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STUDY OF MOISTURE ADSORPTION PROCESS IN COMFREY (*SYMPHYTUM OFFICINALE* L.) ROOTS AT 25 °C

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Sorption isotherms of comfrey (Symphytum officinale L.) root samples with different particle size were obtained at 25 °C. The shape of isotherm was similar to those of high-sugar-content foods and the particle size did not affect adsorption process in the a_w *range used in this study. Blahovec–Yanniotis model was considered to give the best fit over the whole range of* a_w *tested. Various parameters describing the properties of sorbed water derived from GAB and Blahovec–Yanniotis models have been discussed. DSC method was used to measure the glass transition temperatures (*T_g*) of root samples in relation to water activity. The safe moisture content was determined in 13.39 g/100g in dried basis at 25 °C. Combining of the* T_g *line with sorption isotherm in one plot showed that*

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the glass transition temperature concept overestimated the temperature stability for both root samples.

Introduction

The knowledge of the moisture content and water activity (a_w) relationships of food products is very important. These can be estimated by determining of moisture sorption isotherm that can be used for defining storage conditions and in making shelf life determination [1]. Unfavorable storage conditions may lead to deterioration of the product caused both by native enzymes and by microorganisms' activity [2]. A large amount of data has been published considering the sorption behavior of foods and food products including meat [3], cereal products [4] or fruit and vegetables [5,6]. Medicinal herbs and spices used in the folk medicine are also in the scope of the interest [7-9].

Many empirical relations describing the sorption characteristics of foods and food ingredients have been proposed in the literature [10]. The GAB (Guggnheim–Anderson–de Boer) equation is a general model for predicting the sorption isotherm for most products including herbs [8,11]. The monolayer moisture content, a key parameter corresponding to the physical and chemical stability of foods, can be derived from this model. For the purpose of optimizing the storage condition of various products, the sorption model with parameters which can be easily applied to the practice is a better choice. This situation represents the GAB equation with its monolayer moisture content. Similar equations have been proposed including Blahovec–Yanniotis model [12] and Caurie models [13]. Blahovec–Yanniotis equation allows distinguishing between the amount of water bonded to the sorption site of non-soluble solids and the amount of water presents in aqueous solution. Caurie [13] proposed a model which resulted in various parameters such as monolayer value, density of sorbed water, surface area of sorption and the number of adsorbed monolayers. All these parameters may be helpful in understanding the moisture behavior and thus setting appropriate storage conditions.

In the recent years, the concepts related to the a_w have been enriched by those of glass transition temperature (T_g). It is defined as the temperature at which the material changes from the glassy to the rubbery state for a given heating rate. The food biopolymers are stable at glassy state where rotational mobility of molecules is restricted while changes may occur at or above T_g [14]. Despite the fact that T_g and sorption isotherms represent two different criteria, Sablani [15] proposed both concepts to be used for determination of food stability. T_g is product specific and is a function of moisture content (or water activity) of material. In addition, they concluded the both concepts need a substantial revision since there is a discrepancy in setting the so-told safe temperature for storage of various food products. Glass transition temperature as a function of moisture content for some solid and high sugar content food have been reported [16-20].

Comfrey (*Symphytum officinale* L.) belongs to the borage family, and historically it was used to treat gastrointestinal disorders. In 2001, however, the U.S. Food and Drug Administration banned the sale of oral products containing comfrey because of the content of pyrrolizidine alkaloids [21]. Comfrey has been also studied for its anti-inflammatory [22] and antifungal activity [23]. Recent medical studies revealed that comfrey root extracts have pronounced analgesic and anti-exudative properties and may serve as a therapeutic agent in the treatment of facture, ankle distortion or osteoarthritis [24,25]. Comfrey also contains polymeric substances exhibiting antioxidant activity against active oxygen species without cytotoxic side effects [26,27].

In today's market, the herbs can be found in many types of preparations, for instance syrups, infusions or herbal tea. Dry pieces of herb are usually stored at ambient temperature without regard to relative humidity. Since dried herbs have high prevalence of moulds, yeasts [28] or coliforms [29], it is necessary to assess appropriate storage conditions to prevent growing of, or toxin production accompanying microflora.

The purpose of this study was to determine the relationship between moisture content, water activity and glass transition temperature of comfrey root samples at the temperature of 25 °C and thus obtain the critical moisture content or water activity level for safe storage.

Materials and Methods

Sample Preparation

Chopped and pre-dried pieces of roots (approximate size 1.0×0.5 cm) of *Symphytum officinale* L. were purchased in local supply. The approximate composition was determined according to AOAC procedures [30]: moisture content (method 920.36), ash content (method 942.05), ether extract (method 932.02) and crude protein (method 2001.11). The total carbohydrate content was calculated from the difference. All the measurements were done in triplicate in two consecutive trials. The results are summarized in Table I.

For the sorption study, the samples were pre-dried in forced-air oven at the temperature of 50 °C for 24 hours, and then the roots were manually ground in a mortar and sieved to give a fine particle size of ≤ 1.0 mm. The particles greater than 1.0 mm (in the range of 1.0-5.0 mm) were also used in this study. Both types of samples were subsequently dried over silica gel to constant weight.

Table I Approximate chemical composition of comfrey (*Symphytum officinalis* L.) (in g/100 g dry basis, average ±*SD*, six replicates)

	Comfrey
Ash	10.48 ± 0.22
Crude protein (nitrogen content ×6.25)	5.22 ± 0.95
Ether extract	0.57 ± 0.16
Total carbohydrate[a]	70.83

[a]Calculated from differences

Determination of Moisture Adsorption Isotherm

For the adsorption studies, following salt slurries were used according to Stolloff [31]: LiCl, CH_3COOK, $MgCl_2$, K_2CO_3, $Mg(NO_3)_2$, $CoCl_2$, NaCl, KCl and KNO_3. The moisture adsorption isotherms were determined gravimetrically by exposing root samples (approximately 1.0 g) in aluminum pans to different relative humidity generated by salt slurries in the approximate range of 0.12 to 0.93 a_w. A few crystals of thymol were placed in desiccators with relative humidity higher than 75 % to prevent mould growth. After 2-3 weeks, the samples were gradually withdrawn and the true equilibrated water activity at 25 °C was measured in Novasina instrument. After measurement, the sample was immediately placed in forced-air oven and the appropriate moisture content was determined according to AOAC procedure (method 920.36).Each sample was weighed by means of analytical balance (sensitivity ±0.001 g). The results were expressed in g per 100g of dry basis (d. b.). The isotherms were obtained by plotting the moisture content versus a_w. Each isotherm was constructed using data of three replicates.

Isotherm Models

The experimental sorption data of two samples at 25 °C were fitted to three sorption equations, namely Guggenheim–Anderson–deBoer (GAB), Blahovec–Yanniotis (B–Y) and Caurie's equation. The GAB equation is the most versatile for various food products and biomaterials [6] and was used in the following form

$$M = \frac{M_0 C K a_w}{\left[\left(1 - K a_w\right)\left(1 - K a_w + C K a_w\right)\right]} \qquad (1)$$

where a_w is the water activity, M is the experimental moisture contents, M_0 is the monolayer moisture contents on dry basis (g/100g), C is the constant related to the heat of sorption.

Blahovec–Yanniotis [12] introduced a 4-parameter model based on the assumption that a part of the water is sorbed on non-soluble solids forming a monolayer while the rest of the water is available as solvent for soluble solids. The equation was expressed in the form

$$M = \frac{a_w}{a_1 + b_1 a_w} + \frac{a_w}{a_2 - b_2 a_w} \tag{2}$$

where a_w is the water activity, M is the experimental moisture content, a_1, a_2, b_1, b_2 are constants. The first term in Eq. (2) represents the amount of water bonded to the sorption sites of the non-soluble solids and the second term represents the amount of the water in an aqueous solution.

The parameters of the equations (Eqs. (1) and (2)) were estimated using non-linear regression technique (Gauss–Newton) in QCExpert^{TM} v 3.0 (TriloByte s.r.o., Pardubice, the Czech Republic). The goodness of fit was evaluated with the mean relative percentage deviation (%E) using the formula

$$\%E = \frac{100}{n} \sum \left| \frac{M_e - M_p}{M_e} \right| \tag{3}$$

where M_e are experimental and M_p are predicted moisture contents. A model is considered acceptable if the %E values are below 10 % [32]. The sign test of residual values was used as an additional indicator of the model's quality. The following testing model was applied

$$S_g = \frac{n_s - n_t + C}{\sqrt{D_t}} \tag{4}$$

where n_s and n_t are the numbers of sequences of the same sign for experimental and theoretical residuals, D_t is error of theoretical sign sequences, C is correction for continuity. If the sign test reveals a trend in residual values, the model should not be accepted [33].

Caurie's equation was applied in its linear form previously published by Caurie [13]

$$\ln \frac{1}{M} = -\ln DM_c + \frac{2D}{M_c} \ln \frac{1 - a_w}{a_w} \tag{5}$$

where a_w is the water activity, M and M_c are the experimental and monolayer moisture contents on dry basis (g/100g), respectively. D is the constant related to the density of sorbed water (g ml^{-1}). Monolayer values (M_c) were calculated using Caurie's plot of ln(1–a_w)/a_w versus ln(1/M) in the a_w range from 0.12 to 0.93. The surface area (A, m^2 g^{-1}) and the number of adsorbed monolayers (N) were calculated according to Caurie [13] using the formulae

$$A = \frac{54.54}{S} \tag{6}$$

$$N = \frac{2}{S} \tag{7}$$

where S is the slope of Caurie's plot.

The constants of Caurie's equations were computed by the least squares method. The statistical differences were calculated using analysis of variance (ANOVA) at the probability level of $p = 0.05$.

Glass Transition Temperature

The samples were adjusted to various water activities by adsorption process as described above. After the measurement of water activity, the glass transition temperature was immediately measured using differential scanning calorimeter (DSC 204 F1 Phoenix, Netzsch, Germany) equipped with an intracooler precalibrated with indium. An amount of about 8.5 mg was hermetically sealed in aluminum pans. Each sample was cooled to the temperature of –100 °C at the rate of 10 °C min^{-1} and the scanning was performed by heating at the same rate from –100 °C to 100 °C with the empty aluminum pan as a reference. The glass transition temperature was determined from the midpoint of the heat capacity change observed on the second run.

The glass transition temperature and moisture content relationship was modeled using Gordon–Taylor equation [34]

$$T_g = \frac{X_s T_{gs} + h X_w T_{gw}}{X_s + h X_w} \tag{8}$$

where X_s and X_w are the mass fractions of solid and water, respectively. T_g, T_{gs}, T_{gw} are the glass transition temperature of mixture, solids and water, respectively. T_{gw} = 138 K, h is the Gordon–Taylor parameter.

Results and Discussion

Moisture Adsorption Isotherms

The results of the experimental measurements of the equilibrium moisture contents of root samples at 25 °C during adsorption are given in Fig. 1.

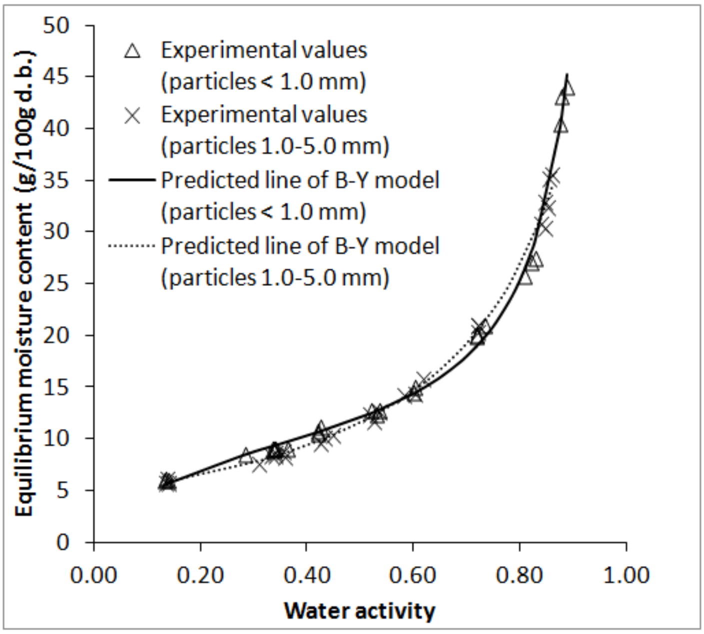

Fig. 1 Adsorption isotherm of comfrey (*Symphytum officinale* L.) at 25 °C

The shape of the isotherms is characteristic of high-sugar-containing foods adsorbing relatively small amount of water at low water activities and large amounts at high water activities, particularly above 0.60a_w. The similar shape was determined in other plant parts such as eucalyptus leaves [8], miscanthus stems and leaves [35] or sufflower and tarragon [36]. The effect of particle size of samples did not affect the adsorption process in *the* a_w range used in this study (p > 0.05). As Strange and Onwulata [37] stated the effect of particle size on the moisture adsorption should not be generalized. Whereas oat fiber adsorbed more water with decreasing the particle size, wheat bran adsorbed less water and corn bran did not correlate with particle size in their study. On the other hand, Mathlouthi and Roge [38] found that moisture adsorption increased with decreasing of sucrose crystal size distribution.

Sorption Isotherm Models

Two isotherm equations (Eqs (1) and (2)) were used for establishing the degree of fit to the experimental data. Estimated parameters for these equations are presented in Table II.

Table II Estimated parameters for selected models of adsorption isotherm equations for comfrey (*Symphytum officinale* L.) at 25 °C

Parameters of isotherm	Comfrey root particles	
	≤1.0 mm	1.0-5.0 mm
GAB (Eq. (1))		
M_0	5.861	6.563
C	264.8	17.25
K	0.974	0.946
$\%E$	5.211	3.876
S_g	3.287	1.172
p	0.001	0.241
	pattern	random
Blahovec–Yanniotis (Eq. (2))		
a_1	0.015	0.016
b_1	0.091	0.164
a_2	0.272	0.142
b_2	0.278	0.131
$\%E$	3.751	2.674
S_g	1.401	0.788
p	0.161	0.431
	pattern	random
Caurie (Eq. (4))		
M_c	7.223	6.949
D	1.805	1.795
A	108.9	105.4
N	4.000	3.871
R^2	0.974	0.974

M_0 – monolayer moisture content (g/100 g d. b.); M_c – bonded water (g/100 g d. b.); D – density of sorbed water (g ml^{-1}); A – surface of sorption (m^2 g^{-1}); N – number of adsorbed monolayers; $\%E$ – mean relative percentage deviation; S_g – sign test of residuals; p – probability level; C, K, a_1, a_2, b_1, b_2 – parameters of adsorption models, R^2 – coefficient of determination

Both GAB and B–Y models fitted well the experimental data with $\%E < 4.00$. However, the sign test showed that the GAB equation was not acceptable for fitting the experimental data of comfrey root samples with particle size ≤ 1.0 mm. Blahovec–Yanniotis equation well predicted the experimental values in both root samples (Table II). This equation (Eq. (2)) well described the sorption data of many food products including starchy and high protein foods, fruits and vegetables, nuts, legumes, and seeds [39]. The advantage of this model over the GAB equation in fitting the experimental data is mainly in the region of the high water activity. Therefore, we concluded that this model better described the sorption data of comfrey and soapwort root samples in this study.

Properties of Adsorbed Water

The physical state of water in foods determines their stability during storage [2]. Therefore it is suitable to generate the information related to various aspects of water in food. The monolayer moisture contents of both root samples estimated from the GAB equation ranged from 5.8-6.6 g/100g d. b. for comfrey. The monolayer values for root samples with particle size ≤ 1.0 mm were significantly lower ($p < 0.05$) in comparison with the particle size greater than 1.0 mm. Blahovec and Yanniotis presented a different approach to moisture sorption behavior in food systems [8,39]. Using their mathematical model, one can analyze the contribution of tightly adsorbed water and solution water in the sample. It is clear from Fig. 2 that the contribution of solution water is small at low water activities but at higher a_w values (> 0.75) the contribution is significant for all the samples.

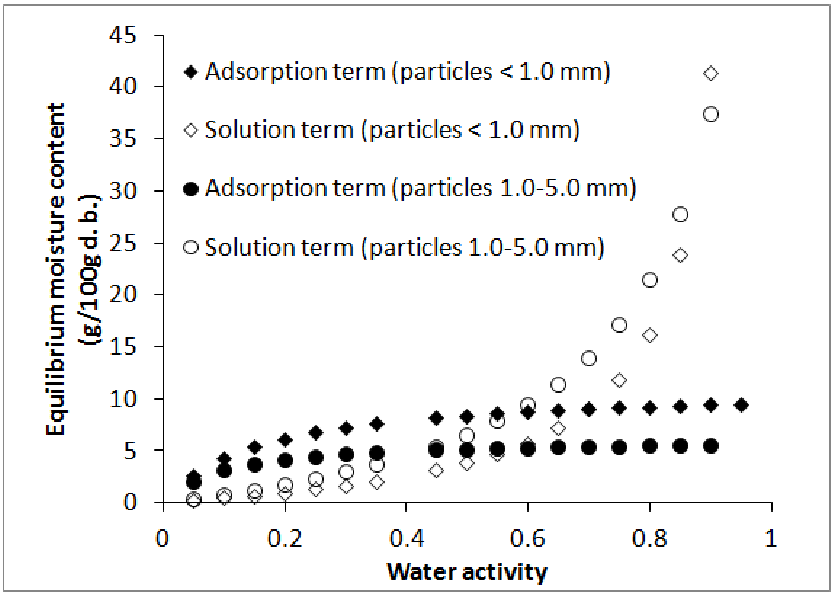

Fig. 2 Contribution of surface adsorption term and solution term in adsorption isotherm of comfrey (*Symphytum officinale* L.) Root samples at 25 °C according to Blahovec–Yanniotis equation (Eq. (2))

Although the overall adsorption isotherms (Fig. 1) did not show any difference between fine particles and those of size of 1.0-5.0 mm, some trend was observed by applying two terms of Blahovec–Yanniotis equation (Eq. (2)). The root samples ground into fine particles (≤ 1.0 mm) tightly adsorbed higher amount of water in comparison with root particle of 1.0-5.0 mm size (Fig. 2). Consequently, free or solution water became dominate above 0.70 and 0.60a_w for root particles ≤ 1.0 mm and 1.0-5.0 mm, respectively.

It was described that in the range of 0.2-0.75a_w the water interacts primarily with polar surface groups [40]. The grinding of sample into fine particles may expose more hydrophilic functional groups, therefore the root sample with particles ≤ 1.0 mm adsorbed a higher amount of water.

The parameters derived from linear regression of Caurie's equation (Eq. (5)) and computed parameters according to Eqs (6) and (7) are presented in Table II. Bonded or non-freezable water is similar to those obtained in GAB models, density of sorbed water ranged from 1.79 to 1.81 g ml^{-1} for all the samples, surface area of sorption was greater for fine particles (≤ 1.00 mm) than for particles > 1.00 mm. This parameter supports our previous finding that root samples with fine particles adsorbed more water as compared to particles > 1.00 mm.

Glass Transition Temperature

The effect of moisture content on glass transition temperature is shown in Fig. 3.

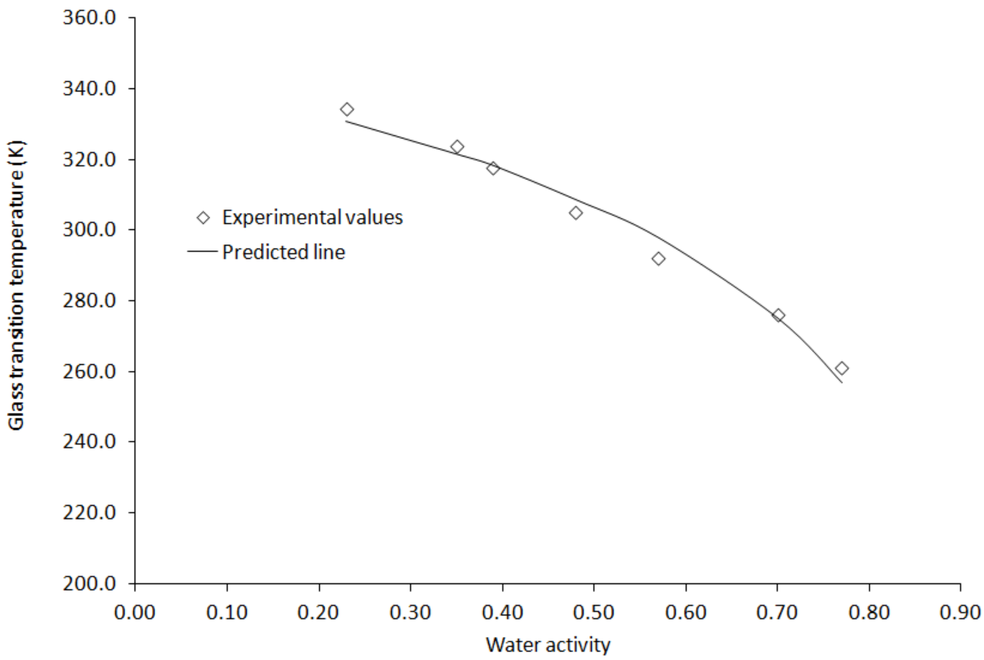

Fig. 3 Gordon–Taylor model (Eq. (8)) to predict T_g as function of water activity for comfrey (*Symphytum officinale* L.)

Glass transition temperature was found to vary with moisture content for both root samples. The decrease with an increase in moisture content was reported in many sugar rich foods such as jaggery granules [17], spray dried açai juice [20], raspberry [19], cassava starch [16] or rice [18]. The Gordon–Taylor equation has been successfully used for prediction of T_g for both root samples. The parameters estimated by nonlinear regression are shown in Table III. This table also presents mean relative percentage deviation (%E) showing that Gordon–Taylor model reasonably predicts T_{gs}.

Table III Values used in prediction of T_g for Gordon–Taylor equation (Eq. (7))

Parameter	Comfrey (*Symphytum officinale* L.) Root particles ≤1.0 mm
T_{gs}, K	341.0
k	0.210
%E	40237

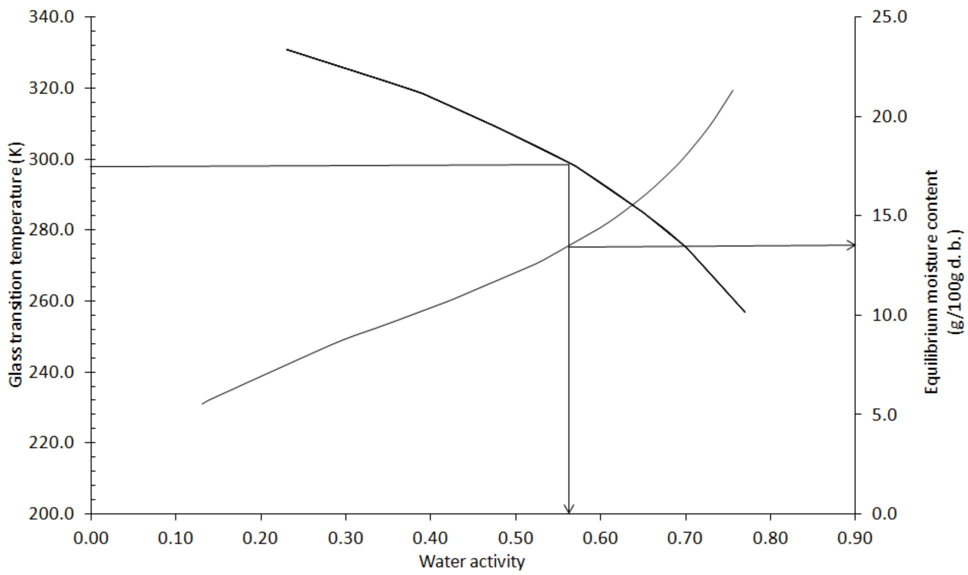

Fig. 4 Water sorption isotherms and glass transition temperatures of comfrey (*Symphytum officinale* L.) as functions of water activity

Combining the glass transition temperature concept with sorption isotherm is a useful tool for estimation of critical values for a_w and moisture content. According to Roos [41], such critical values are defined as those decreasing the T_g to ambient temperature. For estimation of critical moisture content for comfrey and soapwort root samples, the adsorption isotherm using Blahovec–Yanniotis equation and the plot of T_g vs. a_w was constructed (Fig. 4). The critical moisture contents at the temperature of 25 °C are evaluated to be 13.39g/100g d. b. for com-

Table IV Comparison of water sorption isotherm and glass transition concepts for comfrey (*Symphytum officinale* L.) root samples (particles ≤1.0 mm) at the temperature of 25 °C

Sorption isotherm model			Glass transition model		
M_0 (Eq. (1)) g/100 g d.b.	a_w corresponding to monolayer water content	T_g from glass transition model °C	Tg °C	moisture content g/100 g d.b.	a_w at corresponding water activity
5.86	0.144	60.93	40202	13.39	0.561

frey root samples.

The corresponding critical a_w value was 0.561 (Table IV). This means that when the comfrey root sample is stored at the temperature of25 °C, the maximum relative humidity which it can be exposed to is 56.1 % and its moisture content is 13.39 g/100g d. b. It was previously published that there is a surprising discrepancy between the predictions of stable temperature using sorption isotherm and glass transition concepts [15]. For example, Saymaladevi *et al*. [19] found that glass transition concept underestimated the stability temperatures for freeze-dried raspberries. The opposite effect was found in our work, where the sorption isotherm model increased the critical temperature (T_g) to 60.93 for comfrey. On the other hand, the study of Zimeri and Kokini [42] supported the results obtained in this study although they did not construct the combined plot of sorption isotherm and glass transition line. They found that one type of native inulin (Raftiline) had monolayer moisture content 7.22 % at 25 °C and the corresponding T_g can be estimated in the range of 60-70 °C. Moreover, they also concluded that glass transition temperature of inulin depended on the crystallinity, which is the function of moisture content. The detailed study on physicochemical changes of polysaccharides or phenolic compounds at a range of water contents may better elucidate the suitability of moisture sorption isotherm or glass transition temperature concepts to predict stability of root samples.

Conclusion

Blahovec–Yanniotis fits well to the experimental data for comfrey root samples. The particle size did not affect the overall adsorption properties of root samples; however, using adsorption and solution term of Blahovec–Yanniotis equation, the particle size affected the sorption behavior. Due to higher surface area of sorption and probably the higher content of polar groups, the fine particles of root samples adsorbed more water. The glass transition was determined in 13.39 g moisture/100g d. b. for comfrey. The combination of Gordon–Taylor and GAB equations into one plot revealed that there is a discrepancy between predicting the

T_g values. The glass transition temperature concept overestimated the temperature stability for both root samples.

Acknowledgements

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