamination, and may be inaccurate at certain levels of relative humidity.

Labuza et al. (1976) conducted the first major study of water activity measurement. Five electric RH sensors were tested and compared. Ten saturated salt solutions were selected to provide RH values for calibration. The average of the difference between the output value of sensors and the Aw standard were used for comparsion.

Troller (1977) described a calibration procedure for the Sina-scope hygrometer. Four saturated salt solutions, covering a range from 75.5-96.7% RH, were used to determine the accuracy and precision. The percentage of the difference between measured mean value and the standard value was adopted as the criterion for comparison. The concept of confidence interval was used to compare replications. They found that the coefficient of variation of this sensor decreased with measurement of higher Aw (ERH/100) standard solutions. The variation did not exceed 1% for measuring these saturated salt solutions and for two kinds of foods.

Favetto et al. (1983) studied the suitability, precision, and accuracy of Vaisala Humicap electric hygrometers. The Aw measurements were in the range of 75.2-97.4% RH which was provided by six saturated salt solutions. A calibration curve was first fit by a straight line. A nonlinear calibration equation.

$Y = A^{*}Exp(B^{x})$, was also proposed to obtain better accuracy, where x was the reading value from the RH sensors and Y was the value of "true" water activity. After working with three food samples, they concluded that the Vaisala electric hygrometer produced accurate and precise Aw measurements which could be used in most food research.

Stamp et al. (1984) studied two electric RH sensors and a dew point sensing device for ERH measurement. The RH value from saturated salt solutions obtained as the standard to establish the calibration curve. Linear regression equations were adopted. It was found that above Aw of 0.85 and below 0.32, the results given by these instruments were not linear. Each sensor had to be calibrated individually. These instruments did not work satisfactorily for measuring the Aw of foods.

Kitic et al. (1986) tested the Novasina Thermoconstanter electric hygrometer for Aw measurements in the approximate range of microbial growth (0.58-0.97 Aw). Seven saturated salt solutions were used as standards for measurement of Aw. They found a high linear correlation between the reading value and the true value. Three characterstics of this hygrometer were reported: (1) high level of precision, (2) stability of the calibration curve, (3) adequate equilibration time.

From the above discussion, it was found that proper calibration is the key to obtaining accurate Aw measurements with electric RH sensors. Saturated salt solutions are recommended to produce standard relative humidities. Unfortunately, due to the lack of the real standards, the value for the "standards" used to calibrate the instruments is still established individually by each investigator (Stoleff, 1978). The various "standard" Aw values for salt solutions are listed in Table 1. The following account has been presented by Labuza et al. (1976).

1. The standard value was found by averaging values from the literature. Different values are reported for a particular salt and temperature.
2. The values in the literature have been determined by different methods with different degrees of accuracy.
3. The literature is cross referenced.
4. The Aw's of the slurries are temperature dependent.
5. Investigators handle the literature values in different ways.

The lengthy time required is another disadvantage of using salt solutions in calibration. It usually takes one or two hours for the salt solutions to reach the equlibrium state in a calibration bottle. Also since each salt solution exhibits only one Aw value, several salt solutions are needed for a range of relative humidi-

Table 1. Aw values of saturated salt solutions from the literature

source: Labuza et al. 1976)

| Salt | (1) 22 C | (2) 20 C | (3) 20 C | (4) 25 C | (5) 20 C | (6) 20 C |
|---|---|---|---|---|---|---|
| KC2H3O2 | 0.230 | 0.23 | 0.200 | 0.225 | --- | 0.20 |
| MgC12 | 0.330 | 0.33 | 0.340 | 0.330 | 0.336 | 0.33 |
| Zn(No3)2 | --- | 0.38 | 0.420 | --- | --- | 0.42 |
| Ca(No3)2 | 0.522 | 0.56 | 0.560 | --- | --- | --- |
| Na2Cr2O7 | 0.582 | --- | 0.520 | 0.536 | 0.552 | 0.52 |
| NaNo3 | 0.648 | --- | 0.660 | --- | --- | 0.66 |
| NaC1 | 0.756 | 0.75 | 0.765 | 0.753 | 0.755 | 0.76 |
| (NH4)2SO4 | 0.802 | 0.79 | 0.817 | --- | 0.806 | 0.81 |
| KNO3 | --- | 0.94 | 0.942 | 0.925 | 0.932 | 0.94 |

(1) Wink and Sears (1950)
(2) Rockland (1960) (5) W
(3) O'Brien (1948)
(4) Stokes and Robinson (1949)
(5) Wexler and Hasegawa (1954)
(6) Inter. Critical Lab. (1926)

ties.

## II. Factors affecting sorption phenomena

### a. Comparison of EMC data for corn kernels

Four sets of EMC data (Brooker et al. 1974) for corn kernels in a similar environment are shown in Fig. 1. For two sets at 30 °C, there is a 22% difference in m.c. at 40% RH and a 25% difference at 90% RH.

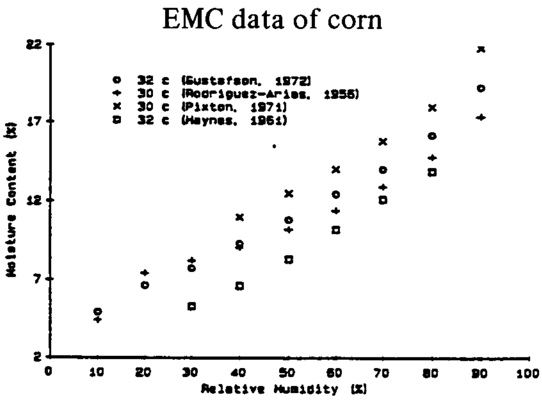


Figure 1. EMC data for corn kernels from different sources

Brooker et al. (1974) concluded that the variation in EMC values was caused by differences in: (1) grain variety, (2) grain maturity, (3) grain history, (4) the relative humidity measuring techniques, and (5) the EMC determination method.

Twenty-seven published reports on corn isotherms were reviewed by Neuber (1981). The important factors related to the EMC value are summaried as followings:

1. Composition of materials – ash, oil (fat) content, fiber, protein, and starch.
2. Characteristics of the materials: variety, harvest year, and particle size.
3. Pretreatment of sample: drying temperature, time, RH of air, drying method, initial m.c., rewetting method.
4. Determination method of m.c. and RH.
5. Method of equilibration.
6. Control of RH and temperature.

Neuber then suggested that a standard method should be established as soon as possible for the determination of sorption isotherms. In order to arrive at a comparable result, it is also necessary to identify the factors which influence the equilibrium moisture curves.

Pfost et al. (1976) compared major factors affecting EMC data for corn kernels from several sources; they encountered great difficulty when trying to summarize data from various sources using different methods. They then suggested either pooling the results of several investigators, or having one investigator test a wide range of varieties.

### b. The effect of the composition of the material

Sorption isotherms of four compositions at 25 °C are shown in Fig. 2.

The sorption isotherm of vacuole only involves the effect of sugars and minerals (Crapiste and Rotsein, 1982). The mois-

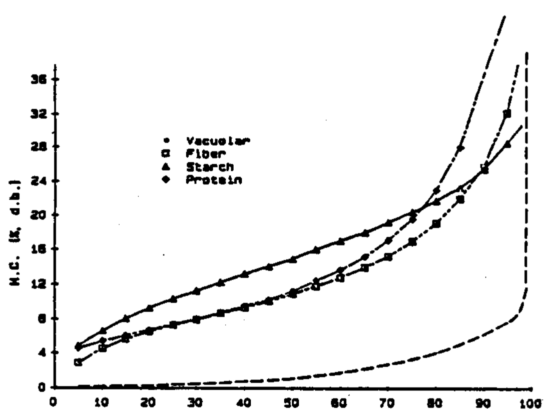


Figure 2. Sorption isotherms of four compositions.

ture content of starch could be higher than that of protein and fiber at the same ERH value in the lower and intermediate RH ranges. In the high RH range, protein absorbs more moisture. The vacuole moisture content becomes dominant as the RH approaches saturation.

Kumar and Balasubrahmanyam (1986) carried out a study of EMC values at 27 °C on 16 foodstuffs that were cataloged by their major constituents. Some sorption isotherms are hown in Fig. 3. The foods with high starch exhibited higher water binding capacity and a wider linear isotherm range. Foods with high oil content have sorption isotherms which tend to flatten towards the ordinate. Above the intermediate RH range, there is a sharp rise in the sorption curve. Protein-rich foods have a sorption isotherm between those of starchy and fatty foods.

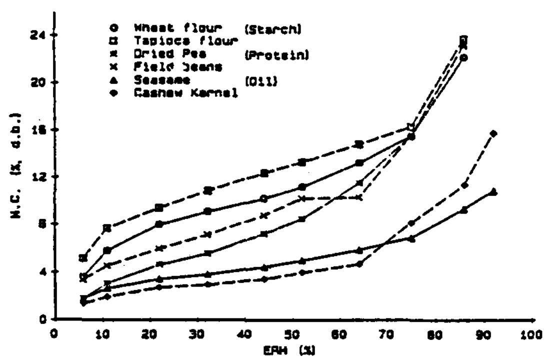


Figure 3. Sorption isotherms of different dominant constitution

The ERH properties of five oilseeds with different oil content were reported by Pixton et al. (1971b). The relationship between EMC and ERH at 25 °C are shown in Fig. 4. The oilseeds with higher oil content adsorbed less water at the same RH value.

From the study of moisture sorption isotherms of air dried minced beef, Iglsias

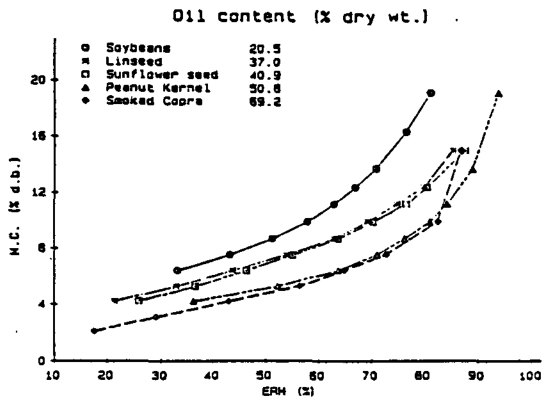


Figure 4. Sorption isotherms of different oil content

and Chirife (1977) found that adsorption by fat was negligible. Comparing samples with different fat quantities, they indicated that the EMC values of all samples were coincident when the values were expressed on a fat-free basis, and suggested therefore that the fat-free basic of moisture content might be adequate to compare the EMC value of minced beef with widely different fat contents.

Konstance et al. (1983) explored the EMC value of bacon slices and observed that differences in bacon isotherms were evident as data were plotted on a fat-free, dry basis. Unfortunately, they did not apply any statistical technique to compare the difference between these sorption isotherms.

c. **The effect of characteristics of materials.**

The effects of variety, harvest year, and maturity have not shown consistent results among several research studies.

Brooker et al. (1974) concluded that the effect of variety and maturity on EMC properties might be due to the slight differences in their chemical analysis.

Pfost et al. (1976) used a statistical

technique to test sorption isotherm data obtained from English and U.S. mazie. They concluded that the two varieties used were significantly different.

Pixton and Henderson (1981) compared EMC data of five varieties of Canadian wheat and two varieties of rapeseed. They found that the difference in the EMC/ERH relations of wheat and rapeseed were small and significant only at low moisture contents.

From the research on EMC properties of corn cobs, White et al. (185) concluded that no significant difference existed in desorption ERH values for three selected corn varieties.

Comparing sorption isotherms between sound and broken yellow dent corn, Chung et al. (1972) found the broken corn had higher sorption rates and higher EMC values than those of sound kernels at the same relative humidity. Gustafson (1973) found that split corn kernels tended to have higher EMC values than whole kernels by approximately 0.5%.

**d. Drying treatment**

Bras (1982) described in detail the consequences of the drying process on components of corn kernels.

The hull became separated because of the sudden increase in the steam pressure in the middle of the grain. Separation of the hull allowed proteins and fine starch granules to be lost.

Germ, which is rich in oil, could lose some of its oil through seepage into other parts of the kernel and could become brown if dried at high temperatures.

The starch of the horny endosperm begins to gelatinize at the start of the high temperature drying process. The starch of the floury endosperm can only gelatinize at the end of the drying process.

The protein is affected from the start of the drying process. The proteinic matrix contracts until an intimate amalgam is formed, therefore, the starch granule-proteinic matrix is difficult to separate.

Tuite and Foster (1963) studied the effect of drying treatment on the ERH properties and noticed that the ability to absorb moisture progressively decreased with increasing drying temperature. Corn dried at temperatures of 60 °C and above supported a higher ERH than corn of the same moisture dried at room temperature. Other variables in the drying treatments – airflow rate, batch and continuous-flow drying methods, and initial corn moisture, did not significantly influence EMC and ERH. This phenomenon is very important for safe grain storage.

In a study of the drying temperature effect on the EMC data of dried beef, Iglesias and Chirife (1976a) found that the higher the drying temperature the lower the sorption capacity of dried beef. Using the BET model to analyze the isotherms, they demonstrated that the water contained in the monolayer was affected by the drying temperature. The effect of drying temperatures on the sorption isotherms is shown in Fig. 5.

Gustafson and Hall (1972) studied density and porosity changes of shelled corn during drying and found that physical changes in dimension were not significantly affected by drying temperature. These investigators concluded that the rate of change in the density tended to be equal for all drying temperatures. From the study of the moisture content effect on the volume change of rice kernels, Yamaguchi et al. (1978) found that the density of the rice kernel was not related

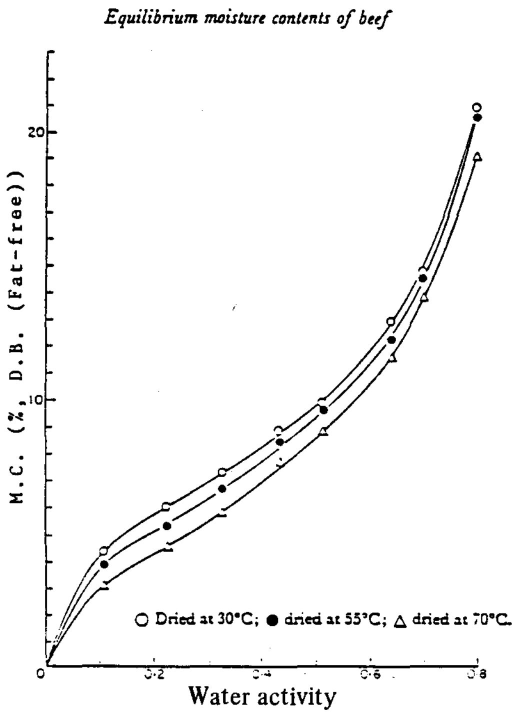


Figure 5. Effect of EMC value of the dried beef by different drying temperature..

to drying temperature except for excessive drying conditions.

Labuza (1968) offered an explanation for the effect of drying teatment on starch. The pre-treatment increases the amount of crystalline water-impenetrable starch at the expense of amorphous starch. The smaller surface area vailable for adsorption means that less water will be adsorbed.

Iglesias and Chirife (1977) concluded that protein changed as a result of heating in the dried state. These changes are: (1) aggregation or cross-linking of unnatured protein, (2) denaturation of proteins followed by aggregation, and (3) interaction of the native or denatured proteins with lipids or carbohydrates. It appears these changes induce the lowering of sorption capacity.

In a study of corn drying conditions and resulting quality for the wet-milling industry, Bras (1982) found that the drying process changed the structure of the grain components including shinkage of the proteinic matrix and the start of gelatinization of the starch. These changes resulted in a different capacity for absorbing water and might explain the effect of drying treatment.

Investigating starch gelatinization during corn nixtamalization, Cabrera et al. (1983) reported that the degree of gelatinization was affected by the temperature and time. The lag time without gelatinization occuring was found for each temperature. The ratio of the ungelatinized portion of starch (1-a) can be expressed as follows:

$$\ln (1\text{-}a) = K^*t$$

t is time and K is the reaction rate which can be analyzed in terms of an Arrhenius-type model. The result may provide a reasonable explanation of the drying effect on the corn kernel.

## e. Hysteresis

Iglesia and Chirife (1976b) reported that the effect of temperature on the magnitude of hysteresis varied among foods. Hysteresis in some cases decreased or was eliminated as temperature increased, while in other cases it remained unchanged or even increased as temperature increased. Hysteresis decreased in magnitude as temperature increased in rice (Wolf et al., 1972; Benado and Rizvi, 1985).

Many theories have been advanced to explain the hysteresis phenomenon in grains. The "ink-bottle" theory was recognized as the best principle (Labuza, 1968; Brooker et al. 1974). This theory assumes that the grain kernel is a porous body having capillaries consisting of narrow, small diameter necks with large-diameter tubes. During desorption, narrow ends of surface pores will trap and hold water internally. In constrast, during adsorption the narrow ends prevent the lage empty pore from being filled.

### f. Temperature effect

Increasing temperature results in a downward shift of the sorption isotherm. The Clausius-Clayperson equation is usually used to express this effect.

$$\ln\left(\frac{Aw2}{Aw1}\right) = \frac{Qs}{R} * \left(\frac{1}{T1} - \frac{1}{T2}\right)$$

Where Qs = heat of sorption in cal/mole.
R = 1.987 cal/mole OK.

Qs is a function of moisture content. Unfortunately, each food has its own Qs value. Therefore, to predict the Aw of a food at any given temperature, one needs to determine at least two sorption isotherms at different temperatures. This method involves indirect and complicated calculations. Therefore, a modified method for adding the temperature term into the ERH/EMC model has been widely adopted by agricultural engineers.

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