



Water sorption isotherms of cooked hams as affected by temperature and chemical composition

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Abstract

This study was focused on the determination of the chemical composition and experimentally obtaining the sorption isotherms for four samples of commercial cooked ham subjected to simulated commercial storage conditions. The isotherms were determined using the gravimetric method. The mathematical models of Guggenheim, Anderson and de Boer; Brunauer, Emmett and Teller; Halsey; Henderson; and Peleg were fitted to the experimental data. The Guggenheim, Anderson and de Boer model was chosen to best describe the isotherms as it had a very good fit. The increase in temperature reduced the equilibrium moisture content of the product. Increased relative humidity resulted in an increase in equilibrium moisture content of the product regardless of storage temperature. The differences in chemical composition between the samples affect the desorption isotherms. The higher the content and availability of the protein or the lower the fat content, the higher the equilibrium moisture content of the product.

Keywords: mathematical modelling; equilibrium moisture content; water activity; protein; fat.

Practical Application: The sorption isotherms of cooked ham can be influenced by storage temperature, by relative humidity and by the chemical composition of the product. The higher the storage temperature is, the lower the equilibrium moisture content. Increased relative humidity results in higher equilibrium moisture content. And the lower the moisture:protein ratio (MPR) or the lower the fat content is, the higher equilibrium moisture content. This information can help the meat industry to maintain the cooked ham humidity and increasing its shelf-life.

1 Introduction

Cooked ham is one of the most popular processed meat products among Brazilian and European consumers (Válková et al., 2007). The growing consumption of cooked ham is linked to recent efforts that focused on increasing its acceptability through the evaluation of physical and sensory characteristics, such as appearance, texture, flavor and color (Ávila et al., 2014; Barbieri et al., 2016; Delahunty et al., 1997; Tomović et al., 2013).

The processing of cooked ham consists basically of incorporating brine in the pork by tumbling and massaging, followed by cooking to solubilize the proteins and adequate cooling to take the ham out of the mold (Talens et al., 2013). Adequate cooling is necessary so that the product does not deform after being taken out of the mold. The final product is then transported and maintained under controlled low temperatures until later use. As a consequence of all these steps, the final quality of the hams depends on many factors, including the origin and composition of the ingredients and the processing conditions (Válková et al., 2007).

The selection of the ingredients is considered essential in ensuring the sensory quality from storage until the consumption of the product (Toldrá et al., 2010). The composition of the

ingredients can affect the way water molecules bind in the food matrix, causing changes in the water activity (a_w) and, consequently, on food stability (Rizvi, 2005). In the specific case of meat products, these alterations can influence not only the microbial growth but also the meat texture. Enzymes such as proteases and lipases may have their activity affected by different a_w conditions (Toldrá, 2006).

Using the sorption isotherms is an interesting way to provide information about the water activity and equilibrium moisture content of a product at a certain temperature and ambient relative humidity (Ahmat et al., 2014). Best processing and storage conditions can be reached by understanding water sorption behavior in food products (Brett et al., 2009; Sharma et al., 2018). This procedure can be carried out at different temperatures, avoiding unwanted changes and increasing shelf life (Staudt et al., 2013). Water sorption isotherms of different meat products have been studied by a number of authors, considering the effect of temperature (Clemente et al., 2009; Delgado & Sun, 2002; Comaposada et al., 2000; Cortés & Chejne, 2010; Lind & Rask, 1991; Lopes Filho et al., 2002). However, little information has been specifically reported about the sorption isotherms of cooked ham.

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Several models (empirical, semi-empirical and theoretical) can be used to mathematically describe sorption isotherms for meat. It is consolidated in literature that the most of the food sorption isotherms can be expressed analytically by the Guggenheim-Anderson-de Boer (GAB) equation (Al-Muhtaseb et al., 2004; Bizot, 1983; Chirife & Iglesias, 1978; Lewicki, 1997). Peleg (1993) proposed a dual power expression which, when compared to the GAB equation, produces a good or better fit to the experimental data. On the other hand, the parameters of the GAB model give insights about the interactions between the sorbent-sorbate, as well as the multilayer-monolayer moisture (Quirijns et al., 2005).

The modeling of water sorption isotherms plays an important role in food storage. A well-fitted model can predict the gain or loss of water through the equilibrium moisture content. For example, Comaposada et al. (2000) stated that if the water activity of pork on the surface is high, an increase in temperature and/or a decrease in relative humidity can produce a significant loss of moisture content. This phenomenon may increase the dehydration speed inside the meat product, which is not always desirable. The water loss during storage can prejudice not only the meat quality, but also the commercial value of the product as they are commonly sold by weight.

In this way, the aim of this research was to study the influence of the chemical composition on the sorption isotherms of four brands of commercial cooked ham. For this, the cooked hams were firstly characterized according to their chemical composition. Then, the corresponding water sorption isotherms were obtained at different common storage temperatures for a wide range of relative humidities. The different proposed models were evaluated after applying the models to the experimental data.

2 Materials and methods

2.1 Sample preparation

Cooked hams of four different brands (Sample 1 – S1, Sample 2 – S2, Sample 3 – S3 and Sample 4 – S4) were purchased at a local store in São José do Rio Preto, São Paulo, Brazil. For each brand, three different lots were bought and used in this study. Each lot purchased was chopped and homogenized. The samples, after homogenization, were packed in polyethylene bags (0.15 mm of thickness) and vacuum packed. They were wrapped in aluminum foil and stored in a freezer (-12 °C) until their use. To conduct the analysis, the cooked ham samples were thawed in cold storage at 5 °C.

2.2 Chemical composition

The chemical composition analysis was done in triplicate for the four samples using the methods described by Association of Official Analytical Chemists (1997). Moisture content was determined in an oven, protein by the micro-Kjeldahl method, ash by incineration in a muffle furnace and fat content using the Soxhlet method. The total carbohydrates were determined by difference.

2.3 Obtaining the sorption isotherms

The sorption isotherms of ham samples were determined by the static gravimetric method (Jowitt et al., 1983), following the procedures for obtaining desorption isotherms at 2, 9, 16, 24 and 30 °C. To produce and maintain the relative humidity between 6.1% and 92%, saturated solutions of LiBr, LiCl, LiI, MgCl₂, NaI, NaBr, KI, NaNO₃, NaCl and KCl were prepared by dissolving sufficient quantities of each these salts (Sigma-Aldrich, St. Louis, USA) in deionized water. The saturation of the solutions was ensured by the presence of a small portion of undissolved salt at the bottom of the flask. At equilibrium, the a_w of salt solutions in Table 1 corresponds to the relative humidity of the air. These values were obtained in a study by Labuza (1963) at different temperatures.

For each measurement, three repetitions of about 1 g of sample of ham were placed in small plastic containers open at the top, which were, in turn, placed on a support in each jar to avoid contact with the salt solution. The jars were then placed in a chamber with a controlled temperature (BOD, Model TE-391, TECNAL, Brazil) at temperatures of 2, 9, 16, 24 and 30 °C. The weights of the samples were monitored until the moisture content, on a dry weight basis, did not change more than 0.1% (after approximately five weeks) determining the point of equilibrium had been reached. For each batch of ham, the initial moisture content was determined according to the Association of Official Analytical Chemists (1997) method for subsequent determination of the equilibrium moisture content (X_{eq}). The values for X_{eq} as a function of a_w at the fixed temperatures were used to plot sorption isotherms curves for the cooked ham samples.

2.4 Modelling of sorption isotherms

Five isotherm models (Table 2) were chosen to fit the experimental data of equilibrium moisture content and a_w at all temperatures studied. Non-linear regressions were carried out using the OriginPro 8.0 software (OriginLab Corporation, Northampton, USA) to adjust the mathematical models. The accuracy of fit of each model was evaluated based on the adjusted coefficient of determination (R_{adj}^2) and the Root Mean Squared Error (RMSE). Coefficients of determination greater than 0.98 indicate a good fit and the RMSE close to zero shows fidelity to the experimental data (Cantu-Lozano et al., 2013; McLaughlin & Magee, 1998; McMinn, 2006).

Table 1. Water activity of the salt solutions at different temperatures.

| Salt solution | Temperature (°C) | | | | |
|-------------------|------------------|-------|-------|-------|-------|
| | 2 | 9 | 16 | 24 | 30 |
| LiBr | 0.076 | 0.072 | 0.068 | 0.064 | 0.061 |
| LiCl | 0.112 | 0.112 | 0.112 | 0.112 | 0.112 |
| LiI | 0.224 | 0.209 | 0.194 | 0.180 | 0.167 |
| MgCl ₂ | 0.334 | 0.334 | 0.331 | 0.327 | 0.322 |
| NaI | 0.434 | 0.428 | 0.415 | 0.396 | 0.374 |
| NaBr | 0.642 | 0.624 | 0.603 | 0.581 | 0.559 |
| KI | 0.740 | 0.724 | 0.708 | 0.693 | 0.679 |
| NaNO ₃ | 0.789 | 0.775 | 0.760 | 0.745 | 0.729 |
| NaCl | 0.756 | 0.755 | 0.754 | 0.753 | 0.752 |
| KCl | 0.881 | 0.871 | 0.861 | 0.851 | 0.842 |

3 Results and discussion

3.1 Chemical composition

In general, the samples presented very similar composition to the ones published in the literature (Del Campo et al., 1988; Desmond et al., 2000; Talens et al., 2013). In order to verify the significant differences among the samples, triplicates were subjected to the analysis of variance and to the Tukey test at 95% of confidence. The results of chemical composition are shown in Table 3.

All the samples were different ($p < 0.05$) with respect to fat content, presenting values between 1.06 and 2.58%. Although the fat contents are statistically different, these differences are considered low and are inherent to the raw material (leg of pork) composition that may have small changes in moisture content, fat and final protein. S4 had a lower amount of fat, which was considered statistically different ($p < 0.05$) from the other samples. According to Dutra et al. (2012), meat products with lower fat contents tend to present softer texture and poor binding properties.

For the protein, the results were between 19.12 and 19.86%, with significant statistical differences. According to Brazilian legislation (Brasil, 2000), the minimum protein content for this

product is 14%, and a maximum MPR ratio of 5.35 is suggested, so all samples are in compliance. These differences are probably a result of variations on the quality of the raw material, as well as on the amount and concentration of the injected brine (Casiraghi et al., 2007). As S4 had the highest protein content ($p < 0.05$), the MPR presented lower values when compared to the other samples. In addition, Spanish legislation characterizes these cooked hams in the extra category, which must have MPR less than 4.13 (Talens et al., 2013).

The carbohydrate contents were also compliant, since the upper limit is 2% according to the Brazilian legislation. These carbohydrates can be represented by dextrose, which is used to provide taste to cooked hams (Toldrá et al., 2010).

3.2 Sorption isotherms

Experimental data of equilibrium moisture content showed that water desorption occurred for all samples. The values of X_{eq} ranged from 5.4% up to 63.4% on a dry basis (5.1% and 38.9%, respectively, on a wet basis), which were lower than the initial moisture content. They increased at lower temperatures and higher a_w , being in accordance to the adsorption and desorption behavior of other foodstuffs such as dairy products, powdered beverages, fruits and vegetables and, especially, meat products (Ahmat et al., 2014; Comaposada et al., 2000; Cortés & Chejne, 2010; Delgado & Sun, 2002; Gabas et al., 2007; Kaymak-Ertekin & Gedik, 2004; Lomauro et al., 1985; Singh et al., 2001).

The best fits to the experimental data were obtained by the Peleg model (Eq. 5, Table 2) and the GAB model (Eq. 2, Table 2). Considering all the samples of cooked ham at the temperatures studied, the R_{adj}^2 for the Peleg model ranged from 0.9960 to 0.9999 and the $RMSE$ from 0.0001 to 0.0075. Meanwhile, for the GAB model, R_{adj}^2 ranged from 0.9854 to 0.9939 and the $RMSE$ from 0.3137 to 0.0086. The Henderson model (Eq. 3, Table 2) had the lowest R_{adj}^2 , implying that this model does not closely describe the experimental data for cooked ham. The Halsey model (Eq. 4, Table 2) showed good accuracy, mainly for lower temperatures. However, at higher temperatures, this model showed a slightly higher lack of fit than GAB model. With respect to the Brunauer, Emmett and Teller (BET) model, the GAB model also presented slight higher accuracy, which might be attributed to the limited range of water activity (up to 0.3-0.4) that the BET model is able to fit (Timmermann et al., 2001).

Table 2. Models used to fit sorption isotherm data from cooked hams.

| Model | Equation |
|-------------------------------------|---|
| BET (Brunauer et al., 1938) | $X_{eq} = \frac{X_m C a_w}{(1 - a_w)(1 - a_w + C a_w)} \quad (1)$ |
| GAB (Chirife & Iglesias, 1978) | $X_{eq} = \frac{X_m C_g K a_w}{(1 - K a_w)(1 - K a_w + C_g K a_w)} \quad (2)$ |
| Henderson (1952) | $X_{eq} = \left(\frac{-1}{H_1} \ln(1 - a_w) \right)^{1/H_2} \quad (3)$ |
| Halsey (Van Den Berg & Bruin, 1981) | $X_{eq} = (-h_1 \ln(a_w))^{(-1/h_2)} \quad (4)$ |
| Peleg (1993) | $X_{eq} = k_1 a_w^{n_1} + k_2 a_w^{n_2} \quad (5)$ |

$C, C_g, K, k_1, k_2, h_1, h_2, n_1$ and n_2 are constants, a_w is the water activity (relative humidity of salt solutions, decimal), X_{eq} is the equilibrium moisture content (dry basis), and X_m is the monolayer moisture content (dry basis).

Table 3. Average values (\pm standard deviation) of the percentage composition of the cooked hams.

| % | Samples | | | |
|--------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| | S1 | S2 | S3 | S4 |
| Fat | 2.18 \pm 0.01 ^c | 2.58 \pm 0.01 ^a | 2.38 \pm 0.01 ^b | 1.06 \pm 0.01 ^d |
| Protein | 19.19 \pm 0.06 ^c | 19.12 \pm 0.04 ^c | 19.67 \pm 0.10 ^b | 19.86 \pm 0.14 ^a |
| Moisture | 74.26 \pm 0.33 ^a | 73.76 \pm 0.46 ^a | 74.09 \pm 0.46 ^a | 74.32 \pm 0.33 ^a |
| Ash | 2.76 \pm 0.01 ^b | 3.18 \pm 0.01 ^a | 3.06 \pm 0.01 ^a | 2.98 \pm 0.01 ^a |
| Water/Protein mean ratio | 3.87 | 3.86 | 3.77 | 3.74 |
| Carbohydrates* | 1.61 | 1.36 | 0.80 | 1.78 |

Averages followed by the same letter in the same line do not show significant differences in the Tukey test at 95% of confidence. *Calculated by difference from the average fat, protein, moisture and ash contents.

Despite the Peleg model having a greater R_{adj}^2 value and lower RMSE, the GAB model was chosen to better describe the sorption isotherms of cooked ham at different storage temperatures. The fitting parameters of the GAB equation are given in Table 4 for all samples and temperatures. The GAB model is considered the most versatile model available in the literature for isothermal fit of food (Al-Muhtaseb et al., 2004; Telis et al., 2000). Moraes & Pinto (2012) also recommended the GAB model to describe the water sorption in food matrices.

This theoretical triparametric model is suitable for application in food engineering and allows a very good fit for almost all types of food with an a_w ranging between 0.1 and 0.9 (Anderson & Hall, 1948; Saravacos et al., 1986). The biggest advantage is that these parameters have physical meaning, different from the empirical and semi-empirical models. These parameters also provide important information on the state of the water in food. In the GAB equation, for example, the concept of monolayer moisture content (X_m) is taken into account (Maroulis et al., 1988). This is related to the stability and shelf-life of the product (Rosa et al., 2010; Yan et al., 2012). The values of X_m extracted from the GAB equation (Eq. 2, Table 2) tended to decrease with increasing temperature. This behavior is reported in the literature from the studies of Iglesias & Chirife (1976 a,b) through to recent reports for different food matrices (Freitas et al., 2016; Owo et al., 2016; Polachini et al., 2016). This tendency

Table 4. Fitting parameters of GAB equation for all samples at the different temperatures.

| | Temperature (°C) | | | | |
|-------------|------------------|---------|---------|----------|---------|
| | 2 | 9 | 16 | 24 | 30 |
| | Sample 1 | | | | |
| X_m | 0.0754 | 0.0688 | 0.0677 | 0.0559 | 0.0536 |
| C_G | 26.7943 | 25.9835 | 27.7396 | 153.6238 | 94.7108 |
| K | 0.9831 | 0.9968 | 0.9986 | 1.0204 | 1.0357 |
| R_{adj}^2 | 0.9912 | 0.9839 | 0.9820 | 0.9913 | 0.9895 |
| RMSE | 0.0126 | 0.0160 | 0.0154 | 0.0090 | 0.0102 |
| | Sample 2 | | | | |
| X_m | 0.0755 | 0.0712 | 0.0679 | 0.0629 | 0.0576 |
| C_G | 23.9674 | 31.1278 | 41.8241 | 55.7274 | 76.1910 |
| K | 0.9902 | 0.9995 | 1.0082 | 1.0183 | 1.0254 |
| R_{adj}^2 | 0.9903 | 0.9909 | 0.9918 | 0.9929 | 0.9939 |
| RMSE | 0.0341 | 0.0337 | 0.0337 | 0.0325 | 0.0307 |
| | Sample 3 | | | | |
| X_m | 0.0787 | 0.0726 | 0.0699 | 0.0664 | 0.0645 |
| C_G | 23.6260 | 31.2144 | 41.6545 | 56.2088 | 50.8047 |
| K | 0.9899 | 0.9994 | 1.0095 | 1.0174 | 1.0170 |
| R_{adj}^2 | 0.9903 | 0.9910 | 0.9920 | 0.9928 | 0.9913 |
| RMSE | 0.0146 | 0.0127 | 0.0113 | 0.0099 | 0.0099 |
| | Sample 4 | | | | |
| X_m | 0.0818 | 0.0736 | 0.0791 | 0.0690 | 0.0638 |
| C_G | 27.2917 | 28.7607 | 26.1043 | 53.7948 | 77.4053 |
| K | 0.9911 | 1.0002 | 0.9951 | 1.0225 | 1.0185 |
| R_{adj}^2 | 0.9926 | 0.9898 | 0.9854 | 0.9930 | 0.9933 |
| RMSE | 0.0133 | 0.0138 | 0.0160 | 0.0105 | 0.0086 |

to decrease is a consequence of the variation in the number of active sorption sites due to changes in temperature and in the chemical composition of the samples.

The graphical representation of the GAB fit is shown in Figure 1 for S4 at all temperatures and in Figure 2 for all samples at fixed temperature. It is clear that the curves took on a sigmoidal behavior, typical of type II isotherms (Brunauer et al., 1940). Type II isotherms were also obtained by Ahmat et al. (2014) when studying the desorption isotherms of fresh beef. This type of isotherm can be divided into three regions: the first corresponds to the monolayer moisture strongly attached to the product matrix; the second follows an almost linear path, which corresponds to the multilayers moisture; and the third part is related to the free water available for reaction (Mathlouthi, 2001).

It can also be seen in Figure 1 that, for a_w between 0.45 and 0.6 (the highlighted and magnified area in that figure), there is the area of greater distance between the curves. In this range of a_w , the decreases in X_{eq} due to the increase in storage temperature were greater in relation to the other a_w intervals studied. At an average relative humidity of 50%, the values of predicted equilibrium moisture content could vary up to 23% among the samples, depending on the storage temperature.

When analyzing Figure 2, the S4 isotherm features a higher X_{eq} when compared to the other samples, for all ranges of a_w studied. In particular, for sample S4, the X_{eq} in the a_w range between 0.45 and 0.6 has become even higher than in the other

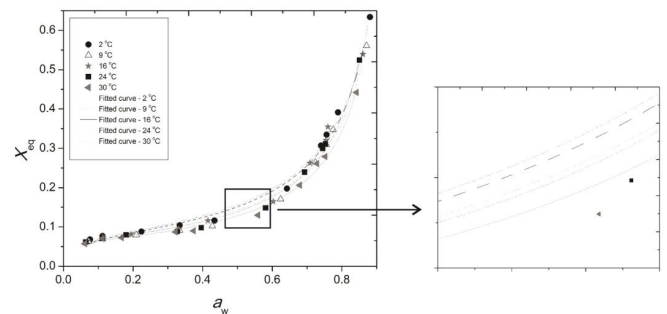


Figure 1. GAB model fitted to the sorption isotherms for Sample 4 (S4) of cooked ham at different storage temperatures.

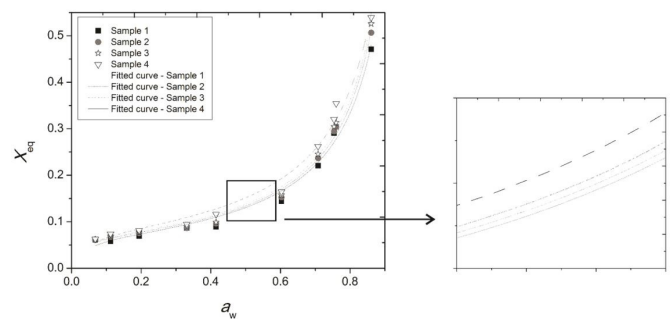


Figure 2. GAB model fitted to the sorption isotherms for Samples 1, 2, 3 and 4 (S1, S2, S3 and S4) of cooked ham at fixed temperature (16 °C).

tracks, as can be seen by a greater displacement between its curve and the curves of the other samples. This phenomenon also occurred to the isotherms obtained at other temperatures (2, 9, 24 and 30 °C). Such behavior is possibly due to differences in the chemical composition of the studied cooked ham. S4 showed higher protein content and thus a lower MPR ratio. Chou & Morr (1979) highlighted the binding properties between water and proteins. They highlighted the great capacity of water to bind to polar amino groups of protein, contributing to a significant increase in monolayer moisture. This fact is in accordance with the values of X_m encountered for the different samples. Sample S4 had higher values of X_m when compared to samples S1 and S2, which presented lower protein contents. Analogously, as the MPR ratio reduced, the higher the X_{eq} became for all ranges of a_w , studied, regardless of the product storage temperature.

Meat proteins, particularly myofibrillar protein, are excellent gelling agents. They are better able to retain water in the system and are largely responsible for the structural characteristics and texture of meat products (Robe & Xiong, 1993; Xiong, 1993). The interactions between the protein and the water are of great importance to the water retention capacity and the congelation in meat products and, consequently, affect the technological properties of the product (Puolanne & Halonen, 2010).

Schut (1976) confirmed that fat can also affect the water retention capacity in meat systems. The author claims that both fat and water are connected to the meat because they are trapped in the protein matrix. So, by reducing the fat content of the system, there must be an increase in the water retention capacity, since more protein is available to bind to water (Trout & Schmidt, 1986). This phenomenon was also seen in this study, since S4 showed an increased availability of protein due to the lower MPR ratio and lower fat content. This possibly contributed to water retention and hence kept X_{eq} higher for all a_w when compared to the other three samples at all temperatures studied. Iglesias & Chirife (1982) also reported that increasing fat content promotes a decrease in equilibrium moisture content based on the assumption that fat does not absorb water. This is in close agreement with what is shown in Figure 2. Samples S1 and S2, which contain more fat, had lower equilibrium moisture content at a specific water activity.

4 Conclusion

The GAB model fitted well to the experimental data for sorption isotherms of cooked ham in storage simulation at various temperatures. Chemical composition of cooked ham demonstrated significant influence on the water desorption isotherms. The lower the MPR ratio or the lower the fat content is, the higher its X_{eq} . Cooked ham is generally a product with a high moisture content of over 70%. Once removed from the original packaging and sliced during marketing, a low storage temperature at high relative humidity reduces water desorption. A cooked ham with low MPR ratio and low fat content can also assist in maintaining the product humidity and sensory aspects, besides increasing its shelf-life.

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