

Methods for measuring water activity (a_w) of foods and its applications to moisture sorption isotherm studies

Lida Zhang, Da-Wen Sun & Zhihang Zhang

To cite this article: Lida Zhang, Da-Wen Sun & Zhihang Zhang (2017) Methods for measuring water activity (a_w) of foods and its applications to moisture sorption isotherm studies, Critical Reviews in Food Science and Nutrition, 57:5, 1052-1058, DOI: [10.1080/10408398.2015.1108282](https://doi.org/10.1080/10408398.2015.1108282)

To link to this article: <https://doi.org/10.1080/10408398.2015.1108282>



Accepted author version posted online: 30 Oct 2015.
Published online: 30 Oct 2015.



[Submit your article to this journal](#)



Article views: 638



[View related articles](#)



[View Crossmark data](#)

Methods for measuring water activity (a_w) of foods and its applications to moisture sorption isotherm studies

Lida Zhang, Da-Wen Sun, and Zhihang Zhang

Food Refrigeration and Computerised Food Technology (FRCFT), School of Biosystems and Food Engineering, Agriculture and Food Science Centre, University College Dublin (UCD), National University of Ireland, Belfield, Dublin, Ireland

ABSTRACT

Moisture sorption isotherm is commonly determined by saturated salt slurry method, which has defects of long time cost, cumbersome labor, and microbial deterioration of samples. Thus, a novel method, a_w measurement (AWM) method, has been developed to overcome these drawbacks. Fundamentals and applications of this fast method have been introduced with respects to its typical operational steps, a variety of equipment set-ups and applied samples. The resultant rapidness and reliability have been evaluated by comparing with conventional methods. This review also discussed factors impairing measurement precision and accuracy, including inappropriate choice of predrying/wetting techniques and unachieved moisture uniformity in samples due to inadequate time. This analysis and corresponding suggestions can facilitate improved AWM method with more satisfying accuracy and time cost.

KEYWORDS

Isotherm; a_w measurement (AWM) method; moisture adjustment; water homogeneity; water activity

1. Introduction

Moisture sorption isotherm (MSI) is a graphic representation of the process wherein water molecules are progressively and reversibly released from all kinds of hygroscopic forces in food system caused by colligative effects, capillary effects and direct bonding (Caballero-Cerón et al., 2015). Knowledge of MSI is extremely important for modeling, designing, and optimizing food processing unit and procedure, such as drying, baking, mixing, storing, and packaging (Bazardeh and Esmaili, 2014; Noriega et al., 2014; Bispo et al., 2015; Yang et al., 2015). Considering that MSI is a curve of equilibrium moisture content (EMC) versus water activity (a_w), MSI can be determined by measuring water content when a_w is controlled, which is called gravimetric method (Suntaro et al., 2014); or conversely measuring sample's water activity when the water content is fixed, referred as a_w measurement (AWM) method by Bell and Labuza (2000). Saturated salt slurry (SSS) method, representative of gravimetric methods, has been proposed as a reference isotherm determination method (Wolf et al., 1985) and has been extensively used in MSI studies of food materials for its advantages of low price, easy handling, and acceptable accuracy (Sablani et al., 2007). However, a typical SSS method takes weeks or months for MSI depiction, which makes it unfeasible to determine MSI for sensitive and spoilable biological samples under relatively high temperatures, such as pharmaceuticals or sewage sludge (Odamtten and Kampelmacher, 1986; Sawhney et al., 1997; Abdullah et al., 2000; Vaxelaire et al., 2000; Nguyen et al., 2004; Igathinathane et al., 2005; Fieldsend, 2007; Igathinathane et al., 2008; Yu et al., 2008; Huang et al., 2009;

Sandoval et al., 2011; Kartika et al., 2012; Sharma and Joshi, 2014;). Due to its drawbacks of lengthy period and possible errors introduced by combined effects of fungi activity, oxidation reaction, and temperature and humidity variations within long residence time, much work has been devoted to the field of accelerating isotherm measurement process.

In comparison with SSS method, AWM method could expedite experiment process to as less as tens of minutes (Demarchi et al., 2013; Argyropoulos and Müller, 2014). It also allows the collection of a large amount of EMCERH data points by simply adjusting the rewetting/drying time during samples' preparation. Additionally, by changing temperature settings in hygrometers during a_w measurement, MSI at various temperatures could be depicted simultaneously within a short period (Chen and Chen, 2014). Last but not least, AWM method is more simple, convenient, and inexpensive because of no need for purchase, preparation, and storage of various salt slurries. Other than SSS method which is to attain samples of constant a_w values by equilibrating with aqueous solutions of various salts for a long time, AWM method is to prepare samples with different moisture contents through drying or wetting and then to allow them to equilibrate before a_w measurement. This alternative technology was firstly introduced for MSI measurement by Pixton and Warburton (1973), but was not popularized until recent tens of years (Chen, 2000; de Souza et al., 2013). This revival of interest in AWM method owes much to the increased accessibility of commercial hydrometers featured in speediness, convenience, and high reliability currently.

In general, a real test by AWM method requires: (I) to change the amount of water in target samples to several levels,

(II) to bring samples of various moisture content to a homogeneous state in terms of temperature and moisture, corresponding to a point on the isotherm curve, and (III) to measure a_w of samples with homogenous and known moisture content. Based on whether the step I is operated manually or automatically, AWM method could be classified into two groups, manual AWM method and instrumental AWM method. This paper reviewed the performance of both types of AWM methods on an overall basis, including fundamentals and operational process, factors impairing data accuracy and proposed ways to improve, effectiveness in expediting experiment process, as well as data comparability with SSS and other traditional MSI measurement methods.

2. Manual AWM method (operational steps and precautions)

2.1. Moisture adjustment

The main concern with AWM method is to bring tested samples to different moisture levels by mild dehydrating or wetting. Desiccant drying has been recommended for sample preparation in desorption experiment. Mulet et al. (2002) dried fresh mushrooms by silica gel for a series of increasing time periods to prepare samples of different moisture values. But the effect of silica drying on isotherm data was not further explored, for example, by testing the data's comparability with other methods. Thermal drying could accelerate experiment term greatly. But the choice of temperature should be cautious to prevent possible effect on sorption characteristics imposed by severe drying. Pixton and Warburton (1973) recommended an oven drying at a temperature not higher than 35°C when AWM method was first introduced. Resulting isotherm data was validated by comparing with a conventional humidity control method. Nguyen et al. (2004) simply exposed fresh pear slices to ambient temperature until lower moisture contents and got desorption isotherms in good agreement with data from SSS method and pressure-controlled humidifier method (PCM). Recently, thermal drying at higher temperatures has also been reported in literature. Fasina (2008) and Chen (2000) used oven drying at 50°C to measure MSI of peanut hulls and peanut pods separately. According to Fasina (2008), the EMCERH data of peanut hulls prepared by oven drying were consistent with data from conventional humidity generating method (HMG) at 45, 60, and 80% RH levels. Chen (2000) also reported a good agreement between traditional SSS method and AWM method between 10 and 90% RH. Additionally, problems with the use of higher temperature have been reported in AWM methods. Demarchi et al. (2013) adopted oven drying at 60°C in AWM method for MSI measurement of fruit pulp formulations. Although the results have a consistency with data from SSS method, Demarchi et al. (2013) found a problem that the measured a_w values of such prepared samples were always higher than $0.363a_w$ even after drying for more than two days. According to Demarchi et al. (2013), it might be attributed to the presence of a sugar crust on the outer layer of pulp formulations during drying at 60°C. Such case hardening phenomenon had also been reported by Yan et al. (2008) in a vacuum drying of bananas under 70°C. Yan et al. (2008) reported the crossovers

of adsorption isotherm curves between vacuum dried and desiccant dried bananas with smaller EMCs at low a_w range and greater EMCs at high a_w range for vacuum dried samples comparing with reference desiccant dried ones. According to Yan et al. (2008), the reason might lie in some damages to bananas' microstructure and case-hardening at the surface of bananas during vacuum drying which limited moisture uptake at low humidity levels. While gelatinized starch caused by heating as high as 70°C could bind more water after the hard case softening at higher humidity. Besides, thermal drying with vacuum assistance is also employed to accelerate moisture evaporation of samples even at relatively low temperatures. Table 1 summarizes various drying techniques adopted for preparation of desorption samples in AWM method.

Different from the diversity of sample preparation strategies for desorption sample, adsorption samples could be wetted either by spraying water directly or by exposing to humid atmosphere. In MSI measurement of peanut hull by AWM method, Fasina (2008) conditioned peanut hulls with initial moisture of 9.1% (w.b.) to a series of higher moisture contents by equilibrating in a humidistat at a controlled humidity of 90% RH for different time. And through fine spraying of predetermined amount of distilled water, Haque et al. (2006) wetted rice samples from initially 12% d.b. to desired moisture levels. However, adding water directly could cause phase transfers and generate unevenly distribution of components due to partial dissolution and solute migration (Gal, 1981). This nonuniformity of ingredients in samples might influence the accuracy of following a_w determination (Schiraldi et al., 2012). Thus, more validation work needs to be taken before the final justification of adding water used in AWM method.

Furthermore, to ensure the continuity of MSI curves depicted by AWM method, the sampling period should be changed during predrying or wetting process. de Souza et al. (2013) wetted starch samples by equilibrating above pure water in sealed desiccators at 25°C and taking out samples of wetted starch more frequently at the former stage until reaching an a_w level higher than 0.7 a_w . Because moisture transfer rates were higher at the beginning of sorption process and a small change of moisture content could affect a_w values greatly in the

Table 1. A variety of drying methods used in AWM method in food area.

Method	Species	Studies
Thermal drying		
30°C	Lemon balm leaves	Argyropoulos and Müller (2014)
40°C	Lemon peel	García-Pérez et al. (2008)
50°C	Peanut kernel and hull	Fasina (2008) Chen, C. (2000)
60°C	Carrot fruit pulp	Eim et al. (2011) Demarchi et al. (2013)
140°C	Soybean	Irigoyen and Giner (2014)
Vacuum drying 25 mbar/30°C	Lemon balm leaves	Argyropoulos and Müller (2014)
Desiccant drying (silica gel)	Fish fillet	Martins et al. (2015)
	Banana flour	Cardoso and da Silva Pena (2014)
	Pork mince	Clemente et al. (2009)
	Mushroom	Mulet et al. (2002)
	Cassava starch	de Souza et al. (2013)
Freeze drying	Lemon balm leaves	Argyropoulos and Müller (2014)

medium a_w range from 0.4 to 0.7 a_w . Similarly, desorption samples were taken out more frequently at the beginning of dehydrating by using silica gel until a point at 0.4 a_w . Such prepared samples had successfully generated MSI data evenly distributed along MSI curves graphically. Similar sampling strategies have also been adopted by Chisté et al. (2015) and Cardoso and da Silva Pena (2014).

2.2. Moisture redistribution

After samples were conditioned to various water contents, they need to be sealed separately and stored for quite a long period to complete moisture redistribution in samples. That is because moisture is usually not homogeneously distributed in samples at the end of predrying and wetting process. After a long stay time, samples can reach hygroscopic equilibrium status and are ready for a_w determination later. This operation might be time-consuming and incomplete moisture redistribution caused by insufficient waiting time could result in false a_w readings (Yu, 2007; Schmidt and Lee, 2012). The required stay time should be varied for different samples and at different moisture contents (Shands and Labuza, 2009; Argyropoulos and Müller, 2014). The length of time is also influenced by previous drying-wetting techniques, storage temperature, and volume of the sealed container. Currently, the length of stay time is usually decided upon empirical experience. And there is still not a reliable and convenient technique to check whether a homogenous status has been achieved at the end of moisture redistribution period. In MSI depiction of pear slices at the range of 0.8–1.0 a_w , Nguyen et al. (2004) sealed pear slices ($\Phi 25$ mm \times 2 mm thickness) in small containers for two days to achieve a hydrous homogeneity status after partially dried in ambient air. This storage duration had been justified by comparing with EMCERH data from SSS method, improved SSS method with fans (SSMF), and PCM. Pixton and Warburton (1973) stored grain samples at 5C for 2 weeks to achieve moisture homogeneity after moisture adjustment by either adding distilled water or drying at 35C, and got good agreement of their results with conventional SSS method. Such a long storage time would also be attributed to a low storage temperature at 5C. Haque et al. (2006) also stored rice kernels at 5C for 2 weeks after wetting by spraying certain amount of distilled water. To keep samples' physical and chemical properties during storage period, Chen, C. (2000) even incubated peanut samples at 2C after the moisture adjustment. Accordingly, they waited 6 weeks to achieve uniform moisture status in samples.

Although moisture homogenization would take a long time, the storage time could be greatly shortened by reducing sample's size to as small as granules or even powders and employing some measures like shaking or stirring. After preparing mushroom samples by silica gel drying, Mulet et al. (2002) sealed the grounded mushrooms and shook them at intervals for only two days to achieve moisture homogeneity in samples. Similarly, Fasina (2008) stored grounded peanut hulls for only 24 hours at room temperature before a_w registration. And the EMCERH data from such designed procedure were consistent with those from HGM technique at 45%, 60% and 80% RH levels. Reported combinations of time and temperature for moisture redistribution in AWM method and corresponding

influential factors (food species and a_w levels) are summarized in Table 2.

Additionally, MSI studies of roasted green wheat by Al-Mahasneh et al. (2012) are also noteworthy. They did not achieve the moisture homogeneity by storing their rewetted wheat samples in sealed containers, but directly online monitored RH changes in the head space with a hygrometric sensor installed in an insulated sample storage chamber. They decided the accomplishment of moisture homogeneity in the samples by a stable a_w reading within the probe's precision limit of 1.5% RH. Similar strategy with an equilibrium criterion based on RH constancy was also adopted by Argyropoulos and Müller (2014) and Chen (2002). However, the reliability of this tactic is unclear because no control data, from traditional SSS methods or from trials with longer storage time, are available for comparison now.

2.3. Measurement of a_w values

Another important step in AWM method is to measure a_w with good precision. Foodstuffs' a_w could be measured in two ways, either by measuring target samples' partial vapor pressure directly (manometric methods), or by analyzing the relative humidity of air immediately surrounding tested samples using hygrometers (hygrometric methods) (Al-Muhtaseb et al., 2002; Caballero-Cerón et al., 2015). For years, a variety of hygrometers have been developed, including electrolytic, capacitance, hygroscopic, dew-point hygrometers, and etc., among which electronic and dew-point hygrometers are most widely used in recent researches (Troller, 2012). Through measurement of condensation point by air cooling, dew-point hygrometers could determine surrounding air's RH values and hence the target specimen's a_w values (Underwood et al., 2012); while electrical hygrometers are based on electronic monitoring of the conductivity of a reference salt solution equilibrating with the air in the insulated sample chamber (Yamazoe and Shimizu, 1986; Li et al., 2008; Farahani et al., 2014;). However, the reference salt (e.g., LiCl) coating electrodes in the electric hygrometers could be contaminated by some organic vapor (e.g.,

Table 2. Reported storage temperature and time for different foodstuffs and a_w levels in AWM method.

Storage process ^a	a_w	Species	Studies
2 C 6 weeks	0.1–0.95	Peanut kernel and hull	Chen (2000)
3 C 4 weeks	0.1–0.9	Oolong tea	Chen and Weng (2010)
4 C 24 h	0.15–0.9	Carrot	Eim et al. (2011)
4 C 48 h	0.09–0.98	Pork mince	Clemente et al. (2009)
4 C 1 weeks	0.1–0.8	Rapeseed Sunflower Flaxseed	Lazouk et al. (2015)
5 C 2 weeks	0.85–1	Rice	Haque et al. (2006)
10 C 24 h	0.2–1	Soybean	Irigoyen and Giner (2014)
22 C 24 h	0.05–0.95	Peanut hull	Fasina (2008)
25 C 10 min	0.05–0.95	Cassava starch	de Souza et al. (2013)
25 C 48 hours	0.1–0.95	Sunflower Seeds Lemon peel Mushroom Fruit pulp	Giner and Gely (2005) García-Pérez et al. (2008) Mulet et al. (2002) Demarchi et al. (2013)

^aApart from food species and a_w range of MSI measurement, storage time could also be influenced by the size of storage chamber and prior drying/wetting techniques, both of which could be found in the original papers.

propylene glycol) from food samples during a_w measurement (Chirife, 1995; Bell and Labuza, 2000). Accordingly, a regular recalibration is important to minimize errors of readings by electrical hygrometers. A further detail of hygrometer sensors on the aspects of measuring fundamentals, precision, accuracy, stability, and applicable samples can be referred to study by Srivastava (2012).

Considering the adjustment of measuring temperatures during a_w registration and request time for thermal and hygroscopic equilibrium between samples and air surround humidity sensors, it should take enough time for obtaining reliable a_w values by hygrometers. Nguyen et al. (2004) proposed an empirical time of 2 hours as sufficient measuring time. In MSI measurement of grounded mushrooms by AWM method at four temperatures, Mulet et al. (2002) adopted Novasina TH-2 hygrometer (Switzerland, Novasina Ltd.) and took around 3 hours for a_w registration at each temperature. Fasina (2008) closely monitored the evolution of RH condition on the top of samples in an insulated measurement system. He reported that the hygroscopic equilibrium could be obtained no more than 4 hours between a specimen and its head space. In MSI determination of rosehip, apple and tomato pulp formulations at 20C and 40C, respectively, by AWM method, Demarchi et al. (2013) took only 5 minutes to measure each sample's a_w by an Aqualab 3TE a_w meter and reported a result in well accordance with conventional SSS method. Further studies are expected to clarify how long one specific sample should be insulated in measuring chambers before registration of reliable a_w data. This length of time may depend on both specific a_w instruments and sample's various hygroscopic properties.

3. Instrumental AWM method

In fact, AquaSorp Isotherm Generator (AIG) (Decagon Devices Inc., America) was invented based on AWM method. This instrument does not control humidity levels (%RH), like common MSI measuring instruments. In AIG instrument, the air with a selected flow rate (10–1000 ml/min) passes through water

(adsorption) or desiccants (desorption) and then entering into the sample chamber. RH probes are fixed inside the chamber. Samples are considered to get equilibrium after achieving a stable reading by RH sensors within its measurement accuracy of commonly 1.5%RH. Then the airflow pauses, and samples' a_w and mass are measured by a chilled mirror dew-point sensor and a magnetic force balance respectively. The schematic graph of AIG instrument is shown in Figure 1. This kind of MIS determination with AIG instrument is also called dynamic dewpoint isotherm (DDI) method (Schmidt and Lee, 2012; Iaccheri et al., 2015). Actually, the equilibrium criterion of a RH change less than 1.5% is not always reliable especially for samples with slow diffusion of water into the matrix. Schmidt and Lee (2012) adopted five materials to compare DDI method with SSS method. The results showed that the similar results of the two methods were found for corn starch, soy protein, MCC, and sucrose, while corn flakes presented lower moisture content using DDI method. The same results with corn flakes were also reported by Shands and Labuza (2009) who compared DDI method, DSV method, and SSS method. They pointed out that the grinded corn flakes with greater surface area for sorption and shorter distance for diffusion could present more comparable MSI data to traditional methods.

It is necessary to emphasize that DDI method is fundamentally different from traditional MSI measurement methods. Actually, this instrumental AWM method generates dynamic isotherms without the requirement for real hydroscopic equilibrium in samples. The generated dynamic isotherms could provide information on sudden changes of sorption properties, which could not to be obtained by equilibrium isotherms. Currently, dynamic isotherms are usually employed to figure out glass transition point, thus could be used as an indicator for crystallization, caking, collapse, deliquescence, and stickiness (Carter and Schmidt, 2012; Carter et al., 2015). Vapor Sorption Analyzer (Decagon Devices Inc., USA) has been newly developed to generate both equilibrium and dynamic isotherms. It combines AIG instrument with traditional DVS sorption analyzer,

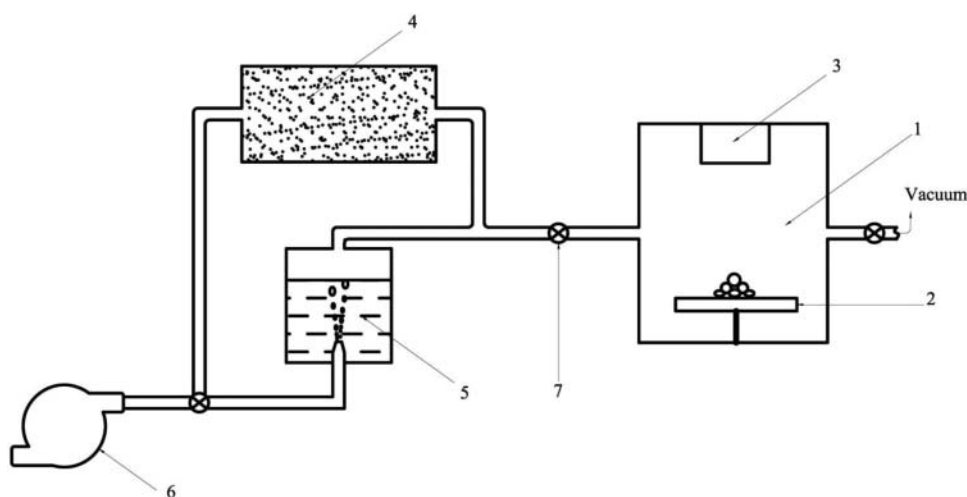


Figure 1. The schematic layout of AIG sorption analyzer. 1. Sample chamber. 2. Microbalance. 3. Dew-point RH sensor. 4. Desiccant. 5. Pure water. 6. Pump. 7. Valves.

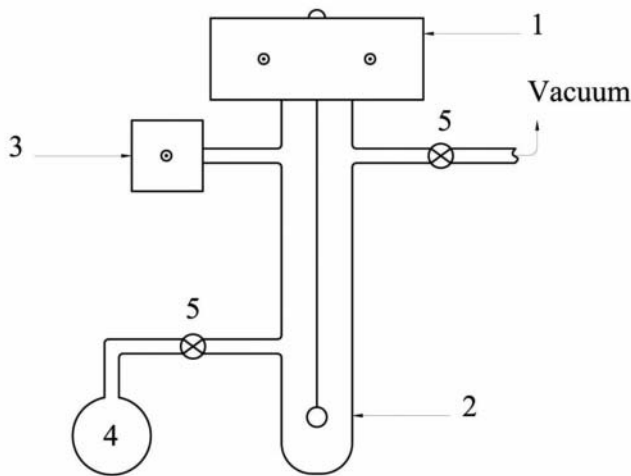


Figure 2. Schematic layout of pressure-controlled humidifier. 1. Microbalance. 2. Sample chamber. 3. Pressure gauges 4. Vapor generator. 5. Gauges.

incorporating merits of both method, and composing a more complete picture of the tested samples' moisture properties (Decagon Devices, 2015).

Jonquière et al. (1998) introduced another simple and easy device for MSI measurement based on AWM method. Different from AIG instrument, this equipment is designed for equilibrium not dynamic isotherms. The sketch of this vapor sorption instrument is shown in Figure 2. Before a real MSI test, boiling water should be placed in its vapor generator and the sorption chamber be carefully evacuated by vacuum pumping in advance. The sorption chamber is then injected a certain amount of water vapor by connecting to the vapor generator for a designed period. On hydrous equilibrium which is indicated by a stable mass of with the use of a microbalance, vapor pressure surrounding the sample was measured by a pressure gauge. And the sample's a_w could be calculated accordingly. Jonquière et al. (1998) also validated the reliability of this vapor sorption equipment by comparing with SSS method, and reported a good agreement in MSI measurement of MCC powders.

4. Assessment on accelerating effectiveness

AWM method can expedite sorption process greatly in comparison with conventional SSS and HGM methods. Before AWM method, samples are forced to be removed or added moisture by desiccants or pure water, and sealed to achieve thermal and hygroscopic equilibrium through a period of stay time. This forced moisture adjustment could reduce sorption process to days or even hours (Mulet et al., 2002; Demarchi et al., 2013;) comparing with commonly weeks or months in traditional methods. Furthermore, MSI curves at various temperatures could easily be obtained because the a_w values under different temperatures could be measured simultaneously with the help of temperature control system in a_w measurement instruments (Chen and Weng, 2010; Chen and Chen, 2014).

Before reviewing time expenses in AWM method, it should be mentioned that the period of AWM method referred in

literature is commonly computed from the time of a_w registration, excluding previous time for moisture adjustment and redistribution (Chen, 2000; Fasina, 2008). This is because there is almost none work required for researchers during sample's storage time until the start of a_w measurement. Chen and Chen (2014) adopted AWM method as a fast adsorption MSI determination method for autoclaved aerated concrete. The final experiment time for depiction of autoclaved aerated concrete's isotherm curves at seven temperatures (5, 10, 15, 20, 25, 30, and, 35C) was only four days, excluding the length of time when samples are adjusted to desired moisture levels by spraying water and storing a long time for moisture redistribution previously. For each temperature level, they insulated specimens for 12 hours in measuring chambers to ensure the arrival of thermo and hydro equilibrium in samples before the registration of final a_w data. Likewise, Mulet et al. (2002) allocated 3 hours for specimens' a_w measurement at each temperature level and completed MSI depiction of mushrooms under different temperature levels (5, 15, 25, and 35C) in days. Theoretically, for materials with higher thermal conductivity and apparent diffusion coefficient, the length of a_w registration should be reduced for samples' quick vapor and heat transfer with surrounding air in measuring chambers. In determination of desorption MSIs of rosehip, apple and tomato pulp formulations, Demarchi et al. (2013) took only 5 min to measure a_w for individual specimen. Comparing with traditional SSS method which took 21 days for MSI determination at 20C and 18 days at 40C, AWM method could expedite isotherm measurement to as less as tens of minutes (exclusive of time for adjustment and redistribution of moisture in samples).

5. Assessment on reliability

According to Bell and Labuza (2000), AWM method is acceptable as long as a_w meter is sensitive and indicative to the whole sample's a_w . Nguyen et al. (2004) adopted four different methods in MSI studies of pear at higher a_w range (0.8–1.0 a_w), including AMM method, SSS method, improved salt slurry method with fans (SSMF), and PCM. The results showed that AMM presented quite similar results with other methods. de Souza et al. (2013) compared AWM method and SSS method for both adsorption and desorption MSI determination of cassava starch at full a_w range (0.04–0.96 a_w), and reported no systematic difference between them. To assess data's reliability numerically and qualitatively, de Souza et al. (2013) fitted Henderson equation to MSI data from both methods. Both the slope and intercept parameters were compared statistically by T-test. Final results showed that AWM method and SSS method were similar at a 95% significance level. Chen (2000) also found that AWM method was not significantly different from the SSS method, by adopting a F-test analysis at 95% confidential level, in MSI measurement of peanut kernel and hull. The same result was also reported by Fasina (2008) and Demarchi et al. (2013), wherein AWM method was found to have a good agreement with SSS method and HGM method, respectively.

However, it is important to recognize and ensure the arrival of moisture homogeneity in samples before the registration of a_w values. Otherwise, incomplete moisture

redistribution, usually caused by insufficient waiting time, could lead to false a_w readings and thus error MSI data. Yu (2007) conducted MSI studies on corn starch, soy protein, MCC, sucrose, and corn flakes, and found a great difference between the results of AWM method and other methods, including SSS, PEC, and DVS methods. Schmidt and Lee (2012) and Shands and Labuza (2009) reported that corn flakes presented consistently lower moisture content using AWM method in comparison to SSS method. And Shands and Labuza (2009) had attributed this deviation to moisture heterogeneity in AWM method. They found that the grinded corn flakes with smaller size and subsequent shorter distance for moisture redistribution could present more similar data to those from traditional methods.

To sum up, AWM method has been proposed as a fast MSI measurement to substitute traditional SSS method and should be effective for samples like powders and syrups requiring short moisture homogenization time. While for solid sample with larger size in which moisture homogenization cannot be accelerated even by forced shaking or stirring, the results from AWM method are probably deviated from traditional RH controlling methods.

6. Conclusions

In this review, AWM method is evaluated thoroughly, in aspects of its fundamentals, procedures, precautions, speed, and reliability. Some conclusions and recommendations on future work could be summed up as following:

- (1) Desiccant, vacuum, thermal drying techniques have been adopted in AWM method to prepare samples with desired moisture contents. Considering very limited reports on their effects on MSI measurement, comparative analysis of MSI data, between pretreated samples and control group without treatments, is recommended to assist researchers with proper choice of moisture adjustment approaches in AWM method.
- (2) To reach moisture homogeneity in samples after moisture adjustment, it is a common strategy to seal samples in small containers or bags within hours or days with various timetemperature combinations. The choice of temperature and waiting time is a tough decision, and is usually decided by empirical experience currently.
- (3) AWM method is superior in MSI measurement at various temperatures. It could obtain a set of isotherms under difficult temperatures simultaneously and easily by just conducting a_w registration at each required temperature for prepared samples.
- (4) In sum, AWM method has the advantages of simplicity, speediness, and comparatively lower cost than HGM and other instrumental MSI measurement methods. It could expedite MSI tests to as less as tens of minutes, exclusive of periods for sample preparation. And the reliability of AWM method has been validated in a variety of materials. But there are also a few reports on discrepancy of MSI data between AWM method and other methods, which is probably attributed to insufficient waiting time to achieve moisture uniformity before and during a_w registration stage in AWM method. Further substantial

advances in this method are expected by developing assistant techniques to accelerate the process of homogenizing moisture inside samples before a_w registration, such as assistant ultrasound treatment.

Acknowledgment

Lida Zhang wishes to thank for valuable comments and language revision by Weiwei Liu.

Funding

The authors thank financial support from UCD-CSC scholarship.

References

- Abdullah, N., Nawawi, A., and Othman, I. (2000). Fungal spoilage of starch-based foods in relation to its water activity (a_w). *J. Stored Prod. Res.* **36**(1):47–54.
- Al-Mahasneh, M., Amer, M. B., and Rababah, T. (2012). Modeling moisture sorption isotherms in roasted green wheat using least square regression and neural-fuzzy techniques. *Food Bioprod. Process.* **90**(2):165–170.
- Al-Muhtaseb, A., McMinn, W., and Magee, T. (2002). Moisture sorption isotherm characteristics of food products: a review. *Food Bioprod. Process.* **80**(2):118–128.
- Argyropoulos, D., and Müller, J. (2014). Effect of convective-, vacuum- and freeze drying on sorption behaviour and bioactive compounds of lemon balm (*Melissa officinalis* L.). *J. Appl. Res. Med. Aromatic Plants* **1**(2):59–69.
- Bazardeh, M. E., and Esmaili, M. (2014). Sorption isotherm and state diagram in evaluating storage stability for sultana raisins. *J. Stored Prod. Res.* **59**:140–145.
- Bell, L. N., and Labuza, T. P. (2000). *Moisture sorption: practical aspects of isotherm measurement and use*. American Association of Cereal Chemists.
- Bispo, J. A. C., Bonafe, C. F. S., Santana, K. M. O. V., and Santos, E. C. A. (2015). A comparison of drying kinetics based on the degree of hydration and moisture ratio. *LWT—Food Sci. Technol.* **60**(1):192–198.
- Caballero-Cerón, C., Guerrero-Beltrán, J. A., Mújica-Paz, H., Torres, J. A., and Welti-Chanes, J. (2015). Moisture Sorption Isotherms of Foods: Experimental Methodology, Mathematical Analysis, and Practical Applications, in: Gutiérrez-Lopez, G. F., Alamilla-Beltrán, L., del Pilar Buera, M., Welti-Chanes, J., Parada-Arias, E., Barbosa-Cánovas, G. V. (Eds.), *Water Stress in Biological, Chemical, Pharmaceutical and Food Systems*. Springer.
- Cardoso, J. M., and da Silva Pena, R. (2014). Hygroscopic behavior of banana (*Musa ssp.* AAA) flour in different ripening stages. *Food Bioprod. Process.* **92**(1):73–79.
- Carter, B. P., Galloway, M. T., Campbell, G. S., and Carter, A. H. (2015). The critical water activity from dynamic dewpoint isotherms as an indicator of pre-mix powder stability. *J. Food Meas. Charact.*
- Carter, B. P., and Schmidt, S. J. (2012). Developments in glass transition determination in foods using moisture sorption isotherms. *Food Chem.* **132**(4):1693–1698.
- Chen, C. (2000). A rapid method to determine the sorption isotherms of peanuts. *J. Agr. Eng. Res.* **75**(4):401–408.
- Chen, C. (2002). Sorption isotherms of sweet potato slices. *Biosystems Eng.* **83**(1):85–95.
- Chen, C., and Weng, Y.-K. (2010). Moisture sorption isotherms of oolong tea. *Food Bioprocess Technol.* **3**(2):226–233.
- Chen, H.-Y., and Chen, C. (2014). Equilibrium relative humidity method used to determine the sorption isotherm of autoclaved aerated concrete. *Build. Environ.* **81**:427–435.
- Chirife, J. (1995). An update on water activity measurements and prediction in intermediate and high moisture foods: the role of some non-equilibrium situations, in: Barbosa-Cánovas, G. V., Welti-Chanes, J. (Eds.), *Food Preservation by Moisture Control: Fundamentals and Applications*. Technomic Publishing, Lancaster, Pennsylvania.

- Chisté, R. C., Cardoso, J. M., Silva, D. A. D., and Pena, R. D. S. (2015). Hygroscopic behaviour of cassava flour from dry and water groups. *Ciência Rural* **45**(8):1515–1521.
- Clemente, G., Bon, J., Benedito, J., and Mulet, A. (2009). Desorption isotherms and isosteric heat of desorption of previously frozen raw pork meat. *Meat Sci.* **82**(4):413–418.
- de Souza, T. C. L., de Souza, H. A. L., and Pena, R. D. S. (2013). A rapid method to obtaining moisture sorption isotherms of a starchy product. *Starch-Stärke* **65**(5–6):433–436.
- Decagon Devices. (2015). *AquaLab Vapor Sorption Analyzer (VSA) Manual*. Decagon Devices, Washington, USA.
- Demarchi, S. M., Ruiz, N. A. Q., De Michelis, A., and Giner, S. A. (2013). Sorption characteristics of rosehip, apple and tomato pulp formulations as determined by gravimetric and hygrometric methods. *LWT-Food Sci. Technol.* **52**(1):21–26.
- Eim, V. S., Rosselló, C., Femenia, A., and Simal, S. (2011). Moisture sorption isotherms and thermodynamic properties of carrot. *Int. J. Food Eng.* **7**(3).
- Farahani, H., Wagiran, R., and Hamidon, M. N. (2014). Humidity sensors principle, mechanism, and fabrication technologies: A comprehensive review. *Sensors* **14**(5):7881–7939.
- Fasina, O. (2008). Physical properties of peanut hull pellets. *Bioresource Technol.* **99**(5):1259–1266.
- Fieldsend, A. (2007). Influence of oil content on the equilibrium moisture content of evening primrose (*Oenothera* spp.) seeds. *Acta Agron. Hung.* **55**(4):485–489.
- Gal, S. (1981). Recent developments in techniques for obtaining complete sorption isotherms, in: Rockland, L. B., Stewart, G. F. (Eds.), *Water Activity: Influences on Food Quality*. Academic Press, New York.
- García-Pérez, J., Cárcel, J., Clemente, G., and Mulet, A. (2008). Water sorption isotherms for lemon peel at different temperatures and isosteric heats. *LWT-Food Sci. Technol.* **41**(1):18–25.
- Giner, S. A., and Gely, M. C. (2005). Sorptional parameters of sunflower seeds of use in drying and storage stability studies. *Biosystems Eng.* **92**(2):217–227.
- Haq, M. A., Sudeepa, M., Shimizu, N., and Kimura, T. (2006). Performance of an accelerated method for the determination of equilibrium moisture content. *Food Sci. Technol. Res.* **12**(1):1–7.
- Huang, Y., Hocking, A. D., Jensen, N., Richardson, K. C., and Miskelly, D. (2009). Microbiological quality of Australian breadcrumbs. *Food Aus.* **61**(12):527–531.
- Iaccheri, E., Laghi, L., Cevoli, C., Berardinelli, A., Ragni, L., Romani, S., and Rocculi, P. (2015). Different analytical approaches for the study of water features in green and roasted coffee beans. *J. Food Eng.* **146**:28–35.
- Igathinathane, C., Womac, A. R., Pordesimo, L. O., and Sokhansanj, S. (2008). Mold appearance and modeling on selected corn stover components during moisture sorption. *Bioresource Technol.* **99**(14):6365–6371.
- Igathinathane, C., Womac, A. R., Sokhansanj, S., and Pordesimo, L. O. (2005). Sorption equilibrium moisture characteristics of selected corn stover components. *Trans. Am. Soc. Agr. Eng.* **48**(4):1449–1460.
- Irigoyen, R. T., and Giner, S. (2014). Drying-toasting kinetics of presoaked soybean in fluidised bed. Experimental study and mathematical modeling with analytical solutions. *J. Food Eng.* **128**:31–39.
- Jonquères, A., Perrin, L., Durand, A., Arnold, S., and Lochon, P. (1998). Modelling of vapour sorption in polar materials: comparison of Flory-Huggins and related models with the ENSIC mechanistic approach. *J. Membr. Sci.* **147**(1):59–71.
- Kartika, I. A., Yuliani, S., Kailaku, S., and Rigal, L. (2012). Moisture sorption behaviour of jatropha seed (*Jatropha curcas*) as a source of vegetable oil for biodiesel production. *Biomass Bioenerg.* **36**:226–233.
- Lazouk, M.-A., Savoie, R., Kaddour, A., Castello, J., Lanoisellé, J.-L., Van Hecke, E., and Thomasset, B. (2015). Oilseeds sorption isotherms, mechanical properties and pressing: Global view of water impact. *J. Food Eng.* **153**:73–80.
- Li, Z., Zhang, H., Zheng, W., Wang, W., Huang, H., Wang, C., MacDiarmid, A. G., and Wei, Y. (2008). Highly sensitive and stable humidity nanosensors based on LiCl doped TiO₂ electrospun nanofibers. *J. Am. Chem. Soc.* **130**(15):5036–5037.
- Martins, M. G., Martins, D. E. G., and da Silva Pena, R. (2015). Drying kinetics and hygroscopic behavior of pirarucu (*Arapaima gigas*) fillet with different salt contents. *LWT-Food Sci. Technol.* **62**(1):144–151.
- Mulet, A., Garcia-Pascual, P., Sanjuán, N., and Garcia-Reverter, J. (2002). Equilibrium isotherms and isosteric heats of morel (*Morchella esculenta*). *J. Food Eng.* **53**(1):75–81.
- Nguyen, T. A., Verboven, P., Daudin, J. D., and Nicolaï, B. M. (2004). Measurement and modelling of water sorption isotherms of 'Conference'-pear flesh tissue in the high humidity range. *Postharvest Biology Technol.* **33**(3):229–241.
- Noriega, M. D. P., Estrada, O., and López, I. (2014). Computational model to design plastic multi-layer films for food packaging to assure a shelf life at the best cost. *J. Plas. Film Sheet.* **30**(1):48–76.
- Odamtten, G. T., and Kampelmacher, E. H. (1986). Influence of packaging material on moisture sorption and the multiplication of some toxigenic and non-toxicogenic *Aspergillus* spp. infecting stored cereal grains, cowpea and groundnut. *Int. J. Food Microbiol.* **3**(2):57–70.
- Pixton, S., and Warburton, S. (1973). The influence of the method used for moisture adjustment on the equilibrium relative humidity of stored products. *J. Stored Prod. Res.* **9**(3):189–197.
- Sablani, S., Kasapis, S., and Rahman, M. (2007). Evaluating water activity and glass transition concepts for food stability. *J. Food Eng.* **78**(1):266–271.
- Sandoval, A. J., Guilarte, D., Barreiro, J. A., Lucci, E., and Müller, A. J. (2011). Determination of Moisture Sorption Characteristics of Oat Flour by Static and Dynamic Techniques with and Without Thymol as an Antimicrobial Agent. *Food Biophys.* **6**(3):424–432.
- Sawhney, I. K., Patil, G. R., Kumar, B., and Grover, S. (1997). Influence of water activity adjustment on sorption characteristics, acceptability and microbial stability of khoa. *J. Food Sci. Technol.* **34**(2):123–127.
- Schiraldi, A., Fessas, D., and Signorelli, M. (2012). Water Activity in Biological Systems-A Review. *Pol. J. Food Nutr. Sci.* **62**(1):5–13.
- Schmidt, S. J., and Lee, J. W. (2012). Comparison between water vapor sorption isotherms obtained using the new dynamic dewpoint isotherm method and those obtained using the standard saturated salt slurry method. *Int. J. Food Properties* **15**(2):236–248.
- Shands, J., and Labuza, T. (2009). Comparison of the dynamic dew point isotherm method to the static and dynamic gravimetric methods for the generation of moisture sorption isotherms. IFT annual meeting poster. Anaheim, CA.
- Sharma, R., and Joshi, V. K. (2014). Development and evaluation of bell pepper (*Capsicum annum* L.) based instant chutney powder. *Indian J. Nat. Prod. Resour.* **5**(3):262–267.
- Srivastava, R. (2012). Humidity Sensor: An Overview. *Int. J. Green Nanotechnology* **4**(3):302–309.
- Suntaro, K., Tirawanichakul, S., and Tirawanichakul, Y. (2014). Determination of Isosteric Heat and Entropy of Sorption of Air Dried Sheet Rubber Using Artificial Neural Network Approach. *Appl. Mech. Mater.* **541–542**:374–379.
- Troller, J. (2012). *Water activity and food*. Elsevier.
- Underwood, R., Cuccaro, R., Bell, S., Gaviolo, R., Ripa, D. M., Stevens, M., and de Podesta, M. (2012). A microwave resonance dew-point hygrometer. *Meas. Sci. Technol.* **23**(8):085905.
- Vaxelaire, J., Mousques, P., Bongiovanni, J., and Puiggali, J. (2000). Desorption isotherms of domestic activated sludge. *Environmen. Technol.* **21**(3):327–335.
- Wolf, W., Spiess, W., and Jung, G. (1985). Standardization of isotherm measurements (COST-project 90 and 90 bis). *Properties of water in foods*. Springer, Netherlands.
- Yamazoe, N., and Shimizu, Y. (1986). Humidity sensors: principles and applications. *Sensors Actuators* **10**(3):379–398.
- Yan, Z., Sousa-Gallagher, M. J., and Oliveira, F. A. (2008). Sorption isotherms and moisture sorption hysteresis of intermediate moisture content banana. *J. Food Eng.* **86**(3):342–348.
- Yang, Z., Zhu, E., and Zhu, Z. (2015). Water desorption isotherm and drying characteristics of green soybean. *J. Stored Prod. Res.* **60**:25–30.
- Yu, X. (2007). *Investigation of moisture sorption properties of food materials using saturated salt solution and humidity generating techniques*. University of Illinois at Urbana-Champaign.
- Yu, X., Martin, S. E., and Schmidt, S. J. (2008). Exploring the problem of mold growth and the efficacy of various mold inhibitor methods during moisture sorption isotherm measurements. *J. Food Sci.* **73**(2):E69–E81.