

## ORIGINAL ARTICLE

# The influence of elevated temperatures and composition on the water activity of egg powders

Marco E. Pérez-Reyes<sup>1</sup>  | Juming Tang<sup>1</sup> | Mei-Jun Zhu<sup>2</sup> | Gustavo V. Barbosa-Cánovas<sup>1</sup>

<sup>1</sup>Department of Biological Systems Engineering, Washington State University, Pullman, WA, USA

<sup>2</sup>School of Food Science, Washington State University, Pullman, WA, USA

## Correspondence

Juming Tang, Department of Biological Systems Engineering, Washington State University, P.O. Box 646120, Pullman, WA 99164-6120, USA.  
Email: jtang@wsu.edu

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## Abstract

This study aimed to investigate water activity ( $a_w$ ) changes in different egg powders (egg white, egg yolk, and whole egg) as influenced by temperature between 20°C and 80°C. A high-temperature test cell was used to measure the  $a_w$  in egg powders of different moisture contents (2 to 30% wet basis) during heating in a closed environment. Then, the net isosteric heat of sorption ( $q_{st}$ ) of the egg powders was calculated. The  $q_{st}$  values were used in the Clausius–Clapeyron equation to generate the isotherms of the egg powders at 40°C and 80°C. The results showed that at fixed moisture content and temperature, egg yolk powder had higher  $a_w$  than whole egg and egg white powders. In addition,  $a_w$  of the three egg powders in closed containers all increased with temperature. Egg white powder had stronger binding capacity to water, as reflected by the higher  $q_{st}$ , compared to the other two egg powders.

## Practical applications

An important determinant for the survival of microorganisms during thermal treatments is food water activity ( $a_w$ ), which changes with temperature. Currently, there is no scientific data regarding  $a_w$  changes in egg powders at elevated temperatures. Therefore, this study aimed to determine the  $a_w$  changes in egg powders as a function of temperature and composition. Data produced by this study regarding  $a_w$  changes provide useful information for designing effective heat treatments to control pathogens in egg powders. The isosteric data should also help estimate the energy efficiency of egg powder drying operations.

## 1 | INTRODUCTION

Low-moisture foods, such as almonds, chocolate, and peanut butter, have recently been associated with numerous *Salmonella* outbreaks (CDC, 2004; Enache et al., 2017; Werber et al., 2005). Thermal treatments are commonly used to inactivate *Salmonella* in intermediate and high moisture foods. But the effectiveness of thermal treatments decreases for food with water activity ( $a_w$ ) lower than 0.6 (Syamaladevi et al., 2016). Thus, higher temperatures (e.g., above 70°C) and long times are typically used in those treatments. An important prerequisite to the design of effective thermal treatments for pathogen control in low-moisture food is to understand how  $a_w$  of the food changes with temperature (Liu et al., 2018).

Water activity is the measure of the state of water in foods. It is defined as the ratio of the water vapor pressure of food to the saturated water vapor pressure at a given temperature (Labuza & Altunakar, 2007). The value of  $a_w$  (between 0 and 1) indicates the degree to which water is able to participate in different chemical and physical processes. These processes can potentially change the quality of a product in storage or during a thermal treatment (Cauvain & Young, 2008; Tadapaneni et al., 2017). The relationship between  $a_w$  and the moisture content (MC) of a food at constant temperature and pressure is described by moisture sorption isotherms (MSIs) (Andrade-P et al., 2011). MSIs provide useful information in the selection or design of adequate processes, storage conditions, or packages in order to extend the quality, stability, and shelf life of

food products (Al-Mahasneh et al., 2009; Bup et al., 2013). Water activity changes in food as a function of temperature, since food components either release or adsorb water molecules at high temperatures. These effects can be assessed by measuring the relative humidity (RH, %) of the headspace above a food sample in a closed environment, which eventually reflects as  $a_w$  changes (Liu et al., 2018; Syamaladevi et al., 2016).

The thermodynamic properties of a food can be analyzed after determining  $a_w$  values at different temperatures. The net isosteric heat of sorption ( $q_{st}$ ) derived from the Clausius-Clapeyron equation (CCE) indicates the binding strength between water molecules and solids in food matrices (Ayala-Aponte et al., 2011). The CCE describes the  $a_w$  of a food as a function of temperature for a given MC (Tadapaneni et al., 2017). This equation was initially developed to evaluate the impact of temperature on the  $a_w$  of a pure system (Labuza & Altunakar, 2007), but has been used to model changes of  $a_w$  in different dried food products or ingredients, including blueberry, pineapple, potato, and green algae, over a wide range of temperatures (Hossain et al., 2001; Lim et al., 1995; McMinn & Magee, 2003; Zuo et al., 2015). Recent studies show that by using the CCE, MSIs can be obtained for a larger range of temperatures and in less time than traditional methods (Jin et al., 2019; Tadapaneni et al., 2017).

Egg powders are popular ingredients for ready-for-use foods because of their desirable functionalities like foamability and stabilization of emulsions (Franke & Kießling, 2002). Thermal pasteurization is used to control pathogens in liquid whole egg and yolk before spray-drying in commercial production. Specifically, liquid whole egg and yolk are pasteurized in plate heat exchangers (e.g., at 60°C for 3.5 min or 64°C for 2 min) prior to spray-drying (Lechevalier et al., 2013). But whole egg and yolk powders can still be contaminated by *Salmonella* during packaging. Egg white, on the other hand, is pasteurized after spray-drying. The pasteurization for egg white powder is achieved by a dry-heating operation that consists of heating packaged powder in a room at 58°C to 60°C for 10 to 14 days (Baron et al., 2003; Boreddy et al., 2014). The dry-heating pasteurization of egg white powder is a lengthy treatment that may need to be reduced to save energy and increase throughputs of industrial operations (Boreddy et al., 2014). Improvements to the thermal treatment processes for egg powders can enhance the microbial safety of food products that include them as ingredients.

Our previous studies have shown that changes in the  $a_w$  of food products during thermal treatments critically influence the thermoresistance of *Salmonella* in low-moisture foods (Liu et al., 2018; Santillana-Farakos et al., 2013; Xu et al., 2019). Therefore, it is desirable to study  $a_w$  changes of egg powders at high temperatures in order to provide data necessary for improving the design of thermal treatments post-spray-drying. The objectives of this study were to obtain MSIs of egg powders between 20°C and 80°C, analyze the influence of egg powders composition on MSIs, and develop a descriptive model based on CCE to obtain MSIs at elevated temperatures.

**TABLE 1** Proximate composition of egg powder samples

|                                     | EWP        | EYP        | WEP        |
|-------------------------------------|------------|------------|------------|
| Moisture (% wt/wt)                  | 6.9 ± 0.3  | 3.7 ± 0.3  | 4.3 ± 0.1  |
| Ash (% wt/wt)                       | 5.3 ± 0.1  | 2.0 ± 0.2  | 5.2 ± 0.1  |
| Fat (% wt/wt)                       | 0.6 ± 0.0  | 55.1 ± 0.3 | 43.2 ± 0.1 |
| Protein (% wt/wt)                   | 84.3 ± 0.7 | 34.7 ± 0.7 | 46.4 ± 0.1 |
| Carbohydrate (% wt/wt) <sup>a</sup> | 2.9 ± 0.8  | 4.5 ± 0.5  | 0.9 ± 0.2  |

Note: Mean ± SD,  $n = 3$ .

Abbreviations: EWP, egg white powder; EYP, egg yolk powder; WEP, whole egg powder.

<sup>a</sup>The carbohydrate content is calculated by difference.

## 2 | MATERIALS AND METHODS

### 2.1 | Sample materials

Egg white powder (EWP) (Hoosier Hill Farm LLC, USA), egg yolk powder (EYP) (Magic Flavors, USA), and whole egg powder (WEP) (Hoosier Hill Farm LLC, USA) were used in this study. The proximate compositions of three egg powders were determined in triplicate following standard analytical methods. The values are shown in Table 1 (AOAC, 2012). According to these values, EWP is a high protein content product, with approximately 84.3% wt/wt protein. The EYP is rich in fats, having 55.1% (wt/wt) fat content. The WEP samples represent a product with intermediate fat and protein contents (43.2% wt/wt fat and 46.4% wt/wt protein).

### 2.2 | Determination of moisture sorption isotherms

#### 2.2.1 | Vapor sorption analyzer

An AquaLab Vapor Sorption Analyzer (VSA) (Meter Group, Pullman, WA) was used to generate the MSIs of EWP, EYP, and WEP at 20°C, 40°C, and 60°C. VSA utilized the dynamic vapor sorption (DVS) method, as presented by Yu et al. (2008). During the measurements, egg powder samples were exposed to a selected RH until a constant sample mass was achieved. That is, the sample reached equilibrium when its moisture content (MC) became stable in a given RH environment. Both  $a_w$  ( $=RH/100$ ) and MCs were recorded; then, the level of RH was increased to obtain another measurement. An increase of 10% in RH was selected to produce  $a_w$  intervals of 0.1 for the MSIs. The values of the MC at each  $a_w$  were calculated using the weight change data (Syamaladevi et al., 2016). The method yields highly repeatable results. Thus, the tests were conducted in two replicates.

#### High-Temperature Cell (HTC)

There had been a lack of commercial devices for measuring  $a_w$  values at temperatures above 60°C. For this reason, researchers at Washington State University have developed the High-Temperature

Cell (HTC) which allows  $a_w$  measurements at temperatures up to over 90°C. HTCs were used in this study to obtain the MSIs of egg powders at 80°C. Images of a HTC are shown in Figure 1. An HTC is basically an anodized aluminum alloy cell with a high temperature RH sensor (Honeywell Humidicon, Morristown, NJ) (Tadapaneni et al., 2017). At equilibrium, the  $a_w$  of the egg powder sample in the sealed HTC was equivalent to the RH of the air headspace above the food sample. The following equation describes the relationship between RH and  $a_w$  under constant temperature and pressure (Roos, 2007):

$$a_w * 100 = RH(\%) \quad (1)$$

Thus, the RH sensor measures the  $a_w$  of the sample at equilibrium (Cauvain & Young, 2008) after heating to a set temperature.

Before generating MSIs at 80°C, the egg powder samples were dried with 10 kPa pressure inside a vacuum oven at 50°C for 24 hr. The water content of the samples was adjusted by storing at room temperature for 5–7 days in airtight containers with saturated salt solutions. Specifically, thin-layer egg powder samples (5g) were exposed to 11.3%, 22.5%, 32.8%, 43.2%, 52.9%, 65.8%, 75%, and 86% RH at 23°C using supersaturated solutions of lithium chloride (LiCl), potassium acetate (CH<sub>3</sub>COOK), magnesium chloride (MgCl<sub>2</sub>), potassium carbonate (K<sub>2</sub>CO<sub>3</sub>), magnesium nitrate (MgNO<sub>3</sub>), sodium nitrite (NaNO<sub>2</sub>), sodium chloride (NaCl), and potassium chloride (KCl), respectively (Fisher Scientific, Houston, TX) (Greenspan, 1977). The weights of the samples were checked daily until they remained stable. Because egg powders have porous structures and are highly hygroscopic, the samples reached equilibration within 5 days. This was much faster compared with other low-moisture samples which might

need 2 weeks (Tadapaneni et al., 2017). The  $a_w$  level after equilibration was verified in triplicate using a water activity meter (AquaLab 3TE series, Meter Group, Pullman, WA) at room temperature (standard deviation of  $\pm 0.005$ ) (Syamaladevi et al., 2016).

After equilibration, the MSIs were determined at 80°C using the HTC. To reach 80°C and an equilibrium state, the HTC was sealed and heated in an oil bath. When the temperature and RH of air in the headspace did not change for 30 min, the  $a_w$  value was recorded (Tadapaneni et al., 2017). After each  $a_w$  measurement, the HTC was removed from the oil bath and cooled to room temperature. Subsequently, the MC of the samples was determined using a vacuum oven (Yamato Scientific America Inc., CA, USA) with 10 kPa pressure at 80°C for 10 hr (Syamaladevi et al., 2016).

The data of  $a_w$  at each MC and temperature from repeated measures were subjected to the analysis of variance (ANOVA) using the General Linear Model (GLM) in Minitab 17 (Minitab Inc., USA). A Tukey pairwise comparison was used to find significant differences between  $a_w$  values at different temperatures of each powder. A value of  $p < .05$  was selected as statistically significant.

## 2.2.2 | MSIs modeling with GAB equation

The Guggenheim, Anderson, and De Boer (GAB) equation is one of the mostly used equations to predict  $a_w$  over a wide range of moisture content. Guggenheim, Anderson, and De Boer independently derived the equation in 1966, 1946, and 1953 as an improved version of the BET model (Andrade-P et al., 2011). The GAB equation has shown to accurately express experimental data for  $a_w$  ranging from 0 to 0.95, it has been recognized as the best equation for modeling MSIs by the International Symposium on the Properties of Water [ISOPOW 3 Beaune (France), 1983] (Labuza & Altunakar, 2007). The GAB equation is described as:

$$\frac{MC}{X_m} = \frac{cKa_w}{(1 - Ka_w)(1 - Ka_w + cKa_w)} \quad (2)$$

where MC is the moisture content (g water/g dry solids) of a food on dry basis,  $X_m$  is the monolayer value (g water/g dry solids),  $c$  is the strength of binding water to the primary binding sites, while  $K$  is a correction factor (Blahovec & Yanniotis, 2008).

Nonlinear optimization by the Excel Solver software program (Microsoft Corporation, Redmond, WA, USA) was used to obtain the parameters in the GAB model. To analyze the GAB equation's goodness of fit, the coefficient of determination ( $R^2$ ) and the mean relative percentage error [ $P(\%)$ ] were obtained using the following equations:

$$R^2 = 1 - \left( \frac{\sum_{i=1}^n (y_{i,obs} - y_{i,calc})^2}{\sum_{i=1}^n (y_{i,obs} - \bar{y})^2} \right) \quad (3)$$

$$P(\%) = \frac{100}{n} \sum_{i=1}^n \frac{|y_{i,obs} - y_{i,calc}|}{y_{i,obs}} \quad (4)$$

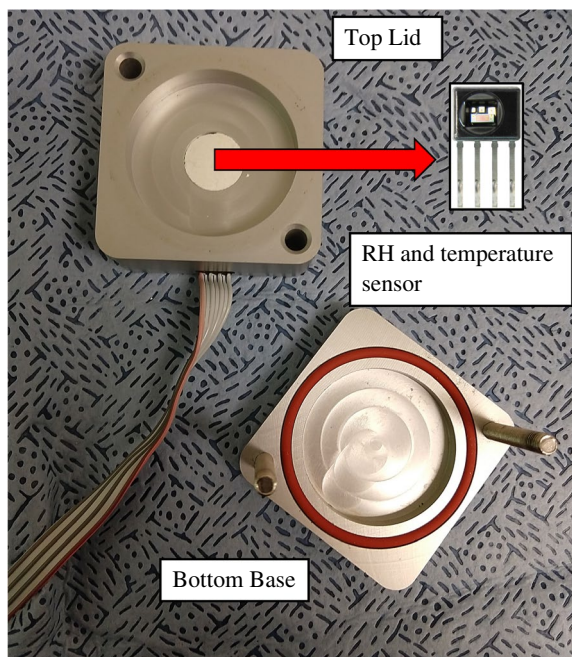


FIGURE 1 High-temperature cell (HTC) used in this study

A higher  $R^2$  and smaller  $P(\%)$  characterized the fit of the GAB model.

## 2.3 | Clausius–Clapeyron equation (CCE)

The CCE and the  $q_{st}$  are used to express the relationship between  $a_w$  and temperature for samples heated in sealed containers. The CCE can generate the MSIs for different products over a wide range of temperature in which  $q_{st}$  is assumed constant for a given MC (Labuza & Altunakar, 2007). The CCE is expressed at fixed MC (g water/g dry solids) by :

$$\ln \frac{a_{w2}}{a_{w1}} = \frac{Q_{st} - \Delta H_{vap}}{R} \left[ \frac{1}{T_1} - \frac{1}{T_2} \right] \quad (5)$$

or

$$a_{w2} = a_{w1} \exp \left[ \frac{Q_{st} - \Delta H_{vap}}{R} \left( \frac{1}{T_1} - \frac{1}{T_2} \right) \right] \quad (6)$$

where  $a_{w1}$  and  $a_{w2}$  are the water activity values of the sample at temperatures  $T_1$  and  $T_2$  (K), respectively.  $Q_{st}$  is the isosteric latent heat of sorption (J/mol),  $\Delta H_{vap}$  is the vaporization heat of pure water (J/mol), and  $R$  is the gas constant with a value of 0.008314 kJ/mol K (Tadapaneni et al., 2017).

The  $q_{st}$  (J/mol) is defined as the excess binding energy for the sorption process of water. It is expressed as the difference between  $Q_{st}$  and  $\Delta H_{vap}$ , often referred to as net isosteric heat of sorption in the literature. In relation to Equation (5), the value of  $q_{st}$  for a fixed MC can be estimated by the following expression (Moreira et al., 2012):

$$q_{st} = Q_{st} - \Delta H_{vap} = -R \left[ \frac{\partial \ln a_w}{\partial \frac{1}{T}} \right]_{MC} \quad (7)$$

That is the  $q_{st}$  is determined by plotting  $\ln(a_w)$  versus  $1/T$  (McMinn & Magee, 2003).

The egg powder samples were conditioned at 23°C to different RH following the previous procedure for the purpose of obtaining the  $q_{st}$  values. The  $a_w$  values of the samples were determined with the HTC immersed in an oil bath at a temperature between 20°C and 80°C. The  $a_w$  value was monitored at 10°C increments. Measurements were recorded at each temperature for a period of 30 min until changes were no longer observed in the %RH value. After each treatment, sample MC (g water/g dry solids) was determined using a vacuum oven at a pressure of 10 kPa and 80°C for 10 hr (Tadapaneni et al., 2017).

Water activity values were plotted as  $\ln(a_w)$  versus  $1/T$ , and the  $q_{st}$  values were estimated from the slope of the plot. These  $q_{st}$  values were expressed as a function of the MC with the following expression:

$$q_{st} = a \exp(-bMC) \quad (8)$$

where  $a$  and  $b$  are constants.  $R^2$  and  $P(\%)$  were used to evaluate Equation (8) for goodness of fit (Zuo et al., 2015).

Using Equations (6) and (8), the  $a_w$  values of the samples were calculated at different temperatures, for a fixed MC. These experimental data were used to generate MSIs of the egg powders at 40°C and 80°C. The predicted MSI from CCE was compared with the MSI of the VSA and the HTC (Tadapaneni et al., 2017). The CCE goodness of fit as compared with the experimental  $a_w$  data attained by the HTC was analyzed with the coefficient of determination ( $R^2$ ) (Equation 3), mean relative percentage error [ $P(\%)$ ] (Equation 4), and standard error of prediction (SEP) (Equation 9):

$$SEP = \sqrt{\left\{ \frac{\sum_{i=1}^n (y_{i,obs} - y_{i,pre})^2}{n} \right\}} \quad (9)$$

Microsoft Excel (Redmond, WA) was used to calculate  $R^2$ ,  $P(\%)$ , and SEP.

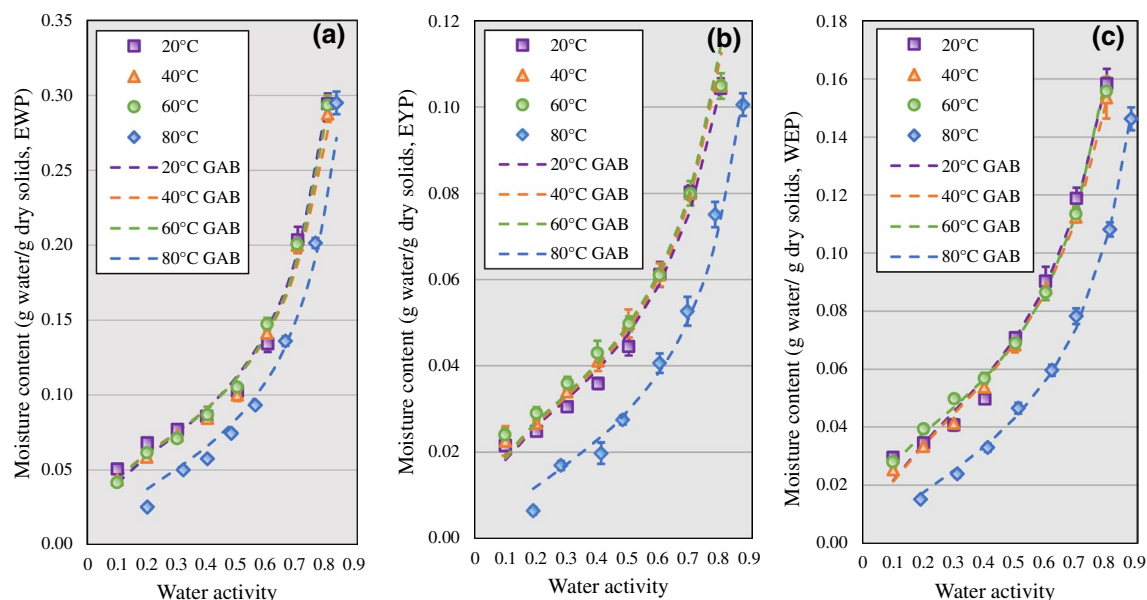
## 3 | RESULTS AND DISCUSSION

### 3.1 | Moisture sorption isotherms of egg powders at different temperatures

The MSIs of each egg powder at different temperatures are shown in Figure 2. The general MSIs shape for the egg powder samples at 20°C, 40°C, and 60°C was an upward concave. This shape is associated with a Type II isotherm, which is the most common for processed foods (Andrade-P et al., 2011). The causes for this shape include: (a) the existence of multilayers at the internal surface of a material, (b) the filling of small pores in the low  $a_w$  zone with subsequent swelling, (c) the filling of large pores, and (d) the solute dissolution in the high  $a_w$  zone (Labuza & Altunakar, 2007). In general, proteins follow the type II behavior, because proteins have a plasticized nature that increases the availability of all polar groups as MC increases. There were two bending regions in type II isotherms: the first one was around  $a_w$  values between 0.2 and 0.4 and the other between 0.6 and 0.7, which were observed clearly in EWP at 20°C, 40°C, and 60°C, due to its high protein contents (Table 1).

The MSIs at 80°C of the three egg powders were better described by a type III behavior, which represents a solvent or plasticizer above the glass transition temperature. There is a significant difference ( $p < .05$ ) in the values of MC for the MSIs from three egg powders in the 60°C and 80°C range. The thermal denaturation above 60°C may have caused these differences. The main proteins in WEP and EWP are ovalbumin and ovotransferrin. The denaturation temperatures of these proteins are 84°C and 61°C, respectively, in water or buffer. Therefore, the structure of these two proteins is stable between 20 and 60°C. At higher temperatures, the denaturation process may generate chemical and structural changes in the ovalbumin and ovotransferrin that are reflected in the shape of the MSIs (Rao & Labuza, 2012). Temperature mainly affects the





**FIGURE 2** Adsorption isotherms of egg powders and GAB modeling at 20, 40, 60 and 80°C for (a) egg white powder (EWP), (b) egg yolk powder (EYP), and (c) whole egg powder (WEP). Moisture content data points are the average of at least two independent samples, mean  $\pm$  standard deviation (SD)

stability of noncovalent interactions in a protein. When temperature is increased, the hydrogen bonding and electrostatic interactions are weakened. As a result, ovalbumin and ovotransferrin become more flexible, resulting in more side groups becoming exposed to the surrounding solvent. When these bonds are broken, the water surrounding the protein forms new hydrogen bonds with the newly exposed groups (Ustunol, 2014).

The yolk is a complex system that contains a variety of fat particles suspended in a protein solution in fresh eggs (Li-Chan & Kim, 2008). As indicated in Table 1, EYP is a high-fat content product. Above 60°C the shape of the MSI in Figure 2 for EYP powder reflects the chemical and structural changes of fats. As an example, at 20°C, the  $a_w$  of a sample with a MC of 0.06 g water/g dry solids was 0.60. When temperature increased to 80°C, the  $a_w$  of the sample was raised to 0.72. Above 60°C, most of the yolk's fatty acids, such as palmitic acid, had a phase change induced by temperature known as melting. This phase change of yolk's fats might have been reflected in the isotherm shift at 80°C (Suzuki, 1927; Walczak et al., 2017). The low-density lipoproteins present in yolk start denaturing at 70°C, and this denaturation process was also reflected in the isotherm behavior at 80°C (Denmat et al., 1999). When lipoproteins were subjected to high temperatures, their hydrogen bonding and electrostatic interactions were weakened, making their structure more flexible. As lipoproteins' structure became more flexible, their side groups are exposed and disrupted, forming new hydrogen bonds with the surrounding water. This disruption in hydrogen bonds caused lipoproteins' hydrophilic groups to be exposed, increasing the number of free water molecules and, in consequence, increasing  $a_w$  (Syamaladevi et al., 2016; Ustunol, 2014). Also, of importance, at thermal treatment temperatures above 60°C, tocopherols and other natural antioxidants present in the yolk

powders are drastically degraded. This degradation exacerbated the denaturalization of lipoproteins and was reflected in the MSIs shape. Additionally, gelation is a phenomenon where heat induces proteins of egg powders to aggregate into filaments or densely branched clusters, which interact as "sticky" reactive molecules. Proteins' gelation temperature in fresh eggs is between 55°C and 70°C. At these temperatures, the behavior changes of the isotherms were observed (Hsien & Regenstien, 1992).

The influence of composition on MSIs at 20°C is illustrated in Figure 3 for three egg powders. At a given moisture content,  $a_w$  of EWP was lower than that of WEP which was in turn smaller than that of EYP. Similar trends were observed at the other temperatures (e.g., 40°C, 60°C, and 80°C). Proteins have a higher water holding capacity that allows for functional properties such as emulsion and foam formation (Huang & Kinsella, 1986). The larger amount of proteins in EWP resulted in more polar groups that tend to bind water molecules and depress water vapor pressure, generating a low  $a_w$ . On the contrary, EYP had a high fat content and less water binding capacity which produced higher  $a_w$  at a fixed moisture content. These properties explain the lower  $a_w$  in the EWP isotherm (for fixed MC values) compared to the isotherms for EYP or WEP (Labuza & Altunakar, 2007).

The GAB equation accurately described the egg powder MSIs at different temperatures (i.e., 20°C, 40°C, 60°C, and 80°C), as indicated by high values of  $R^2$  and small  $P(\%)$  values (Table 2). Figure 3 shows the match between GAB curves and the experimental data for 20°C. The estimated values of monolayer moisture content ( $X_m$ ) for the egg powder samples are listed in Table 2.  $X_m$  decreased slightly with temperature. EWP had the higher  $X_m$  values (0.06 to 0.05 g water/g dry solids) for EWP, again because of its high protein content. The lowest values of  $X_m$  were obtained for EYP, in a range of 0.03 to 0.02 (g water/g dry solids). Lower  $X_m$  values indicate less

hydrophilic components with the reduced adsorption active sites of the egg powders (Labuza & Altunakar, 2007).

### 3.2 | Moisture sorption isotherms by the CCE

#### 3.2.1 | $a_w$ changes measured with HTC

Figures 4–6 show the temperature dependent changes in  $a_w$  of three egg powders at eight different moisture contents. For all the tested moisture levels,  $a_w$  values of EWP increased with temperature

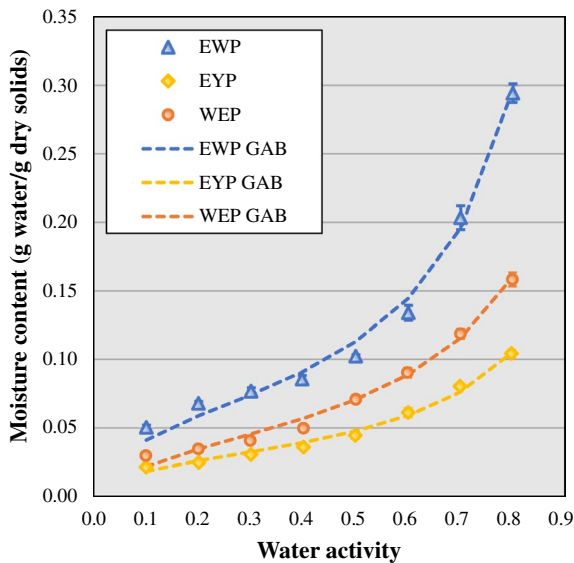
(Figure 4). For example, the EWP sample with MC of 0.084 g water/g solids had an  $a_w$  value of 0.38 at 20°C. When heated to 80°C inside the HTC, its  $a_w$  value rose to 0.52.

Similarly,  $a_w$  of EYP at different MCs also increased with increasing temperature from 20°C to 80°C (Figure 5). However, the  $a_w$  changes for the range of 20°C to 80°C were  $\approx 0.1$  for all the MCs in EYP (Figure 5). The same trend was observed for WEP samples (Figure 6). There was a slight increase in  $a_w$  with the increments of temperature. The increase in the  $a_w$  for the WEP was 0.08 at a MC of 0.152 g water/g solids. The CCE modeling provided a good fit with the experimental  $a_w$  data measured by the HTC with a high  $R^2$  ( $>0.95$ ), low  $P$ (%) ( $<10\%$ ), and SEP ( $<0.04$ ) values. CCE model values are shown in Figures 4–6 as dotted lines.

Understanding the relationship between temperature and  $a_w$  is useful because most ready-to-eat foods are subjected to different thermal treatments and storage conditions where temperature changes. The presented data suggest that temperature affects the mobility of water molecules and the dynamic equilibrium between the vapor and adsorbed phases. Water activity increases as temperature rises at a constant MC. Due to the nature of water binding, egg powders retain less water at higher temperatures than at lower temperatures (Labuza & Altunakar, 2007).

#### 3.2.2 | Net isosteric heat of sorption ( $q_{st}$ )

Figure 7 shows the dependence of the  $q_{st}$  on MC. The energy levels required for sorption were higher for lower MC. This fact demonstrates the difference in the strength of water binding capacity between the initial occupation of highly active polar sites on the surface and in the filling of the less available sites in egg powders with lower bonding activation energies. Similar behavior has been observed in tropical fruits from Asia, plantain pulp, and milk powder (McMinn & Magee, 2003).



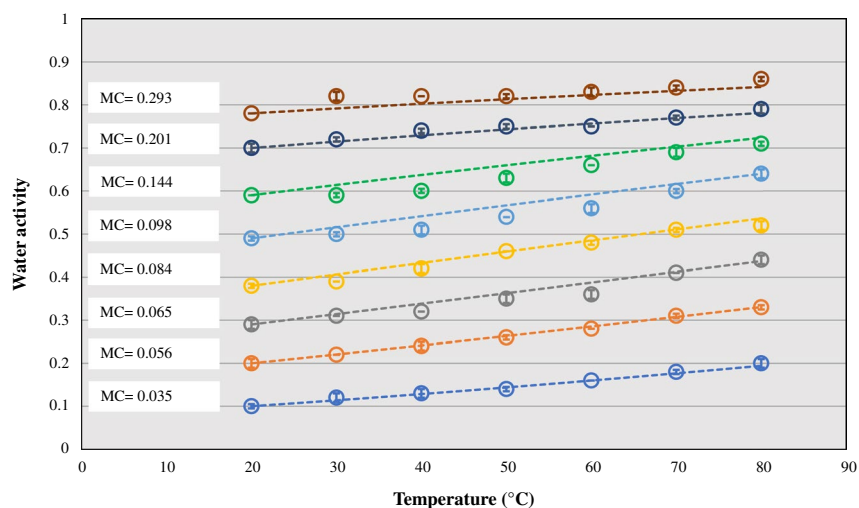
**FIGURE 3** Adsorption isotherms of three different egg powder samples at 20°C. Moisture content data points are the average of at least two independent samples, mean  $\pm$  standard deviation (SD)

| Sample | Temperature (°C) | $X_m$ (g water/g dry solids) | $K$  | $c$   | $R^2$ | $P$ (%) |
|--------|------------------|------------------------------|------|-------|-------|---------|
| EWP    | 20               | 0.06                         | 0.99 | 14.01 | 0.99  | 8.02    |
|        | 40               | 0.06                         | 0.98 | 18.22 | 0.99  | 5.21    |
|        | 60               | 0.06                         | 0.99 | 18.22 | 0.99  | 4.32    |
|        | 80               | 0.05                         | 0.99 | 5.99  | 0.99  | 10.60   |
| EYP    | 20               | 0.03                         | 0.93 | 14.85 | 0.99  | 6.43    |
|        | 40               | 0.03                         | 0.95 | 14.86 | 0.99  | 4.20    |
|        | 60               | 0.03                         | 0.95 | 14.98 | 0.99  | 6.61    |
|        | 80               | 0.02                         | 0.93 | 4.02  | 0.99  | 10.68   |
| WEP    | 20               | 0.04                         | 0.93 | 8.11  | 0.99  | 7.41    |
|        | 40               | 0.04                         | 0.91 | 8.11  | 0.98  | 4.07    |
|        | 60               | 0.04                         | 0.96 | 17.12 | 0.99  | 2.19    |
|        | 80               | 0.03                         | 0.92 | 4.01  | 0.99  | 4.52    |

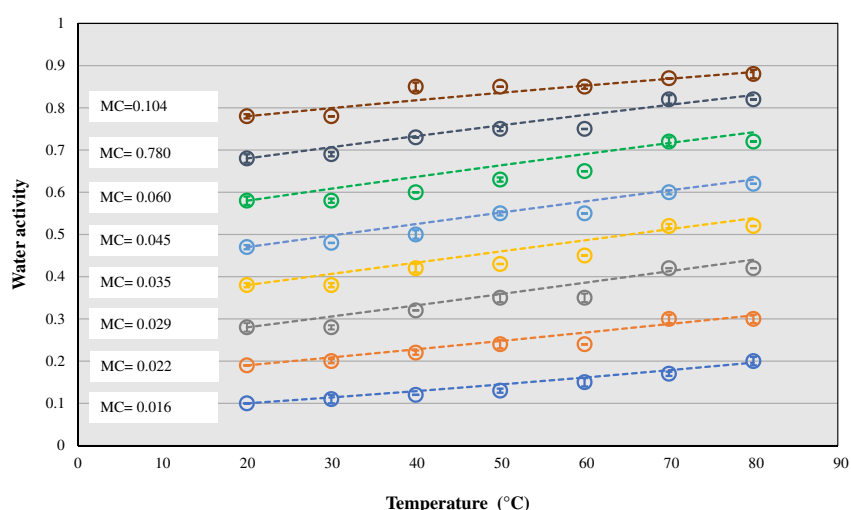
**TABLE 2** GAB model parameters for adsorption isotherms of egg powders at different temperatures

Abbreviations:  $c$ , constant representing the strength of binding water to the primary binding sites; EWP, egg white powder; EYP, egg yolk powder;  $K$ , a correction factor;  $P$ (%), mean relative percentage error;  $R^2$ , coefficient of determination; WEP, whole egg powder;  $X_m$ , monolayer value.

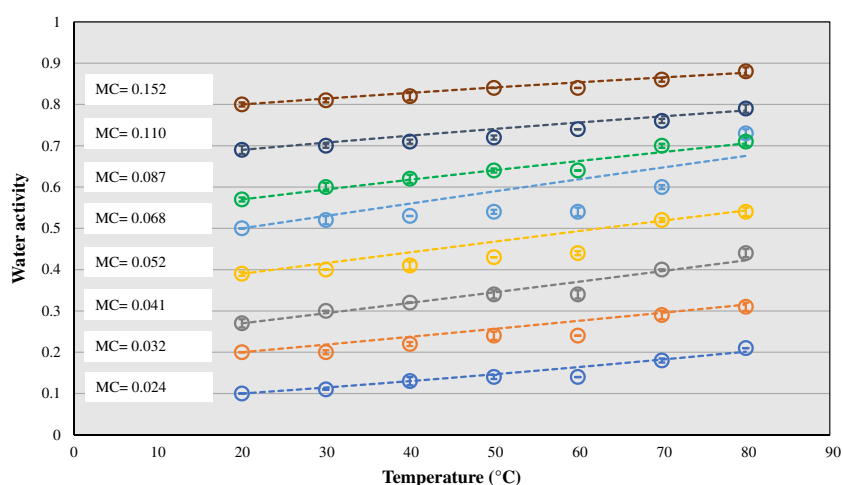
**FIGURE 4** Water activity ( $a_w$ ) of egg white powder (EWP) samples at different temperatures and different moisture contents (MC, g water/g dry solids). The predictions by Clausius-Clapeyron Equation are represented by the dashed lines whereas the data points are the means of duplicate measurements with standard deviations ( $\pm SD$ )



**FIGURE 5** Water activity ( $a_w$ ) of egg yolk powder (EYP) samples at different temperatures and different moisture contents (MC, g water/g dry solids). The predictions by Clausius-Clapeyron Equation are represented by the dashed lines whereas the data points are the means of duplicate measurements with standard deviations ( $\pm SD$ )



**FIGURE 6** Water activity ( $a_w$ ) of whole egg powder (WEP) samples at different temperatures and different moisture contents (MC, g water/g dry solids). The predictions by Clausius-Clapeyron Equation are represented by the dashed lines whereas the data points are the means of duplicate measurements with standard deviations ( $\pm SD$ )



The exponential decrease in  $q_{st}$  with increasing sample MC for each of the studied egg powder was modeled using Equation 8. The results are summarized in Table 3. The  $q_{st}$  of the egg powders behaved similarly to the MSIs. The  $q_{st}$  values for EWP were higher than that of WEP, which were in turn larger than that of EYP (Al-Muhtaseb

et al., 2002). For example, at moisture content of 0.05 g water/g solids, the  $q_{st}$  value for EYP was 4.50 kJ/mol, while the  $q_{st}$  value for EWP and WEP was 6.58 and 5.14 kJ/mol, respectively. However, at moisture content of 0.15 g water/g solids, the  $q_{st}$  values of 1.30 and 1.22 kJ/mol for EYP and WEP, respectively, were similar, while the

$q_{st}$  for EWP of 2.92 kJ/mol was the highest. This trend is attributable to the different composition of egg powders. EWP had a higher content of proteins than WEP and EYP. Because egg proteins have more affinity to water than egg fats, EWP adsorbed and/or lost more water. This behavior appeared to indicate that EWP has more surface polarity, resulting in stronger interactions between water and the EWP components. It is possible that this behavior, to a lesser extent, might have also been influenced by the different particle sizes of egg powders (Al-Muhtaseb et al., 2002; Pedro et al., 2010; Pumacahua-Ramos et al., 2016).

The values of  $q_{st}$  for the egg powders were in the range of 0 to 9 kJ/mol for MC between 0.04 and 0.3 g water/g solids, which were comparable to the  $q_{st}$  values reported for wheat, almond flour, and nonfat milk powder (Martín-Santos et al., 2012; Syamaladevi et al., 2016). The high  $q_{st}$  values at the low MCs for all egg powder samples indicate that water molecules are firmly bonded to monolayer molecules, and a high amount of energy is required to break those bonds (Syamaladevi et al., 2016).

The value of the  $q_{st}$  at a specific MC indicates the state of the adsorbed water molecules, and therefore, reflects the physical, chemical, and microbiological stability of the food material under different conditions. The calculation of  $q_{st}$  also allows a better understanding

of water-solid versus water-water interactions (McMinn & Magee, 2003).

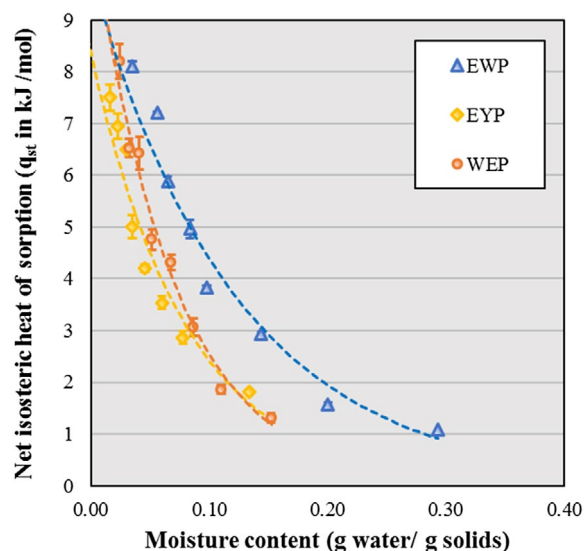
### 3.2.3 | Moisture sorption isotherms modeled with the CCE

Figure 8 shows a comparison between the MSIs of egg powders, at 40°C and 80°C modeled by the CCE, and the MSIs obtained using the HTC and the VSA for the same temperatures.

For MSIs obtained by the CCE, data showed that  $a_w$  increased with increasing temperature. The MSIs for EWP samples from CCE (Figure 8a) showed the changes in  $a_w$  when the samples were heated from 40°C to 80°C in sealed containers (i.e., fixed MC). For example, when the MC of the sample was 0.098 g water/g solids, according to CCE, the  $a_w$  of EWP was 0.54 at 40°C. When the sample was heated to 80°C, its  $a_w$  value increased to 0.63. The MSIs estimated by CCE for EYP (Figure 8b) also exhibited a rise in  $a_w$  with increased temperature. An EYP sample with a MC of 0.035 g water/g solids at 40°C had an  $a_w$  value of 0.43. At 80°C, its  $a_w$  value increased to 0.54. Note that at the higher MC of WEP samples, the  $a_w$  change estimated by CCE was minor compared to those estimated for lower MCs (Figure 8c). For example, at 40°C with a MC of 0.152 g water/g solids, the CCE indicated an  $a_w$  value of 0.83. At 80°C, the  $a_w$  value of the sample was 0.88, only 0.05 higher than at 40°C. The MSIs obtained by the VSA and HTC methods maintained a similar trend in  $a_w$  values with increasing temperature.

Other studies have reported similar trends for wheat nonfat milk powder, almond, and wheat flours, where the  $a_w$  value increased with temperature at fixed MC (Syamaladevi et al., 2016; Tadapaneni et al., 2017). This trend is caused by a decrease in the amount of water adsorbed, so the materials become less hygroscopic at higher temperatures (Langova & Stencl, 2014).

The results presented above indicate that the CCE correctly describes the relationship between  $a_w$  changes and rising temperature in egg powder samples. The MSIs modeled by the CCE are consistent with the curves obtained from the VSA and HTC methods at 40°C and 80°C. Since the HTC method, in combination with CCE, allows for the determination of the  $q_{st}$  of a sample as a function of MC over a wide range of temperature, it can be used to determine the MSIs at different temperatures with good precision. This method is faster when compared to traditional ones. Consistently, Tadapaneni et al. (2017) produced good predictive results for almond flour, organic wheat flour, and nonfat milk. However, further evaluation of



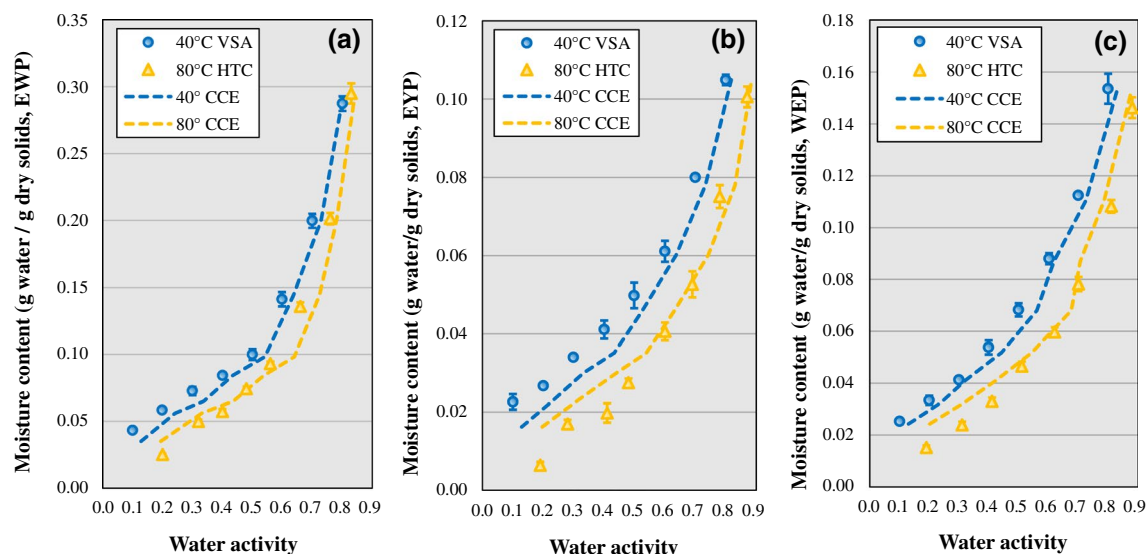
**FIGURE 7** Net isosteric heat of sorption of egg powder samples. Scattered data symbols are the average of at least two independent samples, mean  $\pm$  standard deviations ( $\pm SD$ ), the dashed lines represent the fitting equations for  $q_{st}$

| Food sample | Fitting equation                         | $R^2$ | P(%)  |
|-------------|--|-------|-------|
| EWP         | $q_{st} = 9.88 \exp(-08.12 \text{ MC})$  | 0.96  | 10.38 |
| EYP         | $q_{st} = 8.37 \exp(-12.36 \text{ MC})$  | 0.95  | 10.70 |
| WEP         | $q_{st} = 10.95 \exp(-14.58 \text{ MC})$ | 0.98  | 7.41  |

Abbreviations: MC, moisture content (g water/g dry solids); P(%), mean relative percentage error;  $q_{st}$ , net isosteric heat of sorption (kJ/mol);  $R^2$ , coefficient of determination.

**TABLE 3** The  $q_{st}$  equations obtained for egg powders with different moisture contents





**FIGURE 8** Moisture sorption isotherm of egg powders obtained from Clausius-Clapeyron equation (CCE) modeling at 40°C and 80°C (dashed lines) and experimental data at 40°C from vapor sorption analyzer (VSA) and 80°C from high-temperature cell (HTC). (a) Egg white powder (EWP) (b) Egg yolk powder (EYP) (c) Whole egg powder (WEP). Moisture content data points are the average of at least two independent samples, mean  $\pm$  standard deviation (SD)

this methodology, using other foods with a variety of compositions, is necessary to determine its suitability for broader applications.

## 4 | CONCLUSIONS

Our isotherm results show that for a given MC and at a fixed temperature, EYP had much higher  $a_w$  than WEP and EWP. GAB modeling showed a good fit for the egg powders MSIs at 20°C to 80°C. The rise of  $a_w$  values with fixed MC was corroborated using the HTC. Egg white powder had stronger binding capacity to water, as reflected by the higher isosteric heat, compared to the other two egg powders. The CCE was applied to the HTC data to generate the egg powders' MSIs at 40°C and 80°C. The obtained MSIs showed good agreement with the MSIs obtained from VSA and HTC methods. This study demonstrated the validity of using HTC plus CCE as a fast option to obtain MSIs for food products over a wide range of temperatures. The results of this study should aid future design of thermal treatments for control of *Salmonella* in egg powder products and help estimate energy efficiency of egg powder drying operations.

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## CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

## ORCID

Marco E. Pérez-Reyes  <https://orcid.org/0000-0001-6735-4493>

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