

## WATER SORPTION ISOTHERMS OF MINTS

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### Abstract

*The mints are aromatic plants with utilisation in human been food as aromatic plant as well as medicinal plant. The objective of that study was to provide fundamental data on experimental measurements of water sorption isotherms of different species of mints at various temperatures, using standardized static method. The obtained data of sorption characteristic of mints were fitted by mathematical models. The experiments results were also used in modeling the storage behavior and quality attributes of the dried mints. Seven varieties of Mint have been analysed in this experiment: *M. spicata* L., *M. aquatica* L., *M. arvensis* L., *M. longifolia* L., *M. suaveolens* Ehrh., *M. pulegium* L., and *M. piperita* L. The isotherms experiments have been conducted using standard gravimetric method recommended by Spiess and Wolf (1983). Eight salts are selected to give different relative humidity's in the range of 0.11-0.84. The experiments have been conducted at the 20°C, 30°C, 40°C, 50°C and 60°C constant temperatures. Have been describe the isotherms equation for adsorption and desorption for all mints varieties.*

**Key words:** mints, isotherm of sorption, hysteresis, water activity, BET equation.

### INTRODUCTION

Water plays a very important and unique role in agricultural products. Being present in the highest concentration, it influences a wide range of physical, chemical and biological phenomena, which occur during processing storage. Most importantly, the concentration of water affects practically all-deteriorative processes that are microbiological in nature and enzymatic or non-enzymatic in origin. The rate of the various deteriorative processes depends mainly on water concentration. The potential of water to take part in the deteriorative processes can be characterized by the water activity ( $a_w$ ) which is defined, according to the generalized Raoult's law, as the ratio between the water vapour pressure of the product at a given temperature and the saturation pressure of pure water at the same temperature (Wolf et al., 1985). At equilibrium, the water activity is related to the relative humidity of the surrounding atmosphere (Iglesias and Chirife, 1982). The importance of water sorption equations has been stressed by many researchers including the nutrient retention during dehydration and shelf life of product in a packaging material (Labuza, 1968). They are also needed for evaluating the thermodynamic

functions of the water sorbed in foods (Iglesias and Chirife, 1976), prediction of drying time (Henderson and Perry, 1976) and simulation of drying systems (Park et al., 2002). Such drying simulations could be used to predict drying time, determine the effect of change in certain parameters on the drying efficiency, or minimize operating costs. It also constitutes an essential part of the drying theory (King, 1968), and in the measurement of the latent heat of vaporization (Murata, 1988; Tagawa et al., 1993). Currently, sorption isotherms are gaining in importance as the number of recommendations, official regulations and product specifications which use water activity as an evaluation criterion is growing continuously (Wolf et al., 1985). The objective of drying mints is to extend the shelf life and conserving the fresh characteristics. This is achieved by reducing the water activity ( $a_w$ ) of the product to a value which will inhibit the growth and development of pathogenic and spoilage microorganisms, significantly reducing enzyme activity and the rate at which undesirable chemical reactions occur. The removal of most of the water from the product reduces the weight to be carried per unit product value. This can lead to substantial savings in the cost of handling and transporting the dried

product as compared with the fresh material. A reduction in volume of the dried material, as compared with the fresh, can lead to savings in the cost of storage and transport.

The sorption curves express the hygroscopic equilibrium states of a given product (Kane et al., 2008). Their determination constitutes an indispensable stage for better understanding the problems of modelling the drying processes (Akawasi, 1997; Vaios et al., 1999). Using an experimental approach, these equilibrium curves are determined by the saturated salt solutions method. The experimental sorption curves are described by (GAB), modified Halsey and Peleg equations. The GAB model is recognised as the most widely utilised and versatile (Timmerman et al., 2001), and was recommended by European COST 90 Project (Wolf et al., 1985)

Data of water desorption isotherms are very important in that aspect to properly select the final moisture content which the product is safe for storage and to determine the optimum storage conditions (Aguerte et al., 1989). Due to the complex food composition, theoretical prediction of sorption isotherms is not possible and experimental measurements are necessary (Epure et al., 2005; Gal 1985). The objective of that study was to provide fundamental data on experimental measurements of water sorption isotherms of onion at various temperatures, using standardized static method (Epure et al., 2005).

The depression of water activity in foods is due to a combination of factors each of that may be predominant in a given range of the water activity (Karel, 1973); the sorption properties may change as a consequence of physical and chemical interaction induced by heating or pre-treatments (Iglesias and Chirife, 1976); changes of water sorption it usually undergoes changes of constitution, dimensions and other properties of the product (McLaren and Rowen, 1952). Water sorption leads to phase transformations of the sugar contained in the food (Karel, 1973; Iglesias et al., 1975). Due to that, it is not possible of having a unique mathematical model, either theoretical or empirical for describing accurately the sorption isotherms in the whole range of water activity and different type of foods. The GAB equation was applied by Labuzza et al. (1985) to model moisture sorption

isotherm in the range of 0,1 to 0,9 water activity at various temperatures who reported an excellent fit to data. Spiess and Wolf (1987) used the GAB model to adsorption isotherms at 25°C. Van den Berg and Bruin (1981) have compiled and discussed some of empirical isotherm equations that have been reported in the literature for fitting water sorption isotherms of food. Chirife and Iglesias (1978) made a research of the most of the isotherm equations for fitting moisture sorptions isotherms of foods (Epure et al., 2005).

## MATERIALS AND METHODS

For this research 7 species from genus *Mentha* have been used: *Mentha piperita* L. (Peppermint), *Mentha spicata* L. (Spearmint), *Mentha aquatica* L. (Water mint), *Mentha arvensis* L. (Wild mint), *Mentha longifolia* L./Huds (Horse mint), and *Mentha suaveolens* Ehrh (Apple mint), and *Mentha pulegium* L. (Pennyroyal mint) and the material (herba) have been harvested just before flowering from Botanical Garden fields. Representative samples were taken randomly and cut manually into small pieces. The samples of 5 grams were taken randomly and placed in sorbostats. A small quantity of thymol was placed in each hygostat in order to prevent fungal activity, Wolf et al. (1985). The sorption isotherms of mints were determined using standard gravimetric method recommended by Spiess and Wolf (1983) and AOAC (1995). Eight salts are selected to give different relative humidity's in the range of 0.11-0.84. All the salt solutions used in the experiment were prepared with the reagent grade salts and distilled water, accordingly with Spiess and Wolf method (1983). Saturated salts solution have the advantage of maintaining a constant relative humidity of the air as long as the salt present is above saturation level (Karel, 1975). The effect of pressure on adsorption isotherm is negligible at reasonable levels (Okos et al., 1992). The salts solution used are presented in Table 1. The sorption containers were placed in a temperature-controlled cabinet (HERAEUS type B 5090E Germany) (Figure 1) at 20, 30, 40, 50, and 60°C. Each sample was replicated two times. First weighing was done one week from the start of the experiment using an analytical balance (SARTORIUS type BP

221S of the SARTORIUS AG Göttingen Germany) with 0,1 mg accuracy. Successive weightings were done after every three days. The equilibrium was reached when the sample weight difference between two successive measurements was less than the balance accuracy of 0.1 mg. (Saravacos et al., 1986). The

time required for the mints to reach equilibrium moisture content varied with the relative humidity and the temperature. The moisture content of the equilibrated samples was determined by drying at 105°C using hot air oven until the moisture content became constant.

Table 1. Equilibrium of relative humidity of the saturated salts solutions at six temperatures used in the experiments (Wolf and Spiess, 1983)

Salt Solutions	Temperature of saturation salts, °C				
	20	30	40	50	60
LiCl	0.1240	0.1128	0.1121	0.1110	0.1095
CH <sub>3</sub> COOK	0.2330	0.2161	0.2040	0.1920	0.1800
MgCl <sub>2</sub>	0.3360	0.3244	0.3160	0.3054	0.2926
K <sub>2</sub> CO <sub>3</sub>	0.4400	0.4317	0.4299	0.4265	0.4211
Mg(NO <sub>3</sub> ) <sub>2</sub>	0.5490	0.5140	0.4842	0.4544	0.4727
NaNO <sub>3</sub>	0.6530	0.7314	0.7100	0.6904	0.6735
NaCl	0.7547	0.7509	0.7468	0.7443	0.7450
KCl	0.8511	0.8362	0.8332	0.8120	0.8025



Figure 1. Temperature controlled cabinet HERAEUS type B 5090E with sorbostats

The equilibrium moisture content ( $U_e$ ) of the tomato, expressed in wet basis, was calculated using Eq. (1).

$$U_e = \frac{m_e - m_D}{m_e} 100 \quad (1)$$

For the analysis and presentation of sorption data, the moisture content obtained were converted to dry basis expressed in kg water / kg solid (Iglesia and Chirife, 1982), using Eq. (2).

$$X_e = \frac{U_e}{100 - U_e} \quad (2)$$

The data were graphically presented by plotting  $X_e$  versus  $a_w$ , computed using Eq. (3).

$$a_w = \frac{P_f}{P_0} = \frac{H_e}{100} \quad (3)$$

The data were further analyzed by fitting them to two-parameters isotherm equations (Table 1). Using regression analysis, each equation was linearized to solve for the constant and the degree of the linear relationship between  $X_e$  and  $a_w$  was determined for each temperature by solving the coefficient of correlation R using Eq. (4).

$$R = \frac{n \sum X_e a_w - (\sum X_e)(\sum a_w)}{\sqrt{[n \sum (X_e)^2 - (\sum X_e)^2][n \sum (a_w)^2 - (\sum a_w)^2]}} \quad (4)$$

To determine the percent of variation of the  $X_e$ , the coefficient of determination ( $R^2$ ) was computed. The accuracy of the fit was determined by calculating the root mean square error (RMS) cited by Gal (1981), Greenspan (1977), and Tagawa et al. (1983), using Eq. (5).

$$RMS = \sqrt{\frac{\sum_{i=1}^n (X_{e_i} - X_{c_i})^2}{n-1}} \quad (5)$$

The smaller the value of RMS, the better is the fitting of the equation to the experimental sorption data. The parameters of the BET equation were determined by plotting  $aw / X (1 - aw)$  against  $aw$ . From the slope and intercept of the line, the constant  $X_m$  and  $C$  were determined (Aquerte et al., 1989; Timmerman et al., 2001; van den Berg, 1985).

# RESULTS AND DISCUSSIONS

The results of the experimental measurements of water desorption and adsorption isotherms of mint for all ranges of temperatures analysed are presented in Figures 1 for desorption and Figure 2 for adsorption. The experimental points are based on a mean value of two replications. The obtained sorption isotherms of mint showed the typical sigmoid shape of type II, according to the BET classification (Brunauer et al., 1938) for all 7 species of genus *Mentha* (Peppermint, Spearmint, Water mint, Wild mint, Horse mint, Apple mint and Pennyroyal mint). Higher equilibrium moisture contents were found at the lower temperature for the same relative humidity for all mint species analysed.

The desorption isotherm is higher than the adsorption isotherm at the same temperature and relative humidity for all mint species analysed. The adsorption and desorption isotherms exhibited hysteresis for the entire range of relative humidity and for all species of mint analysed. That is in concordance to the theory of physical sorption (Iglesias et al., 1975), and have been reported also by Kane et al. (2008), Dalgic et al. (2011).

Hysteresis are found to be higher at lower temperatures than higher temperatures. It is also observed from the hysteresis curves that the hysteresis loop decreased with decreasing of relative humidity and have been reported also by Park et al (2002).

The experimental results of determination of isotherm of desorption for Peppermint are presented in Figure 2.

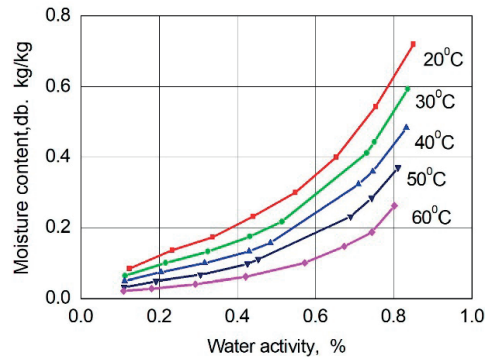


Figure 2. Water desorption isotherms of *Mentha piperita* at different temperatures

After experiments have been conducted, using plotting equation the parameter  $X_m$  and  $C$  of BET equation have been calculated and also the coefficient of determination  $R^2$ , for isotherms of desorption of peppermint (Table 2) and for isotherms of adsorption (Table 3)

Table 2. BET equation parameters and goodness of fit of desorption of Peppermint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1193	39.8203	0.9496
30	0.1053	13.6487	0.9846
40	0.0878	9.6443	0.9837
50	0.0752	4.5277	0.9963
60	0.0556	2.7119	0.9979

The experimental results of determination of isotherm of adsorption for Peppermint are presented in Figure 3. That correspond with data presented by Dolgic et al. (2012). Different values of coefficients of BET equation could occur due to different chemical composition of fresh and dried mints used in experiments due to different crop and harvest conditions accordingly with Iglesias and Chirife (1976), Wolf et al. (1985), Timmerman et al. (2001).

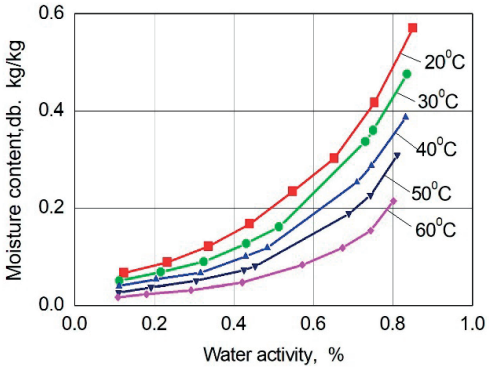


Figure 3. Water adsorption isotherms of *Mentha piperita* at different temperatures

The values of coefficient of determination  $R^2$ , for isotherms of desorption of Peppermint (Table 2) and for isotherms of adsorption (Table 3) indicate that the BET equation could be use with a good estimation of water activity for Peppermint for all ranges of isotherms between 20 and 60 °C.

Table 3. BET equation parameters and goodness of fit of absorption of Peppermint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0932	21.1306	0.9636
30	0.0856	8.0228	0.9815
40	0.0706	6.7580	0.9848
50	0.0632	3.2062	0.9970
60	0.0485	1.9913	0.9951

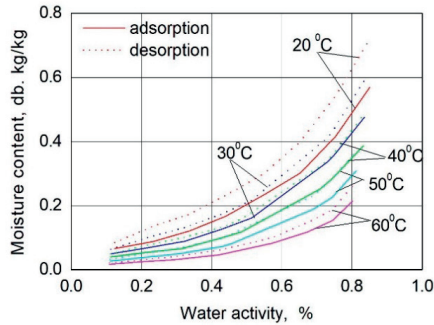


Figure 4. Water sorption isotherms of *Mentha piperita* at different temperatures

The hysteresis loop decreased with decreasing of moisture content of Peppermint and with increasing of temperatures (Figure 4).

The experimental results of determination of isotherm of sorption for Peppermint are presented in Figure 5.

The values of coefficient of determination  $R^2$ , for isotherms of desorption of Spearmint (Table 4) and for isotherms of adsorption of Spearmint (Table 5) indicate that the BET equation could be use with a good estimation of water activity for Spearmint for all ranges of isotherms between 20 and 60°C.

The isotherms present hysteresis for all range of isotherms and all values of water activity for Spearmint (Figure 5).

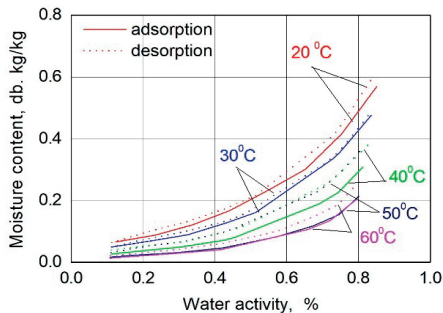


Figure 5. Water sorption isotherms of *Mentha spicata* at different temperatures

Table 4. BET equation parameters and goodness of fit of desorption of Spearmint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0857	8.0225	0.9813
30	0.0875	9.6447	0.9834
40	0.0756	4.5278	0.9969
50	0.0481	1.9912	0.9953
60	0.0474	1.6955	0.9973

Table 5. BET equation parameters and goodness of fit of absorption of Spearmint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0931	21.1309	0.9632
30	0.0704	6.7586	0.9845
40	0.0879	9.6444	0.9834
50	0.0750	4.5279	0.9961
60	0.0481	1.9919	0.9959

The experimental results of determination of isotherm of sorption for Water mint are presented in Figure 6.

The values of coefficient of determination  $R^2$ , for isotherms of desorption of Water mint (Table 6) and for isotherms of adsorption of Water mint (Table 7) indicate that the BET equation could be use with a good estimation of water activity for Water mint for all ranges of isotherms between 20 and 60°C.

The isotherms present hysteresis for all range of moisture content and all values of water activity for Water mint (Figure 6).

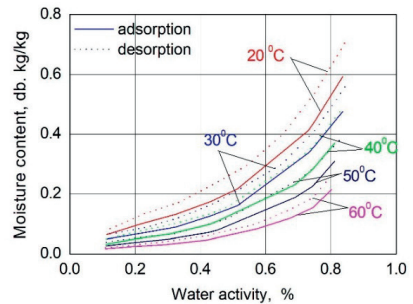


Figure 6. Water sorption isotherms of *Mentha aquatica* at different temperatures

The results for Wild mints are presented in Figure 7. The isotherms present hysteresis for all range of water activity. The calculated values of BET coefficients and coefficient of determination  $R^2$ , for isotherms of desorption of Wild mint are presented in Table 8, and for isotherms of adsorption in Table 9.



Table 6. BET equation parameters and goodness of fit of desorption of Water mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1190	39.8200	0.9591
30	0.0933	21.1305	0.9635
40	0.0871	9.6451	0.9823
50	0.0707	6.7584	0.9854
60	0.0555	2.7118	0.9978

Table 7. BET equation parameters and goodness of fit of absorption of Water mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1053	13.6487	0.9846
30	0.0856	8.0228	0.9815
40	0.0752	4.5277	0.9963
50	0.0632	3.2062	0.9970
60	0.0483	1.9916	0.9953

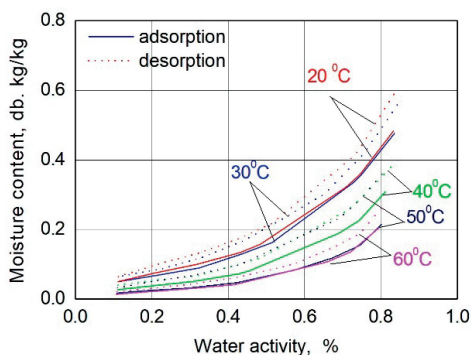


Figure 7. Water sorption isotherms of *Mentha arvensis* at different temperatures

Table 8. BET equation parameters and goodness of fit of desorption of Wild mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1293	39.8401	0.9292
30	0.0956	9.0228	0.9515
40	0.0848	8.6441	0.9431
50	0.0751	5.5277	0.9583
60	0.0556	3.7119	0.9779

Table 9. BET equation parameters and goodness of fit of absorption of Wild mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1083	13.6281	0.9641
30	0.0906	8.7580	0.9648
40	0.0753	4.5255	0.9921
50	0.0585	3.9913	0.9961
60	0.0499	1.9954	0.9777

The results for Horse mints are presented in Figure 8. The isotherms present hysteresis for all range of water activity. The calculated values of BET coefficients and coefficient of determination  $R^2$ , for isotherms of desorption of Horse mint are presented in Table 10, and for isotherms of adsorption in Table 11. The obtained values indicate that BET equation is suitable for describe isotherms of sorption for Horse mint.

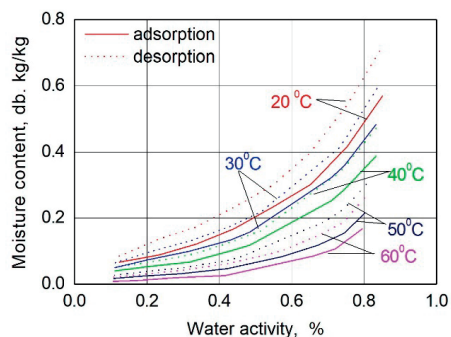


Figure 8. Water sorption isotherms of *Mentha longifolia* at different temperatures

Table 10. BET equation parameters and goodness of fit of desorption of Horse mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1194	38.8203	0.9576
30	0.1053	14.6487	0.9911
40	0.0885	9.6459	0.9872
50	0.0588	2.7119	0.9132
60	0.0477	0.9942	0.9747

Table 11. BET equation parameters and goodness of fit of absorption of Horse mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0939	20.1306	0.9456
30	0.0856	8.9228	0.9815
40	0.0716	6.7577	0.9748
50	0.0621	4.2062	0.9843
60	0.0488	2.1913	0.9641

The results for Apple mints are presented in Figure 9. The isotherms present hysteresis for all range of water activity for all analysed temperatures. The calculated values of BET coefficients and coefficient of determination  $R^2$ , for isotherms of desorption of Apple mint are presented in Table 12, and for isotherms of adsorption in Table 13. The obtained values

indicate that BET equation is suitable for describe isotherms of sorption for Apple mint.

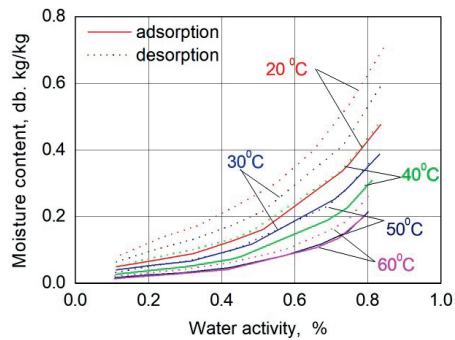


Figure 9. Water sorption isotherms of *Mentha suaveolens* at different temperatures

Table 12. BET equation parameters and goodness of fit of desorption of Apple mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1183	38.7721	0.9191
30	0.1086	13.9487	0.9346
40	0.0996	9.5243	0.9337
50	0.0756	4.5587	0.9873
60	0.0597	2.6134	0.9567

Table 13. BET equation parameters and goodness of fit of adsorption of Apple mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0911	8.0328	0.9445
30	0.0736	6.8151	0.9847
40	0.0649	3.7162	0.9650
50	0.0584	1.9913	0.9431
60	0.0444	1.8313	0.9851

The results for Pennyroyal mints are presented in Figure 10. The isotherms present hysteresis for all range of water activity for all analysed temperatures. The obtained isotherms are similar with isotherms obtained by Kane et al., (2008). The calculated values of BET coefficients and coefficient of determination  $R^2$ , for isotherms of desorption of Pepper mint are presented in Table 14, and for isotherms of adsorption in Table 15.

The obtained values indicate that BET equation is suitable for describe isotherms of sorption for Pennyroyal mint.

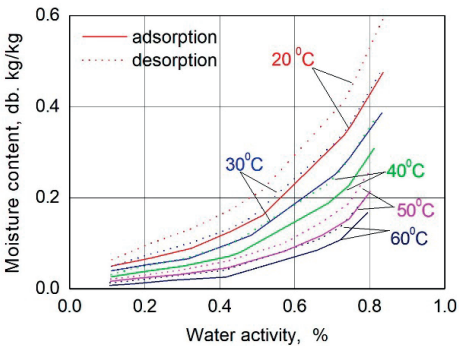


Figure 10. Water sorption isotherms of *Mentha pulegium* at different temperatures

Table 14. BET equation parameters and goodness of fit of desorption of Pennyroyal mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.1054	13.6487	0.9847
30	0.0878	9.6442	0.9838
40	0.0754	4.5277	0.9963
50	0.0553	2.7119	0.9976
60	0.0473	1.6954	0.9977

Table 15. BET equation parameters and goodness of fit of adsorption of Pennyroyal mint at different temperatures

Temperature (°C)	$X_m$	$C$	$R^2$
20	0.0858	8.0227	0.9816
30	0.0706	6.7580	0.9847
40	0.0633	3.2062	0.9971
50	0.0484	1.9912	0.9951
60	0.0438	0.9042	0.9974

The gap of hysteresis obtained in experiments differ from a mint species to another, but hysteresis are presented to all isotherms (adsorptions and desorptions) for all species of mint analysed.

## CONCLUSIONS

The water sorption isotherms of mints were determined using the gravimetric method based on the recommendation of the European Cooperative Project COST 90. Results of the experimental measurement of sorption isotherms (adsorption and desorption), at temperature range between 20 and 60°C, showed a typical sigmoid shape of type II according to the BET classification to all mint species analyzed. The temperature had the expected effect predicted by theory of physical adsorption, the quantity of sorped water at a given water activity increased as the temperature decreased. The quantity of sorpet water differ to one mint species to another. The amount of sorped water depends on the equilibrium temperature and mint species, maybe due to a different chemical composition. The increase of the temperature has as result the increase of the water activity for the same moisture content to all mints varieties. Hysteresis was found for all 7 mint species analysed: *Mentha piperita* L. (Peppermint), *Mentha spicata* L. (Spearment), *Mentha aquatica* L. (Water mint), *Mentha arvensis* L. (Wild mint), *Mentha longifolia* L./Huds (Horse mint), and *Mentha suaveolens* Ehrh (Apple mint), and *Mentha pulegium* L. (Pennyroyal mint); for the entire range of relative humidity, for both adsorption and desorption isotherms. Hysteresis loops decreased with an increase of temperature, but differ to one mint species to another. These indicate the irreversibility of sorption process and chemical and microbiological deterioration during adsorption or desorption of mints.

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