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ORIGINAL PAPER

Moisture Sorption Isotherms of Oolong Tea

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Abstract The sorption isotherms of Oolong tea were determined at temperatures ranging from 5 to 50 °C. Estimated parameters and fitting ability for nine equilibrium relative humidity (ERH) models were evaluated. The modified Oswin equations were found to be an adequate model of three parameters to describe the sorption data. The Andrieu model was the only adequate model of four parameters. In comparing the results of this study with previously published data, it was found that the sorption properties were affected by species and manufacture techniques. The Guggenheim–Anderson–de Boer (GAB) model was not an adequate model as indicated by checking residual plots. The monolayer moisture content calculated from the Brunauer–Emmett–Teller (BET) model was lesser than that calculated from the GAB model. The errors of moisture content determined by measuring the ERH and temperature of samples was within 0.35%.

Keywords Sorption · Model · Oolong tea

Nomenclature

A, *B*, *C*, *D*	constants
C_o	constants
MRPD	mean relative percentage deviation
df	degree of freedom of regression model
e	standard error of the estimated value
RH	equilibrium relative humidity in decimal
M	moisture content by dry basis, %
M_o	monolayer moisture content, %
N	number of data points
R^2	coefficient of determination
T	temperature, °C
Y	measured value by the model
Y′	predicted value by the model
q_n^s	net heat of sorption isosteric, kJ/kg

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Introduction

Tea is a very popular beverage for people in the world. Many tea types have been planted. Different products are manufactured by various techniques. Green tea usually is fermented and dried at 60 °C. Black tea is dried at higher temperature and crushed into small pieces. Oolong tea, which is very popular in Northern Asia is withered by sun, fermented to a moisture content of 50–60%, rolled into a round shape by hand or machinery, and then dried rapidly to a final moisture content of 3–5% with hot air at 110 °C. Until now, Oolong tea is the most expensive product in the tea market.

For drying, mixing, and storing of tea and other agricultural products, moisture sorption isotherm information is required. Sorption models describe the relationship between equilibrium moisture content (EMC), equilibrium relative humidity (ERH), and temperature. The sorption behaviors and fitting model for various agricultural products have been discussed in detail (ASAE 1996; Chen 2000). Recently, sorption isotherm properties of medicinal and aromatic plants have been reported (Arslan and Togrul 2005; Bellagha et al. 2007; Cordeiro et al. 2006; Kaleemullah and Kailappan 2004; Zanoelo 2005).

The traditional method for determining sorption properties is the EMC method. In this method, samples are placed in an environment maintaining constant relative humidity with saturated salt solution or aqueous sulfuric acid solution. When the change in the weight of the sample is negligible, the moisture of the sample is determined and adopted as the EMC value. However, it normally takes a long time to reach the equilibrated state of moisture content. The sorption phenomena of samples during the weighting process may induce measuring errors (Lewicki and Pomaranska-Lazura 2003). Furthermore, mold may easily develop on samples in the high relative humidity environment (Chen and Morey 1989b).

An ERH technique has been developed to measure the sorption isotherms of corn kernels (Chen and Morey 1989b), sweet potato slices (Chen 2002), and several other agricultural products (Chen 2000).

A number of isotherm equations have been developed to describe the relationship between EMC, ERH, and temperature of biological materials. Some equations only model the relationship between EMC and ERH at a fixed temperature (Boquet et al. 1978; Ghodake et al. 2007; Soysal and Oztekin 2001; Temple and van Boxtel 1999). The ability of four ERH models to fit data of 18 cereal grains was evaluated by Chen and Morey (1989a). The models are the modified Henderson, the Chung–Pfost, the modified Halsey, and the modified Oswin equations. Besides these equations, the Guggenheim–Anderson–de Boer (GAB) equation was also adopted as an ERH/EMC model in the ASAE standard D245.4 (ASAE 1996). To enhance the fitting ability of the ERH equation on the sorption data, some parameters of the sorption equation were developed as a function of temperature. The four-parameter equations are the Chen–Clayton equation (Chen and Clayton 1971), the Day–Nelson equation (Day and Nelson 1969), the Kumar equation (Kumar et al. 1978), and the Andrieu equation (Andrieu et al. 1985).

Table 1 Nine models to analyze EMC/ERH data of Oolong tea

Model	Equation
Modified Henderson equation	$\mathrm{RH} = 1 - \exp[-A(T + C)M^B]$
Chung–Pfost equation	$\mathrm{RH} = \exp\left[\frac{-A}{T+C}\exp(-BM)\right]$
Modified Halsey equation	$\mathrm{RH} = \exp\left[-\exp(A + BT)M^C\right]$
Modified Oswin equation	$\mathrm{RH} = \frac{1}{\left(\frac{A+BT}{M}\right)^C + 1}$
Chen–Clayton equation	$\mathrm{RH} = \exp\left[-AT^B \exp(CT^D M)\right]$
Kumar equation	$\mathrm{RH}_R = 1 - \exp\left[-(A + BT)M^{(C+DT)}\right]$
Day–Nelson equation	$\mathrm{RH} = 1 - \exp\left(-AT^B M^{CT^D}\right)$
Andrieu equation	$\mathrm{RH} = \frac{1}{\left(\frac{A+BT}{M}\right)^{C+DT} + 1}$
GAB equation	$M = \frac{M_o BC\mathrm{RH}}{(1-B\times\mathrm{RH})(1-B\times\mathrm{RH}+BC\times\mathrm{RH})}$

A, *B*, *C*, *D*: constants, *M*: percent moisture content on a dry basis in %, *RH*: equilibrium relative humidity in decimal, *T*: temperature in °C, M_o: percent monolayer moisture content in %

Table 2 Equations of the criteria to evaluate ERH models

Criteria	Equation
MRPD	$\mathrm{MRPD} = \frac{100}{N}\sum\frac{\lvert Y-Y'\rvert}{Y}$
e	$e = \sqrt{\frac{\sum (Y-Y')^2}{df}}$

MRPD: mean relative percentage deviation, *e*: standard error of the estimated value, *Y*: measured value, *Y'*: value predicted by the model, *N*: number of data points, *df*: degree of freedom of regression model

The monolayer moisture content M_o is recognized as the optimum moisture content for good storage stability (Labuza et al. 1970). This M_o value is the critical moisture content for tea to keep flavor and quality. The Brunauer–Emmett–Teller (BET) and GAB equations were usually applied to calculate this specific value (Quirijns et al. 2005; Timmermann et al. 2001).

The oven-drying method is the standard technique for the moisture determination of tea. It requires a precise balance to measure the initial and final mass of samples. However, the time required to evaporate all water held by the sample is at least 24 h (Ghodake et al. 2007; Temple and van Boxtel 1999). A moisture determination technique for rough rice and corn kernels has been developed by the measurement of ERH and temperature (Chen 2001). This method was an easy and rapid technique to measure the moisture contents of some agricultural products.

Only a few sorption isotherms of tea have been reported. Wolf et al. (1973) had listed some sorption data of tea at 20 °C. Temple and van Boxtel (1999) measured the sorption data of Central African tea at 40, 60, and 80 °C. Panchariya et al. (2001) determined the EMC of black Darjeeling tea at several temperatures ranging from 25 to 80 °C. Arslan and Togrul (2006) measured Turkish black tea at three temperatures. Ghodake et al. (2007) reported the sorption isotherm of withered leaves, black, and green tea. However, there is no report about the sorption properties of the Oolong tea.

The objectives of this study are (1) to determine the EMC/ERH relationships for Oolong tea at temperatures between 5 and 50 °C for RHs in the range of 10–90%; (2) to evaluate the fitting ability of nine equations for the ERH data of the Oolong tea, especially four-parameter models; (3) to calculate the monolayer moisture content by the BET and GAB equations; and (4) to evaluate the accuracy of the ERH technique on the measurement of Oolong tea moisture content.

Materials and Methods

The tea used for this study was Oolong tea (*Camellia sinensis* (L.) Kuntze) grown and processed at the Doting Tea Research Center, Nantou, Taiwan. The initial moisture content of samples was 3.1% on a dry mass basis (d.b.).

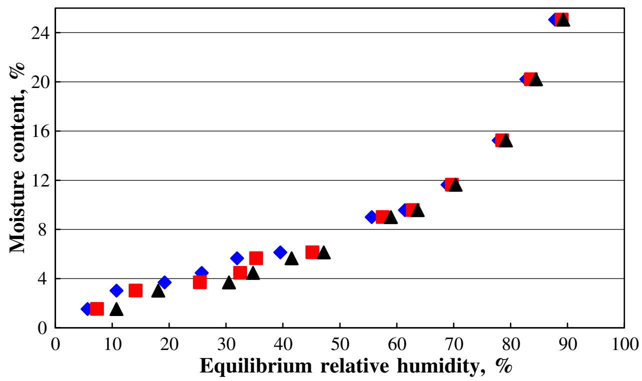

Fig. 1 Sorption isotherm of Oolong tea at three temperatures: *closed diamonds* 5 °C, *closed squares* 25 °C, *closed triangles* 50 °C

The desired moisture content was ranged from 3% to 25%, which was the moisture content for storing and packaging. Because Oolong tea was dried at 130 °C within a shorter time, adsorption data was more important that that of desorption data.

The controlled value of RH from 10% to 90% as stated in the objectives was set by the results of pretest. The initial moisture content was well controlled to be the same in all experiments.

Samples for the adsorption process were rewetted by adding the predetermined amount of water to reach the desired moisture content. The proposed process was similar as the sample preparation of rough rice (Shen and Chen 2007). All samples were sealed in plastic containers and stored at 3 °C for 4 weeks to ensure the homogeneity of the moisture content.

ERH Method

The moisture sorption isotherms of Oolong tea at five temperatures were measured by the ERH method (Chen and Morey 1989b). The volume of the container was 350 ml. Samples of known moisture content were placed in containers in a temperature-controlled chamber. Temperature and RH sensing probes (Shinyei THP-B7T Transmitter, Shinyei Kaisha, Tokyo, Japan) were inserted into the containers. Initially, the temperature of the chamber was set to 5 °C. When the temperature and RH within the sample container reached the equilibrium state, the temperature and RH were recorded. The chamber temperature was adjusted to the next level. All the ERH values were detected at five temperatures (i.e., 5, 15, 25, 35, and 50 °C). From the RH meter readings, standard relative humidity values were established with the help of a previously established calibration equation.

The humidity sensor was calibrated in the standard environment maintained by several saturated salt solutions. The sensor was placed into a closed container with a saturated salt solution. As the air within the container reaches equilibrium, the reading values of the sensor was recorded. The sensor then was moved to other closed containers with different saturated salt solutions. The relationships between the reading values of the RH sensor and the standard values maintained by the saturated salt solutions were established by regression analysis (Chen 2001).

After finishing the ERH experiments, three samples were taken from each container to determine the moisture content. Moisture content was determined by oven drying at 105 °C for 24 h (Ghodake et al. 2007; Arslan and Togrul 2006). All moisture contents are expressed as dry basis (d.b.) in this study.

Moisture Determining by ERH Method

A similar technique was proposed to determine the moisture content of tea. The same type sensor, Shinyei THP-B7T Transmitter, was adopted. The sample containers were filled with tea samples with different moisture contents. The volume of the container was 500 ml. The sensor probe was placed in the central position of container. The cover was then closed. At the equilibrium state, the

Table 3 Estimated parameters and comparison criteria for four three-parameter ERH models of sorption data for Oolong tea

Parameters	Estimated values			
	Modified Henderson	Chung–Pfost	Modified Halsey	Modified Osiwn
A	6.6308×10^{-5}	519.872	1.8577	8.3424
B	1.0621	0.1660	-4.6637×10^{-3}	-2.3443×10^{-2}
C	1198.5	181.31	1.1038	1.7669
R^2	0.966	0.949	0.981	0.983
e	4.91	6.05	3.69	3.44
MRPD	13.6	14.2	15.5	6.81
Residual plots[a]	Systematic	Systematic	Systematic	Random

A, *B*, *C*: constants, R^2: coefficient of determination, *MRPD*: mean relative percentage deviation, *e*: standard error of the estimation values
[a] Systematic or random pattern.

Table 4 Estimated parameters and comparison criteria for four four-parameter ERH models of sorption data for Oolong tea

Parameters	Estimated values			
	Day–Nelson	Kumar	Chen–Clayton	Andrieu
A	3.5553×10^{-5}	5.9253×10^{-2}	3.6372	7.9192
B	2.4421	8.7449×10^{-2}	−0.1305	-2.7903×10^{-2}
C	1.3628	1.1510	0.1867	0.5513
D	-7.0791×10^{-2}	-3.4092×10^{-3}	-4.9195×10^{-2}	2.07993×10^{-2}
R^2	0.968	0.969	0.949	0.993
e	4.78	4.78	6.05	2.45
MRPD	11.33	12.44	14.52	4.84
Residual plots[a]	Systematic	Systematic	Systematic	Random

A, *B*, *C*, *D*: constants, R^2: coefficient of determination, *MRPD*: mean relative percentage deviation, *e*: standard error of the estimation values
[a] Systematic or random pattern.

ERH and temperature of the tea were the same as the interstitial air of the container. The temperature ranged from 20 to 30 °C. The ambient temperature was measured and served as a parameter for the EMC equation. The measurements of temperature and relative humidity data were recorded after 10 min. The measured values of relative humidity were transformed to the standard value by calibration equations. The moisture contents of the samples were calculated by the EMC model that was established in this study. These calculated values were compared with the moisture contents determined by the oven method.

ERH Model

The EMC/ERH data for Oolong tea were analyzed using nine equations (Table 1). The parameters of the GAB model does not consider the temperature term. It is only evaluated for each fixed temperature.

The effect of temperature on the GAB model could be incorporated through the Clausius–Clayperon equation as follows:

$$q_n{}^s = R\left(\frac{T_{a1}T_{a2}}{T_{a2}-T_{a1}}\right)\mathrm{Ln}\left(\frac{A_{w2}}{A_{w1}}\right) \quad (1)$$

where $q_n{}^s$ is the net heat of sorption isosteric in kJ/kg, A_{w1} is the water activity of food stuffs at a temperature of T_{a1}, and A_{w2} is the water activity of food stuffs at a temperature of T_{a2}.

This method requires two sets of isotherm data. Two temperatures must be close enough so that the $q_n{}^s$ value could be assumed to be the same for both isotherms. As the sets of isotherm were obtained by the EMC method, researchers need to plot the fitting curves from scattering data and interpolate the data from plotting the curve at the fixed moisture content. The errors of this method include the experimental error in the isotherm data and the graphical manipulation. This technique is also a time-consuming work.

Data Analysis

The software, "NONLIN.BAS," which was written in QBASIC language, was developed by the authors to execute the nonlinear regression for these ERH models and to calculate all statistics used in this study.

Residual plots were applied as the qualitative criterion to assess the adequateness of the models. If the residual plots indicated a clear pattern, the model could not be accepted. If the residual plot exhibited a uniform distribution, the model can be considered as a good model (Myers 1986).

The quantitative criteria used for comparing the fitting ability of the ERH model were the coefficient of determination R^2, the standard error of the estimated value *e*, and

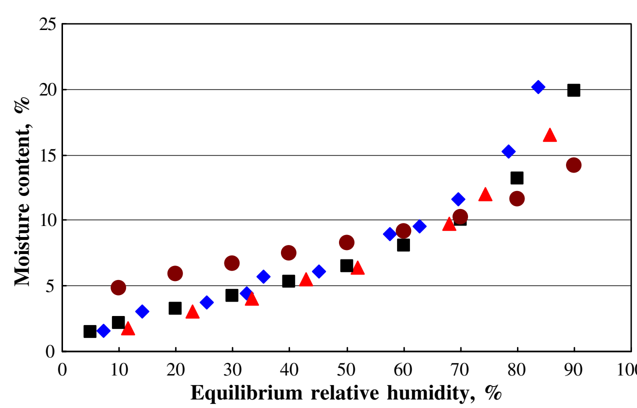

Fig. 2 Comparison of sorption data of tea at 25 °C: *closed diamonds* Oolong tea, present study; *closed triangles* Pfefferminz tea (Wolf et al. 1973); *closed squares* Central African tea (Temple and van Boxtel 1999); *closed circles* Indian green tea (Ghodake et al. 2007)

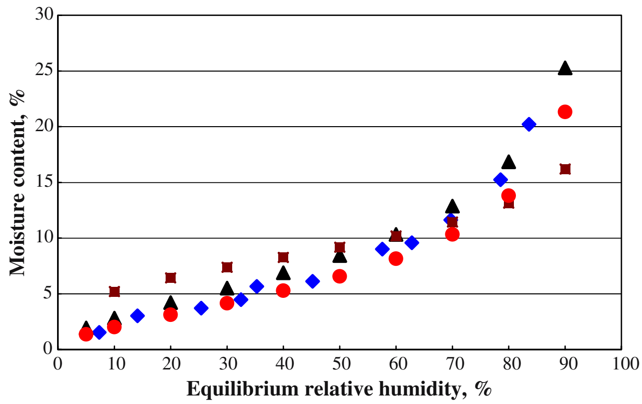

Fig. 3 Comparison of sorption data of tea at 25 °C: *closed diamonds* Oolong tea, present study; *closed triangles* Darjeeling black tea (Panchariya et al. 2001); *closed squares* Indian black tea (Ghodake et al. 2007); *closed circles* Turkish black tea (Arslan and Togrul 2006)

the mean relative percentage deviation (MRPD). These statistics formula are listed in Table 2.

Monolayer Moisture Content of Oolong Tea

The monolayer moisture content of biological materials can be calculated by the BET or the GAB equation. For the BET equation, monolayer moisture content is a parameter of this equation:

$$\frac{\mathrm{RH}}{(1-\mathrm{RH})M}=\frac{1+(C_\mathrm{o}-1)\mathrm{RH}}{M_\mathrm{o}C_\mathrm{o}} \qquad (2)$$

where *RH* is the relative humidity in decimal; M is the moisture content in percent (d.b.); C_o is a constant; and M_o is the monolayer moisture content in percent.

The BET equation is only valid for RH below 40% (Boquet et al. 1978). Only the ERH data below this range were adopted to calculate the M_o value.

The monolayer moisture content of the GAB equation is the parameter of M_o in the GAB equation.

Results and Discussion

Sorption Isotherms of Oolong Tea

The sorption data are shown in Fig. 1. The figure indicated that the sorption temperature significantly influenced the sorption isotherms below the range of 70% RH as verified by an F test. The distribution of data was nearly of the exponential form as the sorption isotherms of high oil products (Chen 2000). The oil in the products did not contain any moisture, which may be the reason the tea sorption isotherms had the same curve as high oil products.

Below the range of 70% RH, the moisture content of Oolong tea of lower temperature could hold more water at the same RH. Because of the increase in temperature, the activation of the water molecules broke away from the water-binding sites and lowered the moisture content of tea at the same relative humidity environment. The effects of temperature on the EMC of Central African tea were consistent with the results of Temple and van Boxtel (1999).

Fitting of Sorption Models to Sorption Data

The results of the estimated parameters and comparison statistics for four three-parameter ERH models are presented in Table 3. The modified Oswin equation had the highest value for R^2 and the lowest values for e and MRPD. The residual plots all displayed a uniform distribution. The other three ERH models had higher value for R^2 and lower values for e and MRPD. The residual plots all displayed a systematic pattern. Table 4 lists the estimated parameters and comparison statistics for four four-parameter ERH models. The Andrieu equation was the only adequate

Table 5 Estimated parameters and comparison criteria for GAB models of sorption data at five temperatures for Oolong tea

Parameters	Estimated values				
	Sorption temperature (°C)				
	5	15	25	35	50
M_o	4.1101	3.8861	4.0802	4.1206	4.1504
B	0.9836	0.9875	0.9737	0.9679	0.9549
C	10.084	11.209	6.9529	5.7564	5.8311
R^2	0.963	0.969	0.972	0.973	0.972
e	1.551	1.521	1.458	1.459	1.468
Residual plots[a]	Systematic	Systematic	Systematic	Systematic	Systematic

M_o, B, C: constants, R^2: coefficient of determination, e: standard error of the estimation values

[a] Systematic pattern.

model. This equation had the largest value of R^2 and the smallest values of e and MRPD. The residual plots displayed the uniform distribution.

The Andrieu equation and the modified Oswin equation had the same form. Because the parameter C in the latter equation was modified as the linear function of temperature, the form of the two equations was similar. Comparing both equations, the Andrieu model had the smallest values of e and MRPD. However, the form of the Andrieu equation was more complex than that of the modified Oswin equation.

Comparison with Published Data

The adsorption isotherms of Oolong tea at 25 °C in this study were compared with the adsorption data of Pfefferminz tea (Wolf et al. 1973), Central African tea (Temple and van Boxtel 1999), and Indian green tea in Fig. 2. At the RH values below 60%, good agreement between the sorption data of Pfefferminz tea, Central African tea, and Oolong tea was found.

The sorption isotherms of Oolong tea at 25 °C obtained in this study were compared with three kinds of black tea (Panchariya et al. 2001; Ghodake et al. 2007; Arslan and Togrul 2006).As can be seen in Fig. 3, the sorption data of the four sets were not fully consistent.

Chen (2000) also reported some inconsistency between the sorption data of the same biological materials within several agricultural products, such as corn kernels, soybeans, etc. The reason for the inconsistency of the sorption data in this study and published data could be explained by different species, measuring method, drying temperature, and size of samples. It is important to determine the sorption isotherms of all varieties of tea.

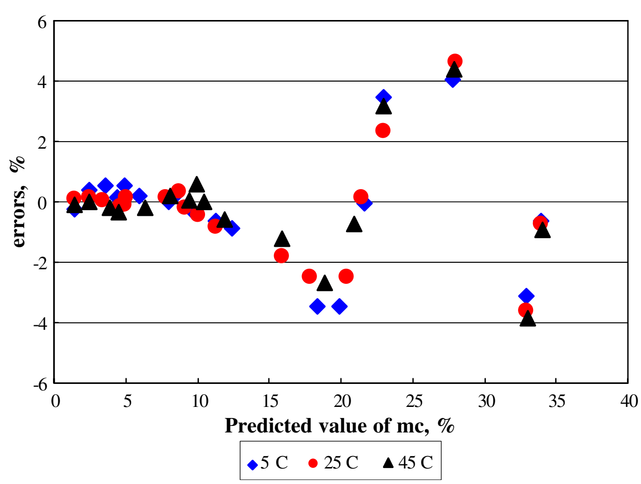

Fig. 4 Residual plots for the GAB equation for sorption data of Oolong tea: *closed diamonds* 5 °C; *closed circles* 25 °C; *closed triangles* 50 °C

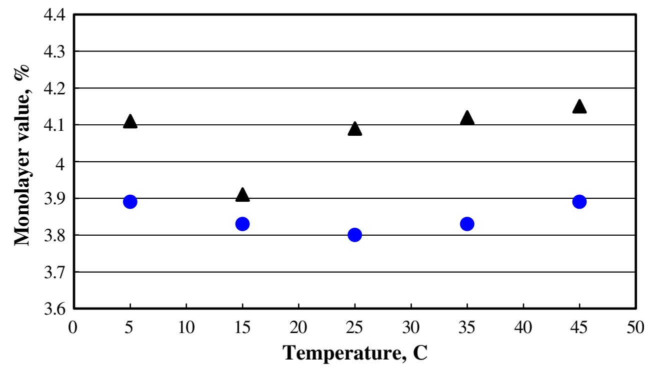

Fig. 5 Relationship between the monolayer moisture content and temperature: *closed triangles* GAB equation; *closed circles* BET equation

Evaluation of the GAB Model

The estimated parameters and statistics of the GAB equation for sorption data in this study are listed in Table 5. All the sorption data at fixed temperature had high R^2 values. However, all residual plots of the GAB model had systematic patterns (Fig. 4).

The parameters of the GAB model does not consider the temperature term. It is only evaluated for each fixed temperature. The number of data of the GAB equation to fit the sorption models was less than that of the eight ERH equations all incorporated the temperature term. It was inadequate to compare these models by R^2 values.

However, all residual plots of the GAB model had systematic patterns (Fig. 4), indicating that the GAB model is not an adequate model for the sorption data of Oolong tea. Hence, this result reflects that of other agricultural products, such as soybeans (Chen and Jayas 1998), sweet potato slices (Chen 2002), and *Maytenus ilicifolia* leaves (Cordeiro et al. 2006).

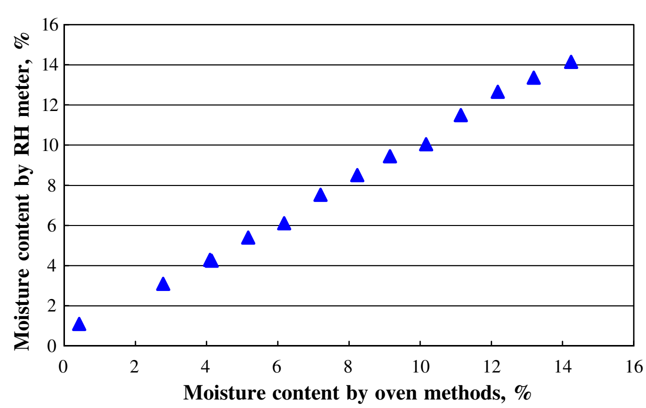

Fig. 6 Comparison between the moisture content calculated by the Andrieu equation and standard values determined by oven drying for Oolong tea

The Monolayer Moisture Content of Oolong Tea

The monolayer moisture content of Oolong tea calculated from the BET and GAB equations is presented in Fig. 5. The M_o values obtained from the GAB equation were higher than that from the BET equation. The reason for the difference between the M_o values obtained from these two equations had been discussed by Timmermann et al. (2001) and Quirijns et al. (2005). The GAB equation cannot be recognized as the adequate model for the sorption data of Oolong tea by an analysis of residual plots. However, the BET equation was the adequate model by checking of the residual plots. Therefore, the M_o value calculated by the BET equation was recommended as the optimum moisture content for long-term storage. The processing works of tea were interested in the effects on storage and package, rather than drying. The value could serve as the adequate index.

The M_o values of the BET equation were proposed as a function of temperature (Timmermann et al. 2001; Arslan and Togrul 2006). In this study, the M_o value was independent of the temperature. The average value of M_o, 3.86%, was recommended as the optimum moisture content. The corresponding ERH at this monolayer moisture content value is nearly 33.5%. The optimum ERH value for food products with longer shelf life was recommended ranging from 30% to 35% (Labuza et al. 1970). The result of this study corresponded with this RH range.

Moisture Determination with ERH Method

After 10 minutes of equilibrium time, the ERH and temperature reading of the transmitter were recorded. The moisture contents calculated by the Andrieu equation were compared with the standard values obtained by the oven method (Fig. 6). The Andrieu model was transformed as the function of RH and temperature as follows:

$$M = \left(7.91922 - 2.79 \times 10^{-2}T\right)\left(\frac{\mathrm{RH}}{1-\mathrm{RH}}\right)^{\frac{1}{(0.5513+2.08\times10^{-2}T)}}. \quad (3)$$

As indicated by Fig. 6, the measured and calculated data points nearly displayed a 1:1 relationship. The errors of tea moisture measurement by this ERH technique were random. The average absolute error was 0.35%. The same products from different batches may have different sorption properties because of the effects of chemical composition, hysteresis, and drying temperature. That is the source of measurement uncertainty of this moisture determination method. This technique provides an easy method to measure the tea moisture content within shorter time.

Conclusions

The ERH and EMC of Oolong tea at five temperatures were determined. The modified Oswin and Andrieu equations are the adequate models. The comparison of sorption data from the literature with the results of this study revealed some inconsistency in the results. The GAB equation was not an adequate equation based on the analysis of residual plots. The monolayer moisture content calculated by the BET equation was 3.86% with the standard deviation of 0.014%. The corresponding ERH is 33.5% for long-term storage. The accuracy of tea moisture determination by the ERH technique was 0.35%.

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