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A novel and accurate method for moisture adsorption isotherm determination of sultana raisins

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Abstract: A novel method (dynamic water transfer based water activity analyzer (DWT) method) based on Fick's law of diffusion for the accurate measurement of moisture sorption isotherm (MSI) has been developed, and was compared with saturated salt solutions (SSS) method and dynamic vapor sorption (DVS) method. MSIs at 25°C of sultana raisins obtained by the three methods were analyzed and compared, and four adsorption models (BET, Halsey, GAB, and Peleg) were used to fit the results. The MSI curves obtained by the three methods all showed the similar type-III isotherm characteristic, but equilibrium moisture content at the same relative humidity (RH) showed some differences, and the repeatability and accuracy were different. Generally, results obtained by the SSS method may have relatively low accuracy due to the relatively high measurement error; results obtained by the DVS

method may lack representativeness due to the small sample size; results obtained by the DWT method may have high representativeness and accuracy at the same time. The fitting results of adsorption models indicated that MSI results obtained by the DWT method had the highest fitting degree with Peleg model. This study may contribute to deepened understandings on MSI measurement of semi-dried foods.

Keywords: Moisture sorption isotherm; Dynamic vapor sorption; Saturated salt solution; dynamic water transfer based water activity analyzer; Sultana raisins

1. Introduction

Under the condition of constant temperature, the relationship between equilibrated water content (typically represented by the water in the mass of each unit of dry basis) and equilibrium water activity (A_w) can be described graphically in a curve, which is called the moisture sorption isotherm (MSI). MSI is of great significance to understand the following information: (1). The relation between the dehydration degree of food in the process of concentration and drying and the relative vapor pressure; (2). The determination of the moisture resistance of packaging materials; (3). The determination of what water content can inhibit microbial growth in food; (4). The prediction of the relationship between chemical and physical stability of food and water content (Labuza et al. 2006; Almuhtaseb et al. 2002; Caballero-Cerón et al. 2015; Van den Berg and Bruin 1981). The shape and values of MSI can be very different in foods, as they are related to the components' properties, physical structure,

pretreatment, and temperature (Almuhtaseb et al. 2002).

MSI rules of foods had been studied through various methods. Based on the relative humidity (RH) control means, the most commonly used MSI determination methods can be separated into two methods: saturated salt solution (SSS) method and dynamic vapor sorption (DVS) method. The SSS method is the most common technique to determine MSI of food. The samples are placed in a closed environment with its RH stabilized by saturated salt solution, and the experiment can be very time consuming (Zelinka et al. 2014). For some food samples, the mold problem easily occurs at high RH terminating the experiment accidentally, e.g., sultana raisins (Domian et al. 2018), and aseptic reagent should be added in the equilibrium environment for such case. For example, additions of toluene (Bazardeh et al. 2014) or thymol (Saravacos et al. 1986; Tsami et al. 1990) in the desiccators could inhibit mold growth. However, these chemicals are harmful to experimenter and environment.

The MSI results obtained by the SSS method can have inevitable fluctuations in different studies, due to the different equilibrium judging criterion as well as the high random error of the method itself. The moisture adsorption equilibrium in the SSS method is considered to be reached when the weights of samples reach a constant value, which is usually judged by that the weight difference from two successive measurements in a defined time interval is equal (Saravacos et al. 1986) or less than a value. The judging criteria can be very different in various reports. For example, less

1 68 than 0.3% weight change for *Agaricus bisporus* (Wang et al. 2013), less than 1 mg/g
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3 69 dry solid for edible starch based films (Bertuzzi et al. 2007), less than 0.001 g for raw
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5 70 and pregelatinized rice flour (Purohit et al. 2016), less than 0.0001 g/day (Tsami et al.
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9 71 1990) or less than 0.001 g between two consecutive measurements (Bazardeh et al.
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11 72 2014) for sultana raisins. Moreover, in the process of determining equilibrium
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13 73 moisture content (EMC), the sample needs to be taken out of desiccators repeatedly
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15 74 for weighing, increasing random error of the weighing results as the sample moisture
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17 75 content will be influenced by environmental temperature and RH. For example,
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20 76 Bradley and Vanderwarn (2001) found a 0.01% decrease of moisture content of the
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23 77 cheese placed on the analytical balance at 50% RH for 5 s or at 70% for 10 s.
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26 78 Additionally, when the samples were taken out for weighing, the equilibrium status of
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29 79 desiccators in SSS method may be interrupted as the external air easily went into the
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32 80 desiccators causing RH fluctuations, extending experiment time between two
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35 81 consecutive weight measurements.
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42 83 Controlling RH by humid flow gas, DVS method is another widely used method for
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45 84 MSI determination on food materials (Atungulu et al. 2018; Xiao et al. 2015; Wang et
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48 85 al. 2014). Due to the small sample capacity (typically 1-30 mg, maximum weight is 5
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51 86 g), it works excellently for samples with small quantity/weight, and thus moisture
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54 87 sorption equilibrium time of DVS method is relatively short. The balance usually has
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57 88 much higher precision than that of SSS measurement, and the equilibrium criterion
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60 89 has to be set manually. Generally, the criterion for moisture adsorption equilibrium of
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1 90 materials **is** a change in mass of 0.002 % per minute over a 10 min period (Besbes et
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3 91 al. 2013; Yang et al. 2016; Lin et al. 2017; Jebri et al. 2018), and several other criteria
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5 92 have also been used. For example, the dm/dt criterion of the manual measurements
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8 93 **was** 0.01 %/min (Sheokand et al. 2016), 0.005 %/min (Castro et al. 2016; Bingol et al.
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10 94 2012) and 0.00002 %/min (Chen et al. 2017).
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18 96 However, for samples with large mass/volume, sample pretreatment may be required
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20 97 (e.g., sampling after grinding) to avoid exceeding the sample capacity (Hill et al.
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22 98 2009). Therefore the intactness of the sample is destroyed, and the real MSI results
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24 99 may not be obtained. On the other hand, for heterogeneous samples, the MSI results
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26 100 obtained with DVS may be too particular due to the small sample size, which does not
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28 101 have enough representativeness (Horwitz 1988). **In such cases, the** DVS method is
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30 102 limited.
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39 104 Dried fruits are produced by reducing the water or other liquid content of the fresh
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41 105 fruits to an acceptable low **content** (Ramos et al. 2003). In this drying system, the A_w
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43 106 of the product must be controlled to avoid moisture migration (Maltini et al. 2003).
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45 107 Besides, sugaring of dried fruit usually occurs during long-term storage with warm
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47 108 and humid storage environment (Bazardeh et al. 2014), leading to changes **in the** MSI
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49 109 of dried-fruit. Due to complexed compositions of dried-fruit (Ghraiiri et al. 2013;
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51 110 Karakaya et al. 2001; Bhourri et al. 2016), MSI measurement of dried fruit has two
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55 111 common problems when using SSS and DVS methods: (1) the moisture equilibration
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status at low A_w is not easy to be judged, as dried fruit contains some components with certain water combining sugars, plant fibers, and tissue cells, affecting moisture desorption; (2) insufficient equilibration may appear as some dried-fruit have semipermeable skin seriously decreasing the moisture exchange between dried fruit flesh and the environment (Fouskaki et al. 2003).

Therefore this work aimed to present a novel method for MSI determination of dried fruit with large sample size (or volume) and to evaluate the accuracy of the method with the other two methods (SSS and DVS). This novel method was based on the diffusion theory of different concentration of moisture, and an instrument named dynamic water transfer based water activity analyzer (DWT) was made for this method. The machine could allow large sample capacity (maximum weight 225 g) and could do multiple independent parallel tests at the same time without taking samples outside the test chamber, to reduce the random error and reach higher efficiency. As one kind of traditional dried fruit, sultana raisin has semipermeable skin impacting moisture adsorption/desorption and relatively large sample weight compared with sample capacity of DVS. Therefore the accuracy differences on MSI curves of sultana raisin by DWT method, DVS method, and SSS method were analyzed and discussed. This study may contribute to the deepened understandings of MSI rule of dried fruits.

2. Materials and methods

2.1. Materials

Commercial sultana raisins were obtained from the Jiexi county Xing Sheng Food Co., Ltd. (Guangdong, China). The water content of sultana raisin was 0.102 ± 0.001 water/g dry basis determined by the vacuum oven (DZF-6020, China) method according to AOAC method No. 934.06 (1990) (0.01 MPa absolute pressure, 70 °C for 6 h). The water activity of sultana raisin was 0.660 ± 0.001 measured by the portable, fast water activity meter (Hygro Palm, Rotronic ag, Switzerland).

2.2. The moisture adsorption isotherm of sultana raisins obtained by SSS method

Nine saturated salt solutions (LiCl, $\text{KC}_2\text{H}_3\text{O}_2$, MgCl_2 , K_2CO_3 , Na Br, KI, NaCl, KCl, KNO_3) were prepared, with corresponding A_w of 0.113, 0.231, 0.328, 0.432, 0.576, 0.689, 0.753, 0.843, and 0.936, respectively. The saturated salt solutions (500 mL) were poured into the bottom of the nine desiccators (4 L volume), and sultana raisins (Ca. 4 g; around **nine** raisins) were put on porous platforms above the salt solutions. Then the desiccators were sealed with hermetic lids and stored at 25 °C for 30 days (Bazardeh et al. 2014). Subsequently, the moisture adsorption equilibrium of sultana raisins in the desiccator was considered to be reached when the variation of the weights was less than 0.1% of **the** initial weight of sultana raisins in every two days. In order to inhibit the mold growth on samples, **a 10-mL beaker containing toluene was placed inside the sealed desiccators along with the samples** with $\text{RH} > 70\%$. Three independent measurements were carried out for each A_w .

2.3. The moisture adsorption isotherm of sultana raisins obtained by DVS method

The moisture sorption curve of sultana raisin was determined using a DVS Intrinsic apparatus (Surface Measurement Systems, London, UK). Experimental data points were analyzed using Microsoft Excel 2010 and DVS Standard Analysis Suite Version 6.3. An intact sultana raisin (Ca. 100 mg) rather than broken pieces were loaded in the sample pan and was exposed to RH of 10, 20, 30, 40, 50, 60, 70, 80, 86%. The equilibrium criterion was that the weight change of the sample did not exceed 0.001g in every one hour. All measurements were performed at 25 °C and done in duplicate.

2.4. The moisture adsorption isotherm of sultana raisins obtained by DWT method

2.4.1. The test principle of DWT apparatus

The moisture sorption curve of sultana raisin was determined using a DWT apparatus (SM-M-4-01, Wuhan Ruizhi Technology Co., Ltd., Hubei, China) with authorized patents (ZL201320788053.5 and ZL201710703278.9, China). The principle of measuring moisture sorption curve of the machine is to monitor sample weight changes online under storage at stable temperature and humidity. The humidity gradient field in the testing chamber is controlled by mixing dynamic dry and wet gas. The sample in the testing chamber reaches the moisture sorption equilibrium according to Fick's law of diffusion, and the moisture sorption/desorption is calculated by the weight change of samples (Fig. 1).

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179 **2.4.2. The structure characteristics of DWT apparatus**

180 This apparatus is mainly composed of four parts, *i.e.*, testing chamber, weighing
181 modulus, N₂/H₂O mixing system, and electronic control systems. The temperature and
182 humidity of the flow N₂/H₂O mixing gas are accurately controlled by the temperature
183 control and automatic humidity generating device, before the gas flowing into the
184 testing chamber (Cylinder, diameter = 350 mm, high = 60 mm, volume = 5.8 L). The
185 N₂ flow rate can be set in the range of 0-3 L/min depending on test requirements on
186 temperature accuracy and stability in the testing chamber. The weighing modulus
187 contains four independent weighing pans, where four independent experiments can be
188 started simultaneously. The maximum weight for each weighing pans is 225 g at
189 weight accuracy of 0.001 g.

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191 **2.4.3. The operation procedure of MSI of sultana raisins testing by DWT** 192 **apparatus**

193 The DWT apparatus was mainly operated as follow (Fig.2): Step 1, turn on the
194 instrument. Step 2, turn on the system and set the instrument temperature and RH.
195 Step 3, run the nitrogen valve to ensure the pressure of pressure reducing valve was
196 0.1 MPa. Step 4, Open the system software and store data. Step 5, Open the test
197 chamber lip and place the designed tray on the sample pan. Step 6, Close the test
198 chamber and balance peeling. When the preset temperature and RH value is reached,
199 go to step 7. Step 7, Open the test chamber cover, put the sample on the designed tray,

and then close the test chamber cover in time. The temperature, RH, and sample weight data were recorded by automatic and real-time recording.

Sultana raisins (approximately 28 g; around 65 raisins) were put on each self-made porous tray in the testing chamber. Sultana raisins were exposed to 10, 20, 30, 40, 50, 60, 70, 80, 90 % RH at 25°C, respectively. For each RH, if the weight difference in 1 h was less than 0.001 g, the moisture adsorption was considered to reach equilibrium.

2.5. Data analyzing method and model evaluation

Four mathematical models (Table 1), including the BET, Halsey, GAB, and Peleg, were selected to fit the experimental data of the MSI of sultana raisins at 0-90 % RH range.

A non-linear least squares fitting (NLSF) method was performed to compute the parameters of the models listed in Table 1, using OriginPro 8.0 software (OriginLab Corporation, USA). The coefficient of determination (R^2) and mean relative deviation modulus (E) were used to evaluate the goodness of fit of each model. The E value was calculated by Eq. (1).

$$E = \frac{100}{N} \sum_{i=1}^N \left| \frac{M_{ei} - M_{pi}}{M_{ei}} \right| \% \quad (1)$$

Where M_{ei} and M_{pi} are the experimental and predicted EMC, respectively. N is the number of experimental data.

3. Results and discussion

3.1. Moisture adsorption isotherm of sultana raisin determined by SSS

The triplicate EMC results of sultana raisin were almost completely overlapped (Fig. 3 A), indicating the good repeatability of the methods. This may also support that the impact of individual differences of sultana raisin on the MSI results **was** minimized by this relatively large sampling size. The EMC of sultana raisin increased with increasing RH, and the values were slowly increased from 0 to 15 % in the range of 10-50 % RH, followed by a sharp rise from 15% to 78.14 % in the range of 50-90 % RH (Fig. 3 B). This typical J-shaped MSI curve was similar to prior reports of MSI of sultana raisins also determined by SSS method (Bazardeh et al. 2014; Saravacos et al. 1986), in which EMC grew slowly under low RH and rose rapidly under high RH, and the differences **in** EMC value at each RH may be due to different types of raisins.

3.2. Moisture adsorption isotherm of sultana raisins determined by DVS

Moisture adsorption equilibrium time of sultana raisins ranged from 32 h to 170 h (Fig. 4), and higher RH resulted in longer equilibrium time. Initial moisture contents of sultana raisins were about 10% (dry basis). EMC **was** basically unchanged in the range of 10-40 % RH, but then sharply increased from 10 % to 75 % in the range of 50-90 % RH (Fig. 5 A&B).

At low RH <50%, the EMC values were almost unchanged, unlike that obtained by SSS method, and this may be explained by that DVS method can reach higher accuracy as the weighing process does not need to take samples out of the chamber,

reducing the random error. However, the main problem for DVS method is that the small sample size, as the recommended maximum sample weight is preferably not to be over 100 mg. Therefore in this experiment, only intact sultana raisin with initial weight (≤ 100 mg) was used, and this meant that the obtained results may more reflect the MSI results of individual samples, and lacked representativeness. This was supported as the moisture content change against time (Fig.4), and MSI curves (Fig. 5A&B) of two randomly selected sultana raisins showed some differences especially at RH >50%, and thus the MSI results obtained by one or few intact raisins may have high systematic error, and did not reflect the typical characteristics of raisins.

3.3. Moisture adsorption isotherm of sultana raisins determined by DWT

Moisture equilibrium time of sultana raisins over an RH range of 10-90% ranged from 28 to 450 h (Fig. 6), which was significantly greater than that of the DVS method. This may be due to the larger sample size. Similar to the result obtained by SSS method, initial moisture contents of sultana raisins were about 10% (dry basis), and EMC was basically unchanged in the range of 10-40 % RH, and then sharply increased from 10 to 95 % in the range of 50-90 % RH (Fig. 7 A&B). The J-curve is the hygroscopic isotherm of a typical high-glycemic food (Almuhtaseb et al. 2002; Basu et al. 2006; Medeiros et al. 2006). Based on the theory of moisture adsorption and diffusion, DWT is also suitable for food samples that conform to other types of curves (LEMUS & R, 2011; Barbosa-Cánovas et al. 2007).

Compared with SSS and DVS method, the quadruplicate MSI curves obtained by the DWT method showed significantly higher overlap ratio, as the four curves were almost the same (Fig. 7A). This may be explained by that a larger sample size (approximate 30 g) could greatly minimize the impact of the particularity of individual sample on the IMS curve, and therefore showed significantly higher repeatability and accuracy than SSS and DVS methods.

3.4. Comparison of three methods for moisture adsorption isotherm determination of sultana raisins

3.4.1 Moisture adsorption isotherm curve comparison

MSI of sultana raisins determined by SSS, DVS, and DWT methods all showed similar J-shaped curve, with an initially slow increasing trend of moisture content (0-15 %, dry basis) at 10-50 % RH, and a following sharply rise trend of moisture content (15-95%, dry basis) at 50-90 % RH. This indicated that MSI of sultana raisins belonged to Type-III isotherm. Precise control of RH determines the accuracy of the EMC values (Bui et al. 2017; Young et al. 1967). Compared with DVS and DWT methods, the samples may have moisture adsorption/desorption during the weighing process, increasing measurement error. This may result in the significant difference in EMC values at each RH when compared with that of the other two methods, especially at high RH. On the other hand, although DVS and DWT method were not impacted by the external weighing problem, their results did still show some difference, which should be explained by systematic error difference between a large

sample size (28 g; DWT) and a small sample size (100 mg; DVS). The DVS method used an intact sultana raisin for testing and did not represent a 'batch of products', while the DWT method used 'multiple samples', which was obviously better representative of the batch.

3.4.2 Theoretical fitting degree differences of SSS, DVS, and DWT methods

Experimental adsorption values of sultana raisins were fitted to four equations, and the results were listed (Tables 2). If the value of R^2 is high than 0.99, and E less than 10%, the experimental data is considered to fit excellently with the model. Among all of the selected models, Peleg equation gave the values of R^2 higher than 0.995 and E values below 5% (Table 2) and therefore should be used to describe the moisture adsorption behavior of sultana raisins. For the model fitting of Peleg method, the DWT method had the lowest E value and highest R^2 values, and this highest fitness may support the highest accuracy of the DWT method in turn.

Conclusion

The MSI determination accuracy and representativeness of sultana raisins obtained by SSS, DVS, and DWT method were analyzed and compared. The moisture equilibration time of three methods were all long due to the characteristic of sultana raisins. Generally, the MSI results obtained by the three methods all showed “J” shaped curve and belonged to Type-III isotherm, and the EMC values at different RH showed some difference. SSS method could have good repeatability, but its accuracy

may be influenced by that the high measurement error as the samples needed to be taken out from the desiccators, which also impacted the equilibration judgment criterion. The results obtained by DVS method could have high accuracy as all weight measurements were done inside the equilibration environment, but the overlap ratio of parallel test results was not as high as that of other two methods, which may be due to the lack of representativeness due to the small sample size. Therefore the results obtained by DVS may reflect more of individual MSI property rather than common MSI property of sultana raisins. The results obtained by DWT method showed high repeatability, and it could minimize measurement error like DVS method, and also have less systematic error than DVS method. Peleg model was found to be the best model to describe the MSI curves of sultana raisins, and the results obtained by DWT method showed the highest fitting degree, which may again support the high accuracy and representativeness of DWT method. This study can benefit MSI analysis on dried fruits and reflect the true hygroscopic characteristics of the sample.

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Compliance with Ethical Standards

Conflict of Interest

The authors declare no conflict of interests.

Ethical Approval

This article does not contain any studies with human participants or animals performed by any of the authors.

Informed Consent

Not applicable.

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