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Sorption Isotherms of Traditional Indian Dairy Products: A Review

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Abstract

The moisture sorption isotherms describe the equilibrium relation between water activity (a_w) and equilibrium moisture content of food at constant temperatures and pressures condition. An understanding of sorption characteristics of traditional Indian dairy products provides valuable information to characterize storage condition and packaging problems. The isotherms of tradition dairy products generally follow sigmoid shape, and are of BET type II classification.

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Introduction

Water is the main component of many food products and the storage stability of products depends upon the interaction of water with other components present in the product. The status of water in product plays a crucial role in the preservation of biological materials in both the raw and processed state. The simplest way of expressing such a status is the concept of water activity. Specific changes in color, aroma, flavor, texture, stability, and acceptability of raw and processed food have been associated with relatively narrow water activity ranges [1-26]. Water activity (aw) is an important tool to predict available water in foods and the physical state of solid foods [27]. Water activity is considered as one of the most important parameters in food preservation and processing, thus several food preservation techniques rely on lowering the water activity so as to reduce the rates of microbial growth and chemical reactions. Water activity and sorption behavior are two most powerful concepts available for understanding and controlling shelf life of foods [17]. Water activity is a physical measure of active water available in foods. Thus, it is the state of water rather than the total water content, which is important as far as microbial proliferation is concerned. It has been observed that the state of water is related to the vapor pressure of food. Greater the proportion of free water present greater the vapor pressure and vice versa. Increased bound water reduces vapor pressure [28-32]. The definition of water activity is based on the chemical potential of water within a food system, which at equilibrium must be same as the chemical potential of water in the surrounding of the food. Hence water activity can be defined as the ratio of the vapour pressure of water in food and the vapor pressure of pure water at the same temperature and pressure conditions [17]. At equilibrium, the water activity is related to the relative humidity of the surrounding atmosphere [28], which is given by:

$$a_w = \frac{P}{P_0} = \frac{ERH}{100}$$

Where,

P=Vapor pressure of water in food material at any given temperature

P_o=Vapor pressure of pure water at that temperature

ERH=Equilibrium relative humidity

Water activity has become one of the most important intrinsic properties used for predicting the growth of microorganisms in food due to its direct influence on product stability and quality [32].

Moisture Sorption Isotherm

The relationship between the equilibrium moisture content (dry basis) and water activity of the food, over a range of values, at a constant temperature and under equilibrium conditions, yields a moisture sorption isotherm when expressed graphically [18]. Generally, food, when kept in different relative humidity, absorbs or desorbs moisture depending on their water activity [13]. This isotherm curve can be expressed in two ways; adsorption or desorption. The adsorption and desorption processes are not fully reversible, therefore a distinction can be made between the isotherms by determining whether the moisture levels within the product are increasing or decreasing. Brunauer et al. [4] described five types of isotherms. Type-I is the well-known Langmuir isotherm, obtained assuming monomolecular adsorption of gas by the porous solids in a finite volume of voids. Type-II is the sigmoid isotherm obtained for soluble products, which exhibits asymptotic trend as water activity approaches to one. Type-III, known as the Flory-Higgins isotherm, accounts for crystalline substance. Type-IV isotherm describes adsorption by a swellable hydrophilic solid until a maximum of hydration sites are reached. Type-V is the BET multilayer adsorption isotherm, observed for adsorption of water vapor on charcoal. Most food products generally show type-II isotherms having a sigmoid shape [11]. Moisture sorption isotherm curve is explained by three regions. Region I describes the strongly bound water with an enthalpy of vaporization considerably larger than that of pure water. In many aspects, it behaves essentially as a part of the solids. It is generally understood that these first water molecules are sorbed at the active polar groups in solids [22]. Region II represents water fraction less firmly bound than the first. Region III water is more or less free water mechanically entrapped in the void spaces of the systems, having nearly all properties similar to those of bulk water. Isosteric heat of sorption often referred to as differential heat of sorption derived from temperature dependent isotherm is used as an indicator of binding energy of absorbed water by solid [6,23]. The net isosteric heat of sorption was determined using the Clausius–Clapeyron equation [19-36]. This provides an indication of the binding strength of water molecules to the solids and has a bearing on the energy balance of drying and freezing equation. Also, the end point of the drying process is generally determined by the desired water activity of the finished product.

Importance of sorption isotherm

Water sorption isotherms illustrate the steady-state amount of water held by the food solids as a function of a_w at constant temperature [17]. Thus, the moisture sorption isotherm is an extremely valuable tool for food scientists since it can be used to predict which reactions will decrease stability at a given moisture; it allows for ingredient selection to change the a_w to increase stability and can be used to predict moisture gain or loss in a package with known moisture permeability [2]. Water sorption isotherms in foods are of special interest in many aspects of food storage, drying, preservation by dehydration, especially for the prediction of the shelf life of a dried product in a packaging material or the prediction of drying times of foodstuffs [11]. The monolayer moisture content of a food product is an effective method of estimating the amount of bound water to specific polar sites in dehydrated food system and at this moisture product seemed to be stable against microbial deterioration [23]. It helps in mixing operations and development of a new product formulation, determination of enthalpy of sorption and desorption of water at two different temperatures, determine critical water activity or moisture content limits for crispness, hardness, and flow properties optimize moisture contents at a safe water activity that maximizes moisture and avoids over drying. Also allow rapid moisture content determination from water activity analysis through isotherm curve [2].

Measurement of moisture sorption isotherm

Gravimetric method: In this method, food is exposed to controlled environment of relative humidity at defined temperature. The weight of the sample is monitored at different intervals till a time there is no change in weight as the food attains equilibrium with the environment. Such determinations at several relative humidity conditions yield a sorption isotherm.

Manometric and hygrometric method: In this method, food of known moisture content is allowed to attain equilibrium with a small headspace in a tight enclosure and partial pressure at that water activity is measured manometrically. Gravimetric method recommended by COST (Co-operation in the field of Scientific and Technical Research in Europe) projects 90 and 90-BIS on physical properties of foodstuffs is the most widely used and reliable method for measurement of sorption isotherm [35].

Hysteresis Phenomenon

Water sorption hysteresis is the phenomenon according to which two different paths exist between the adsorption and desorption isotherm. The Desorption isotherm usually lies above the adsorption and a closed hysteresis loop is formed [12]. Moisture sorption hysteresis has important theoretical and practical implications in foods. The theoretical implications are evidence of irreversible of the sorption process and the validity of the equilibrium thermodynamic process. The practical implication deals with the effect of hysteresis in chemical and microbiological deteriorations. Due to hysteresis a much lower vapor pressure is required to reach a certain amount of water by desorption than adsorption [13]. Interestingly, reported that a few fungi and bacteria grew rapidly in the system prepared by a desorption processes than by adsorption at same water activity [1]. In general the types of changes encountered upon adsorption and desorption will depend on the initial state of the sorbent (amorphous versus crystalline), the transitions taking place during adsorption, and the speed of desorption [37-39]. Thus, the principal factors affecting hysteresis are composition of the product, its temperature, storage time, drying temperature and the number of successive adsorption and desorption. In general, at low moisture content the heat of desorption was significantly higher than the heat of adsorption [9]. Depending on the type of food and the temperature, a variety of hysteresis loop shapes can be observed [35]. In high-sugar or high-pectin foods, hysteresis mainly occurs at below the monolayer region, although the total hysteresis is large, there is no hysteresis above 0.65 [20]. In high protein foods, the phenomenon is extended through an a_w of about 0.85 [12]. Overall, total hysteresis decreases as sorption temperature increases [37].

Isotherm Models

There are various models with corresponding mathematical equations for predicting or data fitting of sorption isotherm of foods. To check whether water activity can be accurately predicted from the experimental data of the moisture using a particular equation is called fitting of equation to that data. The mathematical expression based on two and three parameter models are the most commonly used. The most commonly used two-parameter model is the BET model. However, is limited to lower a_w of up to 0.3-0.5. To account for a wider range a_w, three-parameter GAB model which is applicable up to a_w of about 0.9 and is applied to various foods [34]. Due to the complex nature of foods, no single model is general enough to represent the sorption isotherm of foods. BET and GAB models are most commonly used in predictions of sorption isotherms of foods. Iglesias and Chirife [11] reviewed several equations for modeling equilibrium moisture content and reported that some models were adequate to characterize the sorption behavior of particular foods for the given range of temperature and a_w or relative humidity.

Sorption Isotherm of Traditional Indian Dairy Products

Sorption isotherm of *chhana podo*

Rao et al. [25] studied EMC of *chhana podo* using the static gravimetric method, at 5°C and 35°C, over a range of water activity from 0.23 to 0.98. The isotherms obtained were of sigmoid shape, and of BET type II classification. The EMC, at a given water activity decreased as the temperature increased from 5°C to 35°C. Amongst the 12 sorption models tested, the Curies model best described the EMC/water activity relationship of *chhana podo* by giving a closest fit to the experimental sorption data. The EMC at 5°C and 35°C, increased from 5.75% and 3.55% at 0.23 a_w to 104.82% and 88.64% at 0.98 a_w, respectively. The net isosteric heat of *chhana podo* ranged from 0.081 kJ mol^{-1} at 42% moisture content (db) to 17.26 kJ mol^{-1} at 2% moisture content. The information on isosteric heat of sorption is helpful in determining the energy of water-binding and heat energy requirements during baking.

Sorption isotherm of *sandesh*

Sahu and Das [29] studied the sorption isotherms of the *sandesh* at 20°C and 30°C and indicated that the isotherms were smooth and sigmoid shaped at both the temperatures. The initial moisture content of the *sandesh* samples was 12-14% (wb*).* Three zones on isotherm are noted; zone-I (a_w: 0.0–0.2), zone-II (a_w: 0.2–0.6) and zone-III (a_w: 0.6–1.0). Moisture sorption characteristics of *sandesh* could be predicted agreeably with Caurie model. The values of isosteric heat of sorption was found to increase with decreasing moisture content at lower moisture content and approached the value of heat of vaporization of free water above 17.25% (*db*). A reduction in moisture adsorption and water binding capacity was observed as the temperature of adsorption increased from 20 and 30^{0}C. Monolayer moisture content of *sandesh* [15] was found to be 5.892 and 5.235% (*db*) at 20 and 30°C respectively.

Sorption isotherm of *peda*

Pagire [21] studied sorption isotherms of *peda* prepared from buffalo whole milk at 15, 25 and 35°C. All sorption isotherms demonstrate an increase in EMC with increasing water activity and beyond 0.9 a_w the increase in EMC was sharp. This behavior is manifested in the form of a sigmoid shape curve, thus reflecting a Type II isotherm [4], and GAB model shows best fit to experimental data. The isotherms showed that the product adsorbed proportionately more water towards the later part of the curve. Effect of temperature on isotherm shows that from 0.33 to 0.88 a_w all the three curves of adsorption and desorption were clearly distinguishable. Above 0.88 a_w and below 0.33a_w the curves are superimposing on each other. The hysteresis effect exhibited by *peda* shows that the adsorption and desorption isotherms were distinctly apart from each other. The hysteresis loop was classified as Type-C according to Everett and Whitton classification, which begins about 0.9 a_w and extends over the rest of the isotherm up to 0.12 a_w. The hysteresis effect became minimum beyond 0.85 a_w and the adsorption and desorption isotherm of *peda* coincides with each other at about 0.91 a_w at 15^{0}C, 0.9 a_w at 25^{0}C and 0.88 aw at 35^{0}C. Maximum hysteresis was observed between 0.30 to 0.85 a_w. The effect of increasing the isotherm temperature was found decreasing the total hysteresis from 1.55 units at 15°C to 1.35 at 25°C and 1.21 at 35°C. The total energy of hysteresis decreased with increase in temperature; it was 60.61 kJ/kg of water at 15°C, 45.63 kJ/kg of water at 25°C and 32.25 kJ/kg of water at 35°C.

Sorption isotherm of ready to use *basundi mix*

Sharma et al. [33] studied the effect of temperature on desorption isotherms of ready to use *Basundi mix* in the temperature range of 5-45°C. Isotherms were sigmoid shape corresponding to type II. Modified Mizrahi followed by GAB were found to be best fitted at all the temperatures. The isotherm showed inversion at higher temperatures and adsorbed proportionately more moisture towards the later parts of the curve particularly above 0.7 a_w. Monolayer moisture contents of the *Basundi mix* formulation were in the range of 2.11 to 3.58 g /100 g solids.

Sorption isotherm of ready to use *gulabjamun mix powder*

Govardhan [6] developed sorption isotherms of *Gulabjamun mix powder (GMP)* at 15, 25, 35 and 45°C using a gravimetric-static method followed a type II (sigmoidal) shape. Monolayer moisture content of GMP decreased from 0.1002 g water/g of solids at 15°C to 0.0772 g water/g of solids at 45°C. The heat of sorption was found to increase initially with increase in moisture content and attain a peak value of 8213 kJ/kg at around 9% moisture level and then decrease.

Sorption Isotherm of *Khoa*

Sawhney et al. [30] determined the isotherm for *khoa* at four different temperatures over a_w range of 0.11-0.97. The isotherms obtained were typical type II sigmoid curves and were fitted to the GAB equation. The equilibrium moisture content rise gradually at lower water activity (0.15-0.45), followed by a steep rise above 85% relative humidity. At all a_w, EMC was lower at higher temperature. The a_w of *khoa* increased with increasing temperature up to 0.9; above this the effect of temperature on a_w diminished. Sawhney et al. [31] determined the adsorption and desorption isotherm of *khoa* at 25 and 45°C, no discontinuities were observed and data showed a sigmoid shape of type II isotherm according to BET classification [17]. The hysteresis effect of *khoa* at temperature 25 and 45°C was moderate for the water activity less than 0.1. It increased at high water activities and occurred primarily in the water activity range of 0.35 to 0.65 at both 25 and 45°C. The hysteresis effect diminished beyond 0.8 water activity and the adsorption and desorption isotherm of *khoa* coincided with each other at water activity above 0.96 at 25˚C and above 0.90 at 45˚C. The hysteresis effect was moderate in monolayer moisture content region of *khoa*. The increase in isotherm temperature from 25 to 45°C, the total hysteresis in *khoa* was reduced from 2.74 to 1.59 units and total hysteresis energy reduced from 11.25 kJ/kg to 6.35 kJ/kg of water respectively.

Sorption isotherm of *milk burfi*

Ramkrishna et al. [24] determined the sorption isotherm of milk *burfi*, using sugar and sugar substitutes. The product was prepared by replacing sugar with sorbitol, maltodextrin (MD) + polydextrose (PD), and PD alone, along with aspartame to give an equivalent sweetness level compared to sugar. The sorption curve obtained is sigmoidal in shape, depicting one inflection point, which is characteristic of materials with high sugar content. The curves show three regions: region A, corresponding to <0.2 of a_w, as a monomolecular film of water, region B as a multilayer at a_w 0.22-0.7 and region C for a_w>0.7 corresponding to condensation of water in the pores of the material followed by dissolution of soluble material. The slope of the curve was less at lower a_w, whilst with increasing a_w the slope increased rapidly. In general, the equilibrium moisture content increases rapidly at low water activity a_w (0-0.15), then rises slowly between aw 0.15 and 0.7, followed by a steep rise above a_w 0.7. The sorption curves for *burfi* with sorbitol shifted to the left compared to that of sugar, whereas for those prepared with MD+PD or PD, the curves are similar to that of burfi made with sugar. Out of seven models fitted to experimental data, the GAB model showed a better fit compared to other models, as it is applicable to a wide range of water activity.

Sorption isotherm of *dudh churpi*

Hossain et al. [10] determined moisture sorption characteristics of *Dudh Churpi*, a traditional milk product in India at 15, 25, 35 and 45°C. The isotherms were typical type II sigmoid in shape. Nine equations, namely Bradley, Henderson, Iglesias and Chirife, Khun, Mizrahi, GAB (Guggenheim-Anderson-de Boer), modified Mizrahi, Oswin and Caurie were fitted to the sorption data. Caurie's equation was found to be suited best to predict equilibrium moisture content of *Dudh Churpi*. Monolayer value, density of sorbed water, number of monolayer and area of adsorbent decreased with the increase in temperature.

Sorption isotherm of *kheer*

Kumar et al. [16] developed moisture sorption isotherms of *Kheer* in the temperature range of 10-40°C and indicated that the sorption isotherms were sigmoid type II, common to most foods. While a negative temperature coefficient was evident with regard to the EMC. Water activity exhibited an appreciable interaction effect; the curves representing 25°C and, 40°C intersected each other above water activity of 0.60. This temperature dependence of EMC in relation to water activity could be attributed to the presence of sugar. Sorption data for *Kheer* were best described by GAB model.

Sorption isotherm of *sandesh powder*

Khojre and Khedkar [15] studied the sorption isotherms of *Sandesh powder* at the temperature of 10°C, 20°C, and 40°C and the relative humidity of 0.11-0.97%. It was found that at all temperatures, as the water activity increase EMC also increased. BET, GAB and Caurie models in row were found good over different model to predict the experimental moisture sorption data and to characterize the sorption behavior.

Sorption isotherm of *lal peda*

Kumar et al. [16] studied moisture sorption isotherms of *lal Peda* at 10, 25 and 37°C at water activity range of 0.113 to 0.868 using standard saturated salt solutions. The shape of isotherms obtained was of sigmoid (type II) at all temperatures. The GAB model fitted best at three temperatures out of seven models tested. The Monolayer moisture content as calculated from the best fitted GAB model for desorption and adsorption processes at 10, 25 and 37°C were 3.852, 4.551, 5.235 and 3.214, 4.103 and 5.253% on a dry basis, respectively. The values of isosteric heat of sorption as calculated from Clausius-Clapeyron equation was found to increase with decreasing moisture content at lower moisture content

Sorption isotherm of *thabdi*

Kesha [14] studied the sorption isotherms of *Thabdi* at 15, 25 and 35°C. It showed sigmoid in shape type II isotherm. There was increase in the equilibrium moisture content with the increase in water activity. Out of five different models tested GAB model is best fitted equation for both adsorption and desorption isotherms.

Sorption isotherm of dietetic *rabri*

Ghayal et al. [7] studied sorption isotherm of dietetic *Rabri* at 10, 25, 37°C over the water activity range of 0.11-0.86. Sorption isotherms at 37°C were sigmoid (type II) curves, while at 10°C, they were of type V and Type I at 25°C for both adsorption and desorption processes. Five sorption models were tested to fit the experimental data. Halsey's model for 10°C, Curie’s model for 25°C and Modified Mizrahi's model for 37°C were found to be the best fit. There was a clear hysteresis effect at 10°C, whereas the effect gradually decreased and diminished at 37°C.

Sorption isotherm of *kalakand*

Deshmukh [5] determined the moisture sorption characteristics of *Kalakand* prepared from standardized milk by using static gravimetric method, at 15, 25, 35°C, over the range of water activity (a_w) 0.11 to 0.97. The isotherms obtained were of Type-II (sigmoid) shape. All isotherms show that EMC, at given water activity decreases with increased in temperature. GAB, Caurie, Oswin, Modified Mizrahi and Halsey models were applied to the experimental isotherm data of *Kalakand*. Out of all five models tested, the GAB model was found most suitable for describing of sorption data which was having least RMS value. The sorption data were analyzed to determine monolayer moisture content as calculated by the best fitted GAB model for desorption and adsorption processes at 15, 25 and 35°C were 5.621, 4.589 and 4.185 and 4.249, 3.520 and 3.177 g/100 g solid, respectively.

Conclusion

Moisture sorption isotherms serve as an important tool for predicting moisture relations in products and with the help of modern instrumentation (dynamic vapor sorption method-DVS) it possible to analyze the moisture relations of the product instantly. Moisture sorption isotherms play an important role to quantify the shelf life of tradition products due to their sensitivity to moisture changes. To date, no one equation or model gives accurate results through the whole range of water activities, and for all types of foods. The thermodynamic properties like isosteric heat of desorption of heat desiccated Indian milk product used in calculating energy requirement for dehydration of those products and for designing or evaluating drying equipment and processes. The knowledge of sorption isotherm of TIDP helps to predict their shelf life and optimum storage condition.

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