

Monolayer moisture, free energy change and fractionation of bound water of red chillies

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Abstract

The equilibrium moisture content and water activity data of red chillies within the temperature range 25–45 °C were used to determine the monolayer moisture, free energy change and fractionation of bound water. The BET equation, Caurie equation and local isotherms fixed the safe storage moisture limits of red chillies as 3.84–6.68%, 5.2–6.01% and 6.03–7.69% d.b. in adsorption conditions and 5.85–9.17%, 5.66–6.77% and 6.02–9.76% d.b. in desorption conditions within a temperature range of 45–25 °C, respectively. The free energy was higher in the desorption process than in the adsorption process of chillies at all the temperatures and moisture contents. The maximum value of free energy was 4437.35 kJ/kg mol at 5% d.b. moisture content and the minimum value was 238.37 kJ/kg mol at 50% d.b. moisture content during desorption of chillies in the temperature range of 25–45 °C. It was observed that the primary, secondary and tertiary bound waters end at moisture contents of 1.5%, 14% and 53.6% d.b., respectively. The bound water in red chillies was limited up to a moisture content of 53.6% d.b. and beyond that was free water, which can be removed easily.

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1. Introduction

In studying the behaviour of food or agricultural commodities during storage and processing, it is important to have information which relates environmental conditions to material properties. The monolayer moisture content is of significant importance to the physical and chemical stability of dehydrated materials with regard to lipid oxidation, enzyme activity, non-enzymatic browning and structural characteristics (Labuza et al., 1970).

Change in free energy during moisture exchange between the product and the surroundings is the energy required to transfer water molecules from the vapour state to a solid surface, or from a solid surface to the vapour state. This is the quantity which can be considered a measure of work done by the system to accomplish the adsorption or desorption process. Thermodynamically, equilibrium is reached when the free energy gradient across the interface

is zero (Nayak and Pandey, 2000). Binding energy is the difference between the heat of absorption of the water by the solid and the heat of condensation of water vapour at the same temperature. It can be considered as a measure of the affinity of food for water (Rizvi, 1986). Thermodynamic principles were applied to water sorption concepts in order to study the binding energy (Soekarto and Steinberg, 1981). Kaminski and Tomczak (2004) described the sorption isotherms as a function of water activity, composition and temperature using multilayer perceptron. It was also mentioned that the isosteric heat and monolayer moisture content can be calculated by applying a multilayer perceptron formula.

Chillies are dried ripe fruits of species in the genus *Capsicum*. The most important quality characters in chillies are colour and pungency. The chillies produced for use as a condiment or a culinary supplement are subjected to long term storage. During that time, important physical, chemical and biological changes take place which have a major impact on the colour and pungency. Therefore, it is necessary to investigate the equilibrium moisture content of

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chillies at various relative humidities and temperatures to specify their optimal storage conditions. The moisture sorption isotherms of red chillies were determined and various models were applied for analysing the experimental data of Hossain and Bala (2000) and Kaleemullah and Kailappan (2004) but the safe storage moisture content limits of chillies at various temperatures were not reported. Hence, a study was conducted to determine safe moisture content limits, free energy change and bound water limits of chillies.

2. Materials and methods

2.1. Experimental procedure

Freshly harvested and dried red chillies of the polyster variety (Coimbatore local) were procured from the local market. Equilibrium moisture contents were determined both in desorption and adsorption conditions at three temperatures (25, 35 and 45 °C) and eight levels of water activities (0.115–0.865) using the static method. Super-saturated salt solutions of different relative humidities, viz., lithium chloride (0.115–0.121), potassium acetate (0.184–0.227) magnesium chloride (0.318–0.332), potassium carbonate (0.432–0.438), sodium nitrite (0.605–0.643), sodium chloride (0.748–0.756), ammonium sulphate (0.794–0.803) and potassium chromate (0.846–0.865), were used to provide constant water activities at temperatures of 25, 35 and 45 °C.

A preliminary experiment was conducted to determine the range of expected values of equilibrium moisture contents by storing samples of chillies in the environments corresponding to 45 °C—0.115 water activity (a_w) and 25 °C—0.865 a_w for a period of 30 days. The range of equilibrium moisture contents was 3–50% dry basis (d.b.). Hence, the samples for adsorption and desorption had to be pre-conditioned. The samples were conditioned to 75% d.b. for desorption purposes and to 2.5% d.b. for adsorption purposes. The samples were conditioned by drying the samples in a hot-air vacuum oven at 70 °C until the desired moisture contents were obtained.

Another preliminary experiment was conducted with conditioned samples to determine the actual time required for attaining the state of equilibrium. After 10 days, the samples were weighed once every two days until a constant weight was observed. It was found that a period of 30 days was required for equilibrium. Hence, a 40-day period was selected to provide a margin for variations in the time to achieve moisture equilibrium.

Glass desiccators (16 cm diameter × 16 cm height) containing about 350 ml saturated salt solutions were used to provide constant relative humidities varying from 0.115 to 0.865. The desiccators were placed in an electric oven at the desired constant temperature and the environment inside the containers allowed to equilibrate. Samples of pre-conditioned chillies (10 g each for desorption and 5 g each for adsorption) were placed in Petri dishes and these dishes

were placed on plastic platforms inside the desiccators to avoid direct contact between chillies and the salt solution. After a 40-day period, the Petri dishes were removed from the controlled atmosphere space and stored in an activated silica gel environment for 3 h to allow them to attain room temperature (Verma and Gupta, 1988). The moisture content of each sample was determined by keeping the samples in a vacuum oven at 70 °C and ≤ 100 mm Hg pressure (AOAC, 1995) until the weight measurement was constant. All the experiments were replicated three times and the averages of three values were used in the analyses.

2.2. Monolayer moisture

Monolayer moisture content is a crucial parameter in the determination of the surface potential of moisture sorbed in food. It was suggested (Rockland, 1969) as a satisfactory specification for the lower limit of moisture in dehydrated food. BET (Brunauer, Emmett and Teller) and Caurie isotherm equations were used to determine the monolayer moisture of chillies (Caurie, 1981). The monolayer moisture content of the BET equation was computed for the samples up to a water activity of 0.592 (Labuza, 1984). The Caurie's monolayer moisture content was computed for the entire range of water activity.

BET equation:

$$\frac{a_w}{M(1-a_w)} = \frac{1}{C_b M_b} + \left(\frac{C_b-1}{C_b M_b}\right) a_w, \quad (1)$$

Caurie equation:

$$\ln\left(\frac{1}{M}\right) = -\ln(C_c M_c) + \left(\frac{2C_c}{M_c}\right) \ln\left(\frac{1-a_w}{a_w}\right), \quad (2)$$

where a_w is the water activity, decimal; M is the moisture content, % d.b.; M_b is the BET's monolayer moisture content, % d.b.; M_c is the Caurie's monolayer moisture content, % d.b.; C_b is the BET's constant; C_c is the Caurie's constant.

The monolayer moisture content of chillies was determined by plotting $a_w/[M(1-a_w)]$ vs. a_w in the case of the BET equation and $\ln[(1-a_w)/a_w]$ vs. $\ln(1/M)$ in the case of the Caurie equation. The values of the slopes and constants of the straight lines of the plots were determined and solved to get the monolayer moisture values of both the equations.

2.3. Local isotherms

Rockland and Nishi (1980) described the concept of localized isotherms using change in Gibbs' free energy data. The change in Gibbs' free energy (F) is given as

$$F = -RT \ln(a_w), \quad (3)$$

where R is the universal gas constant, 8.314 kJ/kg mol K; T is the temperature, K; a_w is the water activity, decimal.

Free energy vs. equilibrium moisture contents was plotted and two sharp changes in the plot were recorded as the first and second break points of the local isotherm.

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