

MODELLING THE MOISTURE SORPTION ISOTHERMS OF ROSELLE (*HIBISCUS SABDARIFFA* L.) IN THE TEMPERATURE RANGE OF 5–35 °C

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Abstract

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Water sorption tests of Roselle (*Hibiscus sabdariffa* L.) carried out under laboratory conditions are presented together with mathematical analyses of the moisture sorption isotherms (MSI's). Moisture equilibrium data for adsorption and desorption of water from Roselle powder were investigated at near ambient air temperatures in the range of 5 and 35 °C and water activity (A_w) ranging from 0.11 to 0.97. The manometric method has been used for water sorption tests. Models for MSI's are exponential equations. Coefficients of determination are 0.998 and 0.996 (for adsorption and desorption at 5 °C, respectively), 0.998 and 0.999 (for adsorption and desorption at 20 °C, respectively), and 0.998 and 0.999 (for adsorption and desorption at 35 °C, respectively). The equilibrium moisture content (EMC) of Roselle samples increased with an increase of A_w at a constant temperature both for adsorption and desorption. Adsorption curve equates to desorption curve at higher temperatures of tests carried out. Critical values of EMC of samples tested corresponding to the A_w equal to 0.6 were between 13.401% moisture content wet basis (MC w.b.) and 15.934% MC (w.b.) for moisture adsorption and desorption, respectively. These values are useful for storing conditions optimisation from point of view microorganisms grow and structural changes analyses. Crystal structure changes were observed during adsorption and desorption in the microscope, too. It was found out glass transition in dependence on the water content of samples tested.

water activity, equilibrium moisture content, hysteresis, prediction, grow of microorganisms, glass transition

Roselle (*Hibiscus sabdariffa* L.) belongs to the family *Malvaceae*. It originated in India and it is widely distributed in tropical and subtropical regions as a potential new food crop of considerable economic potential. Roselle is a short-day annual erect shrub and can grow to a height of 1–3 m, depending on the species. Its calyces have been suggested for the production of soft drinks, Roselle tea, jam, juices, and natural food colourants (Chen *et al.*, 2005; Chang *et al.*, 2012). The extracts of calyces and leaves

of Roselle show a significant antihyperlipidemic activity (Ochani and D'Mello, 2009).

Roselle calyx (Fig. 1) is usually harvested at high MC (w.b.), 85%. Therefore, drying is an important postharvest treatment prior to reduce the MC and to increase the shelf life. Moreover, the main purpose of drying the products is to minimize packaging requirements, to prolong the shelf life and to reduce shipping weights (Vengaiah and Pandey, 2007). Tab. I shows the chemical composition of Roselle calyces (Babalola *et al.*, 2001).

1: Fresh Roselle (Saeed *et al.*, 2008)

I: Chemical composition of dried Roselle calyces

Element	Calyx types		
	Green	Red	Dark red
Crude protein (%)	17.9	17.4	8.6
Crude fibre (%)	11.2	8.5	9.8
Ash (%)	6.6	6.5	6.8
Ascorbic acid (mg/100g)	86.5	63.5	54.8
Moisture (%)	88.3	86.5	85.3

In food industry hot air drying is probably the most widely used drying technique since it is easy to operate. However, many disadvantages of hot air drying have been identified, including oxygen-rich nature, which leads to quality degradation in terms of physical and nutritive qualities (Feng and Tang, 1998; Bai *et al.*, 2002). One way to alleviate the adverse effect of hot air drying is a pretreatment of the product, either physically or chemically (Górnicki and Kaleta, 2007). Acid pretreatment is method which can to improve the quality of product through inactivation of enzymes and texture modification. The colour of product can also be maintained due to the chelating properties of some acids (Branen *et al.*, 2002). Effect of ascorbic acid pretreatment on the colour of apple cubes was studied by Zhu *et al.* (2007). It was found that ascorbic acid was effective in slowing down the rate of enzymatic browning.

Water sorption properties of the Roselle were tested, because this surface layer is in direct contact with the near ambient air during storage and moreover it creates a natural consumable package. MC plays an important role in food quality: it influences texture and porosity, physical, chemical, and microbial properties. Values of Aw were measured from the viewpoint of the food safety (Beuchat, 1981), too. It is necessary to determine how sensitive the EMC of the food is to temperature changes.

Many other similar plants have been investigated from the aspect of moisture sorption characteristics, e.g. green instant tea powder (Sinija and Mishra, 2008), black and green tea leaves (Ghodake *et al.*, 2007), *Chenopodium ambrosioides* L. leaves (Jamali

et al., 2006), *Mentha crispa* L. leaves (Park *et al.*, 2002), safflower (*Carthamus tinctorius* L.) petals and tarragon (*Artemisia dracunculus* L.) (Kaya and Kahyaoglu, 2007), and bitter orange leaves (Mohamed *et al.*, 2005).

Critical Aw also exists below which no microorganisms can grow. For most foods, this is in the range 0.6–0.7. In general, dehydrated foods have Aw's less than 0.6; semi moist foods, such as cereal grains, raisins, syrups and intermediate moisture pet foods, usually have Aw between 0.62–0.92. Greater Aw values than 0.92 have cheeses, jams, jellies, meat, and fish. Therefore, with knowledge of the MSI, we can predict the maximum moisture that the food can be allowed to gain during storage. It means that higher Aw's can be allowed if other factors such as pH, salt content, antimicrobial agents, and temperature are taken into consideration, e.g. Štencl (1999).

The principal methods for determination of Aw are gravimetric, manometric, and hygrometric. The gravimetric method is the most common type of sorption test. It is possible to obtain MC changes of samples continuously or periodically using a static system, usually a closed jar containing saturated salt solutions or sulphuric acid solutions which give a certain equilibrium relative humidity (ERH), or a dynamic system, circulated air with a constant flow rate (Štencl and Homola, 2000). Principle of the manometric method is measurement of partial pressure of water vapour in the air above the sample. The hygrometric method is direct method based on knowledge of relationship between MC and Aw of the sample tested (Iglesias and Chirife, 1982).

The manometric static method (Iglesias and Chirife, 1982) like a most common type of sorption tests in the temperature range of 5–35 °C was used for Roselle EMC.

Changes in the mechanical properties of the material are linked to changes in moisture and temperature and can be predicted by applying the glass transition concept (Palzer, 2005). Many processes and properties encountered in food science are affected by or caused by glass transition phenomena. Some examples include frozen storage stability, freeze drying, collapse during freeze drying (melt-back), food texture, powder properties (including ease of spray-drying, stickiness, caking/clumping, crystallization and recrystallization, and starch gelatinization. Thus glass transition phenomena are important to those who seek to improve product stability and functionality (Schenz, 1995).

The glass transition can roughly be categorised as a second order phase change that is accompanied by thermodynamic changes in enthalpy, changes in dielectric properties, and mechanical changes (Carter and Schmidt, 2012). The molecular mobility of the molecules within an amorphous matrix increases with increasing temperature. Thus, the previously rigid glass-like substance is becoming first rubbery and later viscous-plastic. During this transition the liquid-like (viscous) properties are more and more dominating over the solid-like (elastic) texture components (Palzer, 2005).

Both Aw and glass transition have been used extensively in the literature to evaluate the storage stability. Product is most stable at its monolayer moisture content, i.e. Aw value of about 0.1–0.3, or at or below the corresponding glass transition temperature (Goula *et al.*, 2008). Glass transition temperature is characteristic for each material and it is the temperature range over which these discontinuities or changes occur (Jouppila, 1999).

Objectives of the study were to determine water sorption properties of Roselle powder in the temperature regime 5, 20, and 35 °C, to evaluate the effect of temperature on course of MSI's, to determine equations and coefficients of determination of sorption curves, to determine critical MC of samples corresponding Aw = 0.6, and to estimate glass transition of the Roselle powder in dependence on temperature and relative air humidity (RH) of near ambient air.

MATERIAL AND METHODS

Samples tested of Roselle (*Hibiscus sabdariffa* L.) were directly imported from Thailand. Calyces of flowers were traditional harvested, collected and treated with soaking in ascorbic acid and consequently dried in hot air at 60 °C. Form of packaged samples was powder with strong red colour and they were stored before measurements in room temperature and RH up to 70%.

Manometric static method was used for sorption tests (Iglesias and Chirife, 1982) in temperature regimes of 5, 20, and 35 °C. Weights of samples were 1213 mg in average. MC of samples was determined in the range of RH between 11% and 97%, both for water adsorption and desorption.

EMC values of samples tested were determined using six certified hygroscopic salts. These salts were: LiCl for RH 11%, MgCl₂ for RH 33%, Mg(NO₃)₂ for RH 58%, NaCl for RH 75%, BaCl₂ for RH 84% and K₂Cr₂O₇ for RH 97%. The procedure of testing was as follows: after reaching the EMC of each sample at certain ERH and at constant temperature, the RH (corresponding salt) was changed and the new equilibrium was obtained under these conditions. Halogen moisture analyser was used for gravimetric determination of samples MC (w.b.). Measurements of each test were done in triplicate and average value of the EMC was taken for mathematical analysis. The experimental EMC data were processed using the nonlinear regression procedure of Maple program.

Roselle powder was structurally analyzed during adsorption and desorption. Structure of crystal modification of Roselle powder was observed by microscope with magnification 28×–160×.

RESULTS AND DISCUSSION

The mean experimental values of EMC and Aw based on triplicate measurements for respective Aw at 5, 20, and 35 °C both for adsorption and desorption, are presented in Tab. II.

Measured data were mathematically processed and analysed using Maple. Equations were created for the functional dependence of EMC on Aw at constant temperature.

Higher EMC values were found in lower temperatures to the same RH. This could be explained by elevation of the temperature where the water molecules are activated by the high energetic level, became less stable and leaving the place of water links of food, reducing the water content of the monolayer (Leite Medeiros *et al.*, 2006).

The profile of MSI is characteristic of the hygroscopicity of a product. The measured part of MSI in the range Aw of 0.11 to 0.97 corresponds to MSI of II type of Brunauer-Emmett-Teller classification (Brunauer *et al.*, 1938), which is typical to the most of the foods. The resultant curve is caused by the additive effects of Raoult's law, capillary effects, and surface-H₂O interactions. There are the results of the changes in magnitude of the separate physical-chemical effects (Labuza, 1984).

Equations (1) to (6) model the dependence of EMC (w_c) of Roselle powder on Aw over the temperature range of 5–35 °C.

Adsorption:

$$5\text{ }^{\circ}\text{C: } w_{c,5} = 804.780Aw^{-0.409} + 11.553 \quad (1)$$

$$20\text{ }^{\circ}\text{C: } w_{c,20} = 372.281Aw^{-0.334} + 8.572 \quad (2)$$

$$35\text{ }^{\circ}\text{C: } w_{c,35} = 238.429Aw^{-0.275} + 7.931 \quad (3)$$

II: EMC (w.b.) of Roselle powder at different temperatures and A_w 's for moisture adsorption and desorption

		t ₁ = 5 °C	t ₂ = 20 °C	t ₃ = 35 °C
RH (%)		w _{e1} ^a (%)	w _{e2} ^a (%)	w _{e3} ^a (%)
Adsorption	11	11.448	8.434	7.875
	33	12.260	9.137	9.212
	58	15.155	14.081	13.070
	75	21.138	21.150	21.245
	84	27.895	26.799	25.828
	97	40.948	45.948	38.178
Desorption	11	12.293	8.964	7.531
	33	13.813	9.783	9.291
	58	18.489	14.624	13.202
	75	27.461	22.407	22.478
	84	32.243	38.082	30.090
	97	40.948	45.948	38.178

a, mean of $n = 3$ replications; RH, relative humidity (%); t , temperature (°C); w_e , equilibrium moisture content (EMC) of samples tested (% w.b.)

III: EMC's for critical $A_w = 0.6$ both for adsorption and desorption

Temperature	Critical EMC for adsorption	Critical EMC for desorption
5 °C	15.142% (w.b.)	15.934% (w.b.)
20 °C	13.401% (w.b.)	14.591% (w.b.)
35 °C	13.855% (w.b.)	14.054% (w.b.)

Desorption:

$$5\text{ °C: } w_{e,5} = 1479.675A_w^{-0.460} + 13.155 \quad (4)$$

$$20\text{ °C: } w_{e,20} = 200.341A_w^{-0.258} + 8.465 \quad (5)$$

$$35\text{ °C: } w_{e,35} = 168.153A_w^{-0.232} + 7.461 \quad (6)$$

Equations were used to determine of EMC's of Roselle powder equal to critical $A_w = 0.6$. EMC's for critical $A_w = 0.6$, are listed in Tab. III.

The coefficients of determination for the equations are 0.998 and 0.996 (for adsorption and desorption at 5 °C, respectively), 0.998 and 0.999 (for adsorption and desorption at 20 °C, respectively), and 0.998 and 0.999 (for adsorption and desorption at 35 °C, respectively).

Figs. 2, 3, and 4 show the course of MSI's of Roselle powder using Maple in temperatures 5, 20, and 35 °C. The EMC of Roselle powder was dependent on temperature. MSI's demonstrate an increase the EMC of Roselle powder samples with increasing A_w at a constant temperature both for water adsorption and desorption (Sawhney *et al.*, 2011).

The MSI's exhibited the phenomenon of hysteresis at 5 and 20 °C, in which the EMC was higher at a particular ERH for desorption curve than for adsorption. Adsorption curve equated to desorption curve at 35 °C. Therefore Roselle powder probably does not accumulate moisture in itself during desorption process at 35 °C. Storage temperature at 35 °C does not affects EMC at adsorption and desorption process.

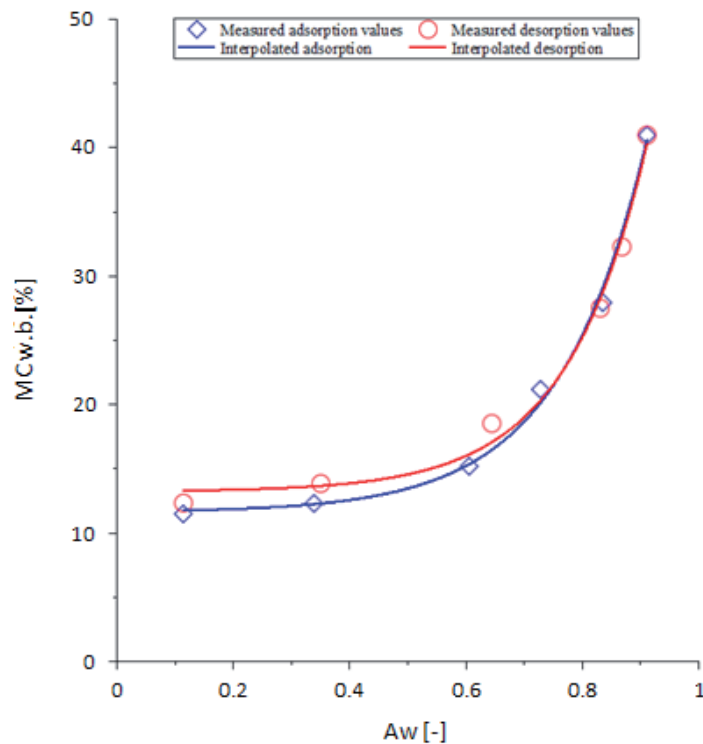
MSI's are useful for optimisation of storage conditions for wet food products, in general. The best storage condition for Roselle powder is MC

lower than corresponding to the critical value of A_w equal to 0.6. For example, the critical value of MC is 13.401% (w.b.) for adsorption in temperature 20 °C.

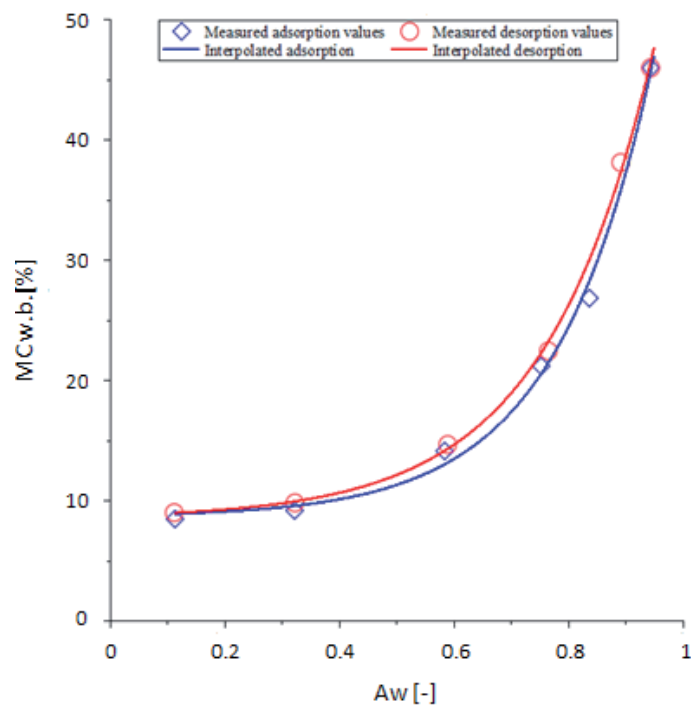
Initial structure of Roselle powder before sorption tests is shown in the Fig. 5. Structure of crystals during sorption tests was changed as shown on Figs. 6–11. Structure of Roselle powder of the surface has even appearance before sorption test. Structure of Roselle powder on the surface depends on increasing or decreasing MC. Crystals are growing by influence of adsorption of MC. The surface of Roselle powder is not fine by influence of the desorption which leads to loss of moisture. Crystals of Roselle powder clump together, form cracks and the structure of the surface is furrowed during desorption. The crystal changes are stronger during desorption process than adsorption process. It is interesting that are no visible structural changes between the modifications of crystals at different observed temperatures. The maximum crystallization occurred at RH of 58–84% for adsorption. Recrystallization and glass transition is probably in this range of RH.

While the concept of investigating and even determining glass transitions using moisture sorption seems feasible, it has not been extensively studied. The moisture sorption properties of amorphous materials change when a phase transition occurs making it theoretically possible to observe the glass transition as a change in the isotherm (Carter and Schmidt, 2012).

Phase transition from glassy to rubbery results in drastic changes in molecular mobility of food



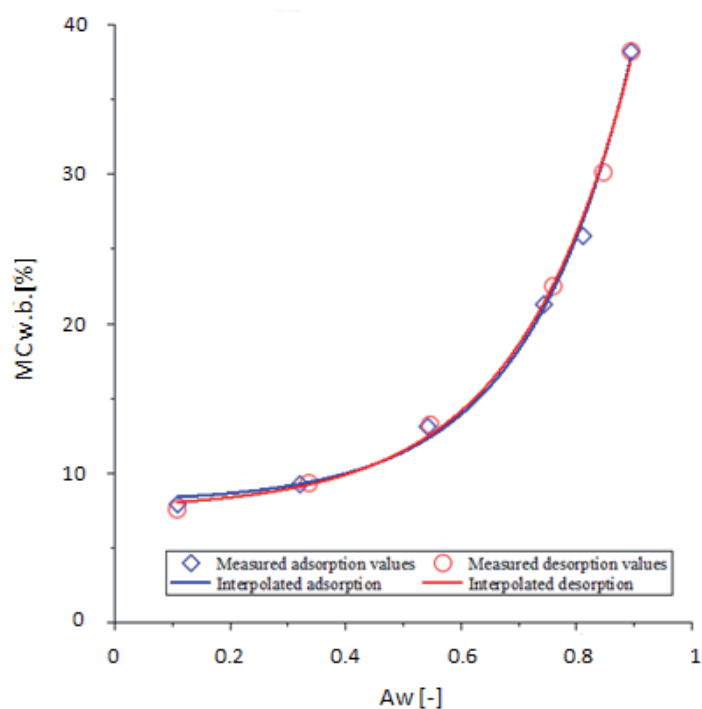
2: MSI's for Roselle powder at 5 °C



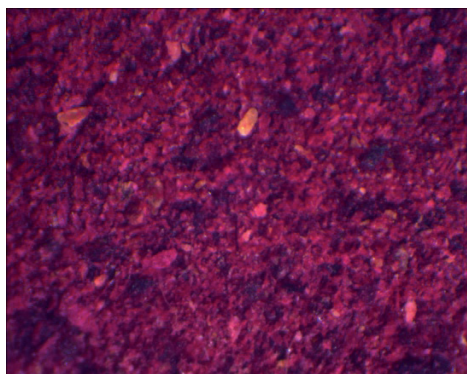
3: MSI's for Roselle powder at 20 °C

polymers. This has been linked to changes in product quality and can result in a loss of stability for low moisture amorphous foods (Carter and Schmidt, 2012). Linked to the glass transition of

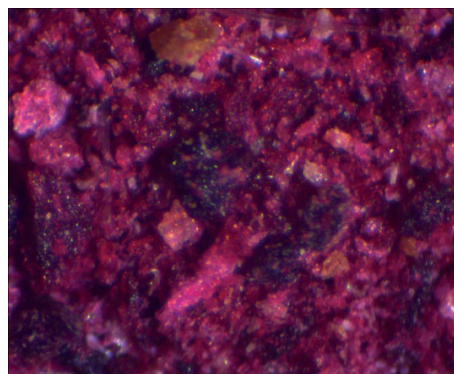
amorphous materials is their viscoelastic behaviour. Viscoelasticity depends on the strain rate, the temperature and the moisture of the food product (Palzer, 2005).



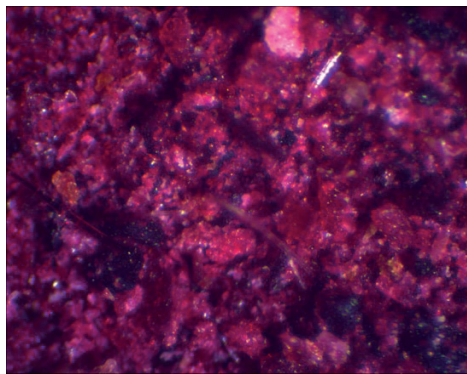
4: MSIs for Roselle powder at 35 °C



5: Roselle powder before sorption tests



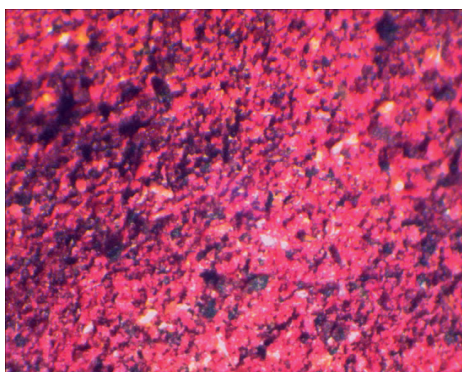
6: Adsorption of Roselle: 58% ERH at 5 °C, EMC = 15.155% (w.b.)



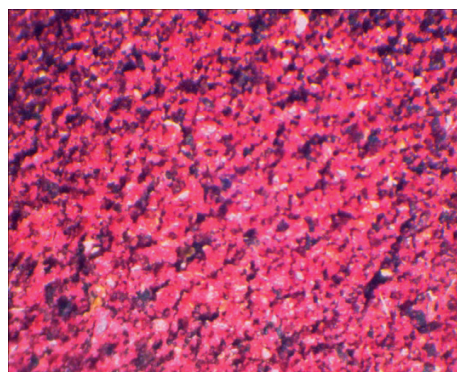
7: Adsorption of Roselle: 84% ERH at 5 °C, EMC = 27.895% (w.b.)



8: Adsorption of Roselle: 58% ERH at 20 °C, EMC = 13.990% (w.b.)



9: Adsorption of Roselle: 75% ERH at 20 °C, EMC = 21.125% (w.b.)



10: Adsorption of Roselle: 75% ERH at 35 °C, EMC = 21.241% (w.b.)



11: Adsorption of Roselle: 84% ERH at 35 °C, EMC = 25.912% (w.b.)

CONCLUSIONS

Experiments carried out under laboratory conditions for known levels of temperature and RH and data used to determine the water sorption behaviour of Roselle powder. The EMC decreases with increasing temperature, at constant A_w . Temperature affects the sorption behaviour. The measured part of MSI in the range A_w of 0.11 to 0.97 corresponds to MSI of II type of Brunauer-Emmett-Teller classification, which is typical to the most of the foods. The critical values of EMC of Roselle powder tested, corresponding to the A_w equal to 0.6, were 15.142–15.934% (w.b.) at 5 °C, 13.401–14.591% (w.b.) at 20 °C, and 13.855–14.054% (w.b.) at 35 °C. Below this value the growth of moulds does not occur. The hysteresis effect was visible only for Roselle powder at 5 and 20 °C. Observation of glass transitions is important for determining the conditions for different technological processes. Glass transition occurred at RH from 58 to 84% for adsorption. It is used to evaluate of the storage stability of powder properties.

SUMMARY

The paper presents results of water sorption tests of Roselle (*Hibiscus sabdariffa* L.) together with mathematical analyse. It was to determine effect of ambient temperature on adsorption and desorption of moisture from pretreatment ascorbic acid samples of Roselle powder. Measurements were carried out under laboratory conditions in the temperature range of 5 to 35 °C and A_w ranging from 0.11 to 0.97 using manometric static method. Created MSI's can be used to observe changes that result from increased and decreased moisture sorption. It was determined critical MC from point of view $A_w = 0.6$, below no microorganisms can grow. Critical values of EMC of tested samples corresponding to the A_w equal to 0.6 were between 13.401% MC (w.b.) and 15.934% MC (w.b.) for moisture adsorption and desorption, respectively. The EMC of Roselle powder being dependent on temperature. Sorption capacity of sample decreased with an increase in temperature at constant A_w . The MSI's demonstrated an increase in EMC with increasing A_w . MSI's exhibited the phenomenon of hysteresis at 5 and 20 °C, in which the EMC was higher at a particular A_w for desorption curve than for adsorption. Adsorption curve equated to desorption curve at higher temperature. Crystal modifications of the material tested at EMC measured are presented. The structures reported in this paper provide understanding sorption behaviour of Roselle powder during adsorption and desorption. Glass transition of Roselle powder occurs at 58–84% RH. The results would be valuable in appraising the shelf life of Roselle powder under varying storage conditions.

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