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Psychrometry as a Methodological Tool for Optimizing the Spray Drying Process

Eric Rondet, Gilles Baylac, and Bernard Bataille
Laboratoire de Génie des Procédés Eau et Bioproduits, Université Montpellier 1, Faculté de Pharmacie, Montpellier, France

Because psychrometry takes into account a great number of variables reflecting the quality of the drying air, it is an interesting tool to improve the control and the optimization of the spray drying process. In this article the authors study the evolution of the psychrometric variables according to the values taken by four inlet parameters (inlet air temperature, liquid flow rate, solid concentration of the spray dried liquid, and nature of the product). The results highlighted the existence of mathematical models making it possible to optimize the process, but also to underline the influence of the nature of the product on the drying mechanism.

Keywords psychrometry; spray drying; process optimization; maltodextrin; ascorbic acid

INTRODUCTION

By definition, spray drying is a one-step process that transforms a liquid into dry particles after it is dispersed as a spray in a hot medium (Masters, 1985). Spray drying gives the finished product very interesting functionalities, either for direct use (Luna-Solano, Salgado-Cervantes, Rodriguez-Jimenes, & Garcia-Alvarado, 2005; Narayan, Marchant, & Wheatley, 2001; Teunou & Poncelet, 2005) or for a later, more elaborate transformation (Hall, 2001; Sougnez, 2001). Present in all the sectors since the seventies, the initial optimization of spray drying was initially based on a global solution (Schuck, 2002). However, because of the number and the sophistication of the current uses, but also due to questions of law and quality constraints, researchers and industrialists are obliged to consider a more rigorous approach, based on process optimization. This is all the more true that a great number of parameters influence spray drying (Masters, 1985), and they can be grouped into three families: formulation, operational, or technological parameters.

The study we carried out aimed to improve the comprehension of the mechanism of drying within a spray drying column so as to be able to optimize the process in real time. Psychrometry constitutes the method of analysis on which we based our work. This methodology can be applied for the monitoring of all the generations of dehydratation technology (Vega-Mercado, 2001) provided that the movement of the air is higher than 0.5 m/s in the drying chamber. After a review of the fundamentals of psychrometry, the first part of the article will be dedicated to the development and the validation of the measuring apparatus. Then, we will see how psychrometry allows the control of the drying conditions and thus the quality of the end product. Lastly, we will see how and up to what point the product influences the quality of the air and thus the process of drying within the spray drying column.

Psychrometry: Fundamental Background

Psychrometry is the ideal tool for controlling the environment during the drying of a product by a hot medium, during the desiccation of humid air, or during the mixture of humid airs. This methodology can thus be used in various industrial areas: climatology, refrigeration (Mago & Sherif, 2003), food industry, and pharmacy, where it can be applied to any drying processes as long as the air movement is higher than 0.5 m/s. The dairy industry approached the relation between spray drying and psychrometry as a means of controlling its process (Jeantet, 2001). It is not, to our knowledge, the case in the pharmaceutical industry.

During a psychrometric analysis, the study of the air and its associated steam make it possible to access data characterizing the drying medium (Nguyen, 1992; Schallcross, 1997, 2005). A psychrometer is an apparatus made of two thermometric probes. One is used to measure the ambient temperature (dry bulb temperature) and the other to measure the wet bulb temperature. In practice, the latter corresponds to the temperature indicated by the wet thermometer surrounded by a porous material saturated with water. The wet bulb temperature makes

Address correspondence to Bernard Bataille, Laboratoire de Génie des Procédés Eau et Bioproduits, UMR CIRAD 016, Université Montpellier 1, Faculté de Pharmacie, 15 avenue Charles Flahault, B.P.14491, 34093 Montpellier Cedex 5, France. E-mail: bernard.bataille@univ-montp1.fr

it possible to know the precise temperature of the product within the drop (Nguyen, 1997). Thanks to the reading of the dry and the wet bulb temperature, it is possible, through the psychrometric diagram, to know the state of the humid air. Under atmospheric pressure, the psychometric diagram represents the variation of specific humidity according to the change of the dry bulb temperature. Its construction is based on the following equations:

$$P_v = P_{vsat} - (P_{atm} \cdot A \cdot (T_{db} - T_{hb})) \tag{1}$$

P_{vsat} can be calculated thanks to Rankine's relation:

$$P_{vsat} = \left(13.7 - \left(\frac{5120}{T_{hb}}\right)\right) \cdot P_{atm} \tag{2}$$

The psychrometric equation (1) makes it possible to know the specific humidity (H_{spe}) as well as saturated specific humidity (H_{spesat}) through the following equations:

$$H_{spe} = d\frac{P_v}{P_{atm} - P_v} \tag{3}$$

$$H_{spesat} = d\frac{P_{vsat}}{P_{atm} - P_{vsat}} \tag{4}$$

The knowledge of these two values will make it possible to have access to many variables among which are:

- Relative humidity of the drying air (Φ):

$$\Phi = \frac{H_{spe}}{d + H_{spe}} \cdot \frac{d + H_{spesat}}{H_{spesat}} \tag{5}$$

- Specific volume (υ_m):

$$\upsilon_m = \frac{RT_{db}}{P_{atm}} \cdot \left(\frac{1}{M_w} \cdot H_{spe} + \frac{1}{M_{da}}\right) \tag{6}$$

- Enthalpy (h):

$$h = C_{da} \cdot T_{db} + H_{spe} \cdot (C_w \cdot T_{db} + L_0) \tag{7}$$

The psychrometric probe gives us access to fundamental variables for the characterization of the drying air. The alteration in their value has drastic consequences on the final product. The enthalpy represents the energy available for the drying of the product. A diminution of this variable implies a fall in the efficiency of the drying that can lead to a drop in the yield. On the contrary, if the enthalpy is too high, it can lead to a degradation of the product, especially if it is thermosensitive. The effects of the alterations of the relative humidity in the drying column are well known. A drop in this value causes a diminution of the density of the product linked to a higher porosity generated by a shrinkage phenomenon (Goula & Adamopoulos, 2005b; Lin & Gentry, 1999; Masters, 1985). This also improves solubility and product recovery (Maury, Murphy, Kumar, Shi, & Lee, 2005). On the contrary, a higher relative humidity generates a powder with a higher density. But the main consequence of a high relative humidity is the beginning of the agglomeration process. The surface of the particles remains humid and they can coalesce into small agglomerates whose size is bigger than the size of the initial particle. These drying conditions leading to agglomeration are used for the synthesis of instant powders.

MATERIALS AND METHODS

Apparatus

The tests were carried out using a Büchi Mini Spray Dryer 190. This is standard equipment for spray drying laboratory experiments. The dimensions of the glass drying chamber is 47.5 cm high and its diameter is 10.5 cm. The compressed and drying airflows are fixed respectively at 600 Nl/h and 25.5 m^3/h . These values are compatible with an optimal sensitivity of the psychrometer.

Psychrometer

The psychrometer consists of two Pt-6 copper-constantan thermocouples (Physitemp Instrument). The first one is surrounded by a silicon pipe that protects it from the spray dried product and measures the dry bulb temperature. The second one is surrounded by a humidified gauze that measures the wet bulb temperature (Figure 1). Before each spray drying experiment, the gauze is saturated with distilled water by a syringe linked to the probe by a silicon pipe. This condition of saturation is obtained when water runs out of the gauze.

In the drying chamber, the probes are situated at 17 cm from the top of the spray drying column. Preliminary experiments proved that the sensitivity is maximal when they are located in this area. The probes are maintained by a metal ring preventing them from sticking to each other or to the walls and enabling them to keep an identical position in the transverse axis from one test to another.

The thermocouples are alternatively connected to the thermometer hygrometer Testo 625 for measurements. The resolution and the accuracy of the measurements are 0.1°C and ± 0.5°C, respectively.

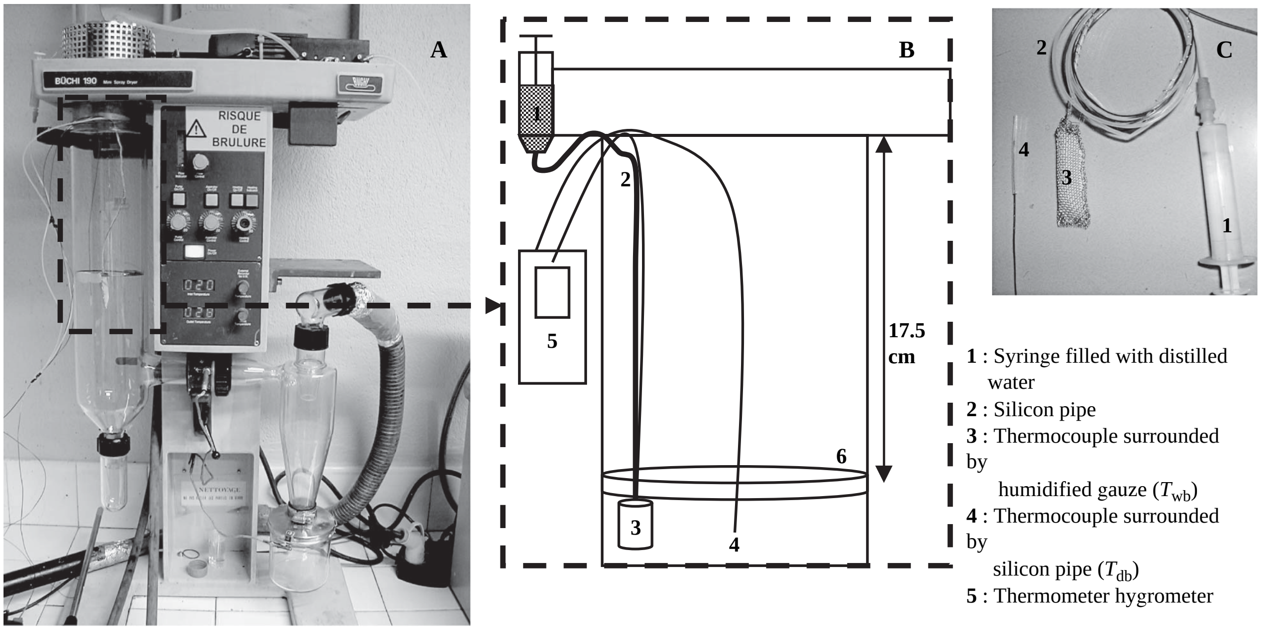

FIGURE 1. Location of the thermocouples in the spray drying column. A = general view of the Büchi Mini Spray Dryer, B = detailed view of the column, and C = the temperature probes and the syringe for humidifying the gauze around the humid bulb temperature.

Raw Materials

The raw material, in conformity with the specifications of the *European Pharmacopoeia, 5th edition*, will be spray dried after solubilization in distilled water. The first studied product is maltodextrin LYCATAB DSH® (Roquette et Frères, French batch no. 637630), a pharmaceutical excipient commonly used as spray drying support (Cano-Chauca, Stringheta, Ramos, & Cal-Vidal, 2005; Krishnan, Bhosale, & Singhal, 2005; Belghith, Ellouz Chaabouni, & Gargouni, 2001; Truong, Bhandar, & Howes, 2005). The second is L-ascorbic acid (vitamin C) fine powder (BASF A.G., German batch no. 060336V0). Unlike LYCATAB DSH®, the spray drying of this product is difficult and it requires spray drying support most of the time (Esposito, Cervellati, Menegatti, Nastruzzi, & Cortesi, 2002).

Methods

The methodology of our study is based on the two synoptics presented in Figures 2 and 3. The value of the specific humidity within a spray drying column is obviously dependent on the quantity of pulverized liquid, but also on the quantity of steam present in the drying air which is firstly sucked from the immediate environment of the spray drier then heated to reach the inlet temperature. The quantity of steam in the drying air varies

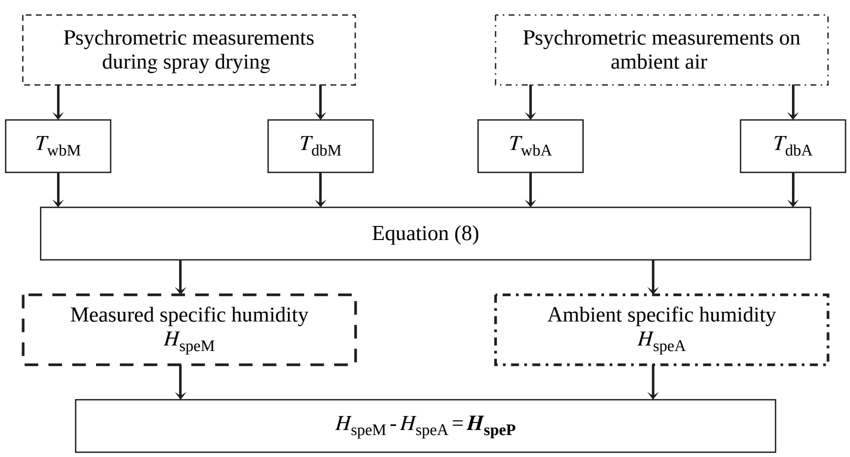

FIGURE 2. Synoptic of the different stages allowing the determination of the real specific humidity.

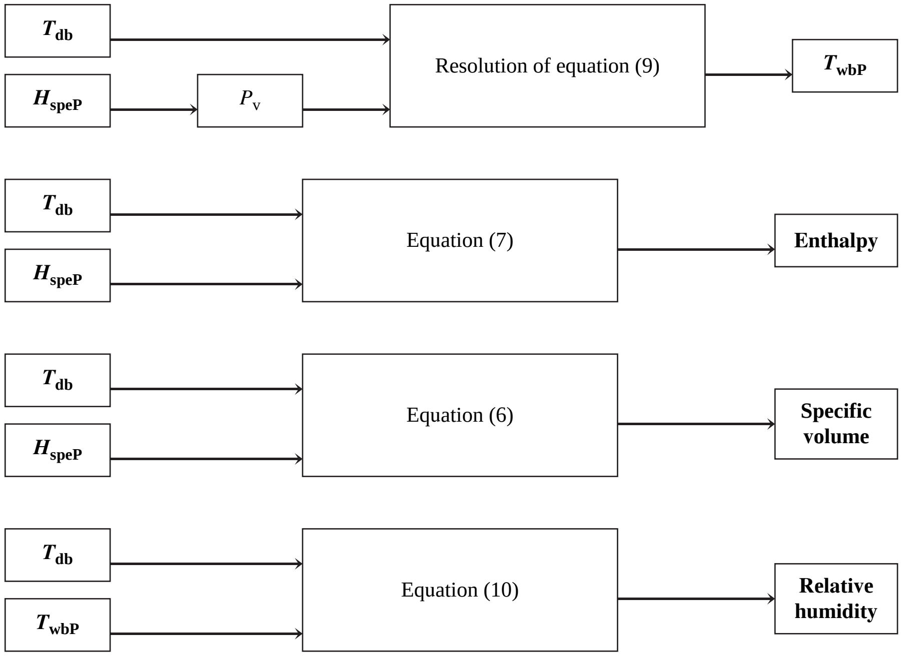

FIGURE 3. Synoptic of the program giving psychrometric data thanks to T_{db}, T_{hbP}, and H_{speP}.

daily according to climatic conditions. So as to correct these fluctuations, it is necessary to withdraw from measured specific humidity (H_{speM}) the specific humidity of the ambient air (H_{speA}). This result represents the real specific humidity (H_{speP}), which is the share of the humidity measured by the psychrometer due only to the evaporation of the liquid present in the spray dried solution.

Figure 2 describes how to obtain H_{speP}. For that, the wet bulb temperature and the dry bulb temperature must be measured either in the ambient air, or during spray drying, alternatively. The values of H_{speM} and H_{speA} are obtained while inserting the terms of Equation 1 and 2 into Equation 3:

$$H_{spe} = d \cdot \frac{\left(\exp\left(13.7 - \frac{5120}{273.15 + T_{wb}}\right) \cdot P_{atm} - P_{atm} \cdot A \cdot (T_{db} - T_{wb})\right)}{\left(P_{atm} - \exp\left(13.7 - \frac{5120}{273.15 + T_{wb}}\right) \cdot P_{atm} + P_{atm} \cdot A \cdot (T_{db} - T_{wb})\right)} \quad (8)$$

Due to the dry bulb temperature and $H_{speP,}$ it is necessary to recalculate all the psychometric data so as to know what the results would be if the relative humidity of the surrounded air was null. The synoptic presented in Figure 3 describes the calculation if it were in a completely dry atmosphere. According to Equation 3, it is possible to deduce P_v from H_{speP}. Then, it is possible to find the value of the wet bulb temperature corresponding to H_{speP} (T_{wbP}) starting from the Rankine's relation (Equation 2) and the psychrometric equation (Equation 1):

$$P_v + P_{atm} \cdot A \cdot (T_{db} - T_{wbP}) = \exp\left(13.7 - \frac{5120}{T_{wbP}}\right) \cdot P_{atm} \quad (9)$$

According to Equations 6 and 7, it is possible to calculate the enthalpy and the specific volume starting from the values of the dry bulb temperature and H_{speP}. Lastly, the relative humidity is obtained starting from Equation 1 and Rankine's relation:

$$\Phi = \frac{P_v}{P_{vsat}} = \frac{\left(\exp\left(13.7 - \frac{5120}{273.15 + T_{wb}}\right) \cdot P_{atm} - P_{atm} \cdot A \cdot (T_{db} - T_{wb})\right)}{\left(\exp\left(13.7 - \frac{5120}{273.15 + T_{wb}}\right) \cdot P_{atm}\right)} \quad (10)$$

The access to the psychrometric data makes it possible to study their evolution but also the relation to each other based on the variation of the parameters related to the spray drying process. Four of these inlet parameters will be studied: the nature of the product, its concentration, the inlet temperature, and the liquid flow rate. The analysis of the results is based on the experimental design presented in Table 1. The data pro-

TABLE 1
Experimental Design

Test No.	Product	Concentration w/w	Liquid Flow ml/min^{-1}	Inlet Temperature °C
1	Vitamin C	5	4	100
2	Vitamin C	5	4	120
3	Vitamin C	5	4	150
4	Vitamin C	5	8	100
5	Vitamin C	5	8	120
6	Vitamin C	5	8	150
7	Vitamin C	5	12	100
8	Vitamin C	5	12	120
9	Vitamin C	5	12	150
10	Vitamin C	15	4	100
11	Vitamin C	15	4	120
12	Vitamin C	15	4	150
13	Vitamin C	15	8	100
14	Vitamin C	15	8	120
15	Vitamin C	15	8	150
16	Vitamin C	15	12	100
17	Vitamin C	15	12	120
18	Vitamin C	15	12	150
19	Maltodextrin	5	4	100
20	Maltodextrin	5	4	120
21	Maltodextrin	5	4	150
22	Maltodextrin	5	8	100
23	Maltodextrin	5	8	120
24	Maltodextrin	5	8	150
25	Maltodextrin	5	12	100
26	Maltodextrin	5	12	120
27	Maltodextrin	5	12	150
28	Maltodextrin	15	4	100
29	Maltodextrin	15	4	120
30	Maltodextrin	15	4	150
31	Maltodextrin	15	8	100
32	Maltodextrin	15	8	120
33	Maltodextrin	15	8	150
34	Maltodextrin	15	12	100
35	Maltodextrin	15	12	120
36	Maltodextrin	15	12	150

Experimental design allowing the study of the influence of inlet parameters on the psychrometric data.

cessing was carried out using the software Statgraphics Plus® (Sigma Plus).

RESULTS AND DISCUSSION

Psychrometer Validation

The first phase of validation of the psychrometer aimed to check the adequacy of the results obtained compared with those resulting from other methods of measurements.

Various studies (Goula & Adamopoulos, 2005a and 2005b; Luna-Solano et al., 2005) show that a reduction in the outlet temperature of the drying air involves a rise in the relative humidity within the column. As it is shown in Figure 4, the correlation between the outlet temperature and the relative humidity within the column again makes it possible to observe this phenomenon. Psychrometric measurements are thus in conformity with the results admitted in spray drying.

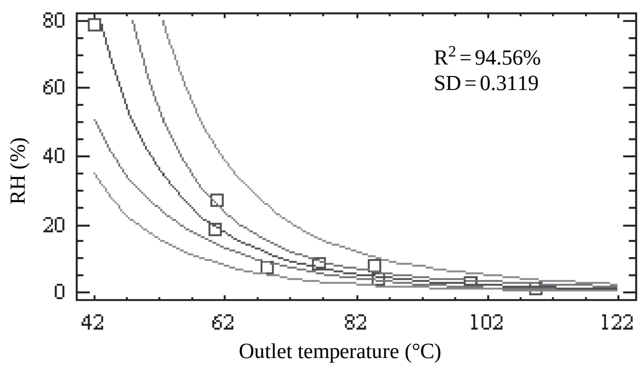

FIGURE 4. Statistical model of regression between outlet temperature and relative humidity in the column.

The study of sensitivity and reproducibility of psychrometric measurements are based on the answer provided by the sensor during the variation in the inlet temperature and the liquid flow rate (which in this case is distilled water). The results of these tests appearing in Table 2 are then quoted in the psychrometric diagram of Figure 5 representing:

- In x-coordinate: the dry bulb temperature (°C)
- In ordinate: the specific humidity in kg of water/kg of dry air

It is moreover composed of four types of curves:

- Isotherms (straight lines perpendicular to the x-axis)
- Isenthalpes (oblique straight lines in full feature)
- Isochores (oblique dotted straight lines)
- Relative humidity curves

Under these experimental conditions, all these results highlight the sensibility and the reproducibility of the psychrometric analysis. Any change in the value of one of the inlet parameters involves indeed a visible modification in the location of the experimental data on the psychrometric diagram. On the contrary, when inlet parameters remain constant from one test to another, the experimental results show a great repro-

TABLE 2
Validation of the Psychrometric Probes

Liquid Flow ml/min^{-1}	Inlet Temperature °C	Dry Bulb Temperature °C	Wet Bulb Temperature °C	RH %	Enthalpy kJ/kg^{-1}	Specific Volume m^3/kg^{-1}
4.0	100	65.1	31.1	9.2	100.6	0.971
4.0	100	65.2	31.6	9.8	103.3	0.973
4.0	100	64.9	31.4	9.8	102.2	0.972
4.0	120	85.9	36.8	5.1	132.1	1.037
4.0	120	85.3	37.2	5.5	134.9	1.037
4.0	120	86.0	36.8	5.6	132.2	1.037
4.0	150	115.8	40.4	1.3	154.2	1.118
4.0	150	116.4	41.3	1.7	161.3	1.124
4.0	150	115.3	41.3	1.8	161.5	1.121
8.0	100	53.0	31.2	22.0	102.3	0.945
8.0	100	53.7	31.2	21.0	102.3	0.946
8.0	100	53.8	31.2	20.8	102.3	0.946
8.0	120	75.1	37.1	10.0	135.5	1.015
8.0	120	74.4	36.8	10.1	135.6	1.012
8.0	120	74.0	36.8	10.8	135.5	1.011
8.0	150	103.9	40.9	3.2	159.9	1.095
8.0	150	102.5	40.8	3.4	159.3	1.091
8.0	150	102.6	40.9	3.4	160.1	1.092
12.0	100	32.0	31.6	97.2	106.6	0.898
12.0	100	31.5	31.4	99.3	105.6	0.896
12.0	100	31.7	31.1	95.8	103.9	0.896
12.0	120	52.9	35.9	34.0	130.1	0.960
12.0	120	53.6	36.0	32.8	130.7	0.962
12.0	120	54.0	36.1	32.3	131.3	0.963
12.0	150	83.9	40.4	8.6	158.7	1.049
12.0	150	82.2	40.5	9.5	159.7	1.046
12.0	150	82.8	40.3	9.0	158.1	1.046

Results of psychrometric measurements for various inlet temperatures and liquid flow rate of distilled water (n = 3).

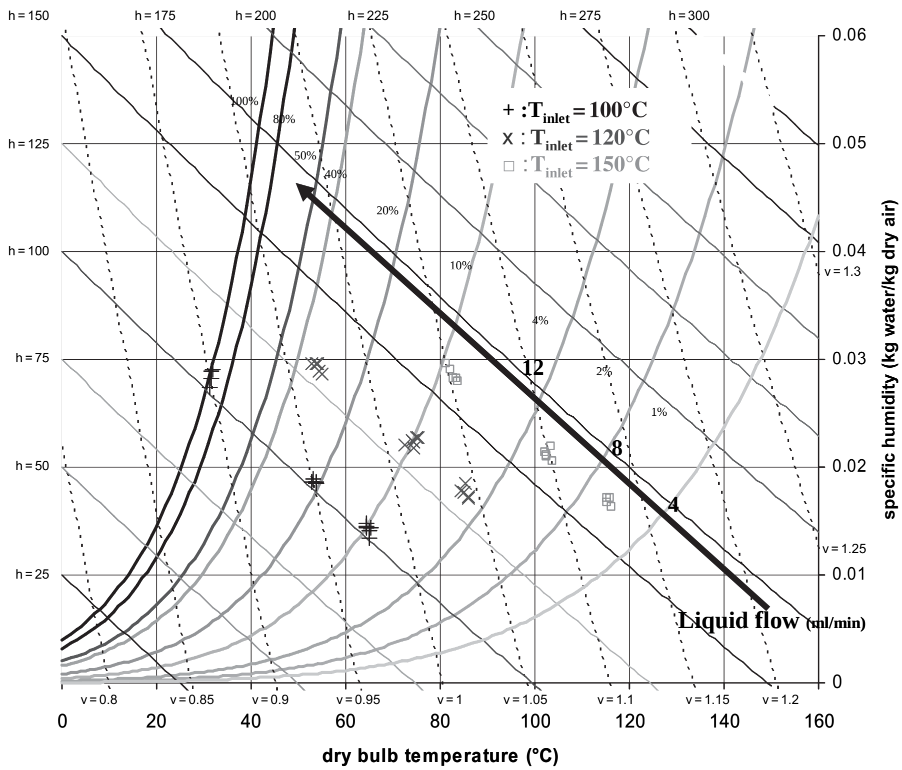

FIGURE 5. Results of the spray drying of distilled water (for three inlet temperatures [100, 120, and 150°C] and three liquid flow rates [4, 8, and 12 ml/min^{-1}]) plotted on the psychrometric diagram.

ducibility. This experimental setup can thus be used as a reliable tool to study the variation of the psychrometric variables based on the inlet parameters.

Experimental Design

From the measurements carried out by the sensor, it is possible to determine all the psychrometric data. Due to this knowledge, it is possible to optimize the process in real time by controlling the processus related to the quality of the air, the mass balances, and the influence of the nature of the products to be spray dried.

Control of Processus Related to the Quality of the Air

Relation Between Temperature and Relative Humidity. Measured using the psychrometer, the differential of temperature (ΔT) expresses the difference between the dry bulb temperature (T_{db}) and the wet bulb temperature (T_{wb}). Results, consigned in Table 3 and Figure 6, and their statistical analysis allow the establishment of a model connecting the relative humidity to ΔT according to the following equation:

$$\Phi = \exp(4.226 - 0.050\Delta T) \quad (11)$$

The relative humidity is a fundamental value for the control of the unit operations using air as a drying agent. Indeed, it is its value that will make it possible to know the direction and the intensity of the exchanges of water between the air and the product. Its control in the column is directly dependent on the value of ΔT.

Relation Between Wet Bulb Temperature (T_{wb}) and Residual Enthalpy. The residual enthalpy corresponds to the furnished energy for which it is necessary to withdraw absorbed energy. Its value is measured directly by the psychrometer. The results are presented in Table 4 and Figure 7. Their statistical analysis

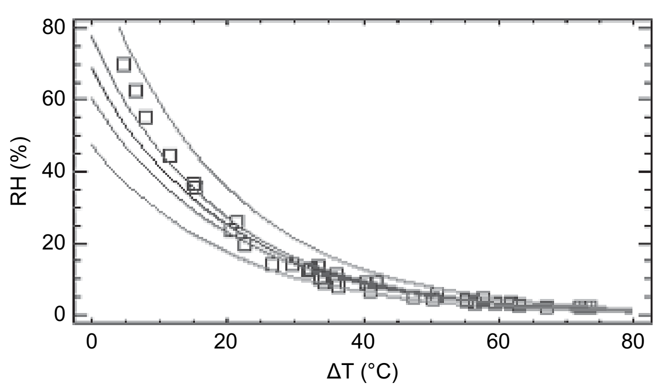

FIGURE 6. Statistical model of regression between ΔT and the relative humidity within the column.

TABLE 3
Evolution of the Relative Humidity with ΔT

Product	Concentration w/w	Liquid Flow ml/min^{-1}	Inlet Temperature °C	ΔT °C	RH %
Vitamin C	5	4	100	33.7	10.4
Vitamin C	5	4	120	40.6	8.8
Vitamin C	5	4	150	71.9	2.2
Vitamin C	5	8	100	26.6	14.1
Vitamin C	5	8	120	41.1	7.1
Vitamin C	5	8	150	62.8	2.8
Vitamin C	5	12	100	4.9	69.7
Vitamin C	5	12	120	35.9	9.4
Vitamin C	5	12	150	51.0	5.7
Vitamin C	15	4	100	31.9	12.4
Vitamin C	15	4	120	56.5	3.6
Vitamin C	15	4	150	73.4	2.2
Vitamin C	15	8	100	32.2	12.8
Vitamin C	15	8	120	21.3	25.8
Vitamin C	15	8	150	61.7	3.4
Vitamin C	15	12	100	11.4	44.4
Vitamin C	15	12	120	29.7	14.0
Vitamin C	15	12	150	59.4	3.6
Maltodextrin	5	4	100	34.2	9.0
Maltodextrin	5	4	120	47.5	4.9
Maltodextrin	5	4	150	67.1	2.3
Maltodextrin	5	8	100	20.5	23.8
Maltodextrin	5	8	120	34.4	10.8
Maltodextrin	5	8	150	55.3	4.1
Maltodextrin	5	12	100	8.1	54.9
Maltodextrin	5	12	120	15.3	35.7
Maltodextrin	5	12	150	33.4	13.4
Maltodextrin	15	4	100	36.2	8.1
Maltodextrin	15	4	120	50.3	4.5
Maltodextrin	15	4	150	72.8	2.3
Maltodextrin	15	8	100	22.7	19.9
Maltodextrin	15	8	120	36.1	11.2
Maltodextrin	15	8	150	57.8	4.5
Maltodextrin	15	12	100	6.5	62.7
Maltodextrin	15	12	120	15.1	36.9
Maltodextrin	15	12	150	41.9	9.1

Results of psychrometric measurements after the spray drying of vitamin C and maltodextrin for three inlet temperatures (100, 120, and 150°C), two concentrations (5 and 15 w/w), and three liquid flow rates (4, 8, and 12 ml/min^{-1}).

led to a model connecting T_{wb} and h according to the following equation:

$$h = \exp\,(3.179 + 0.047T_{wb}) \qquad (12)$$

This model is totally independent of the inlet variables (inlet temperature of the drying air, concentration of the product in the spray dried liquid, liquid flow rate, and the nature of the spray dried product). The measurements of the wet bulb temperature allow the calculation of the available energy in the area of the sensor. As it was mentioned earlier, the residual enthalpy represents the energy available for the drying of the product. If, in the area of the sensor, the residual energy is not sufficient for the drying to follow up, the product will keep its residual moisture content. Two possibilities can be considered:

TABLE 4
Evolution Enthalpy with the Wet Bulb Temperature

Product	Concentration w/w	Liquid Flow ml/min^{-1}	Inlet Temperature °C	Wet Bulb Temperature °C	Enthalpy kJ/kg^{-1}
Vitamin C	5	4	100	33.3	112.2
Vitamin C	5	4	120	38.5	144.7
Vitamin C	5	4	150	43.7	182.0
Vitamin C	5	8	100	29.3	92.5
Vitamin C	5	8	120	35.1	122.5
Vitamin C	5	8	150	40.5	156.5
Vitamin C	5	12	100	28.9	92.2
Vitamin C	5	12	120	34.0	116.2
Vitamin C	5	12	150	41.3	164.6
Vitamin C	15	4	100	34.4	119.2
Vitamin C	15	4	120	39.2	148.1
Vitamin C	15	4	150	44.4	188.4
Vitamin C	15	8	100	35.3	124.5
Vitamin C	15	8	120	36.0	130.4
Vitamin C	15	8	150	42.1	170.0
Vitamin C	15	12	100	30.3	98.9
Vitamin C	15	12	120	33.8	115.6
Vitamin C	15	12	150	41.3	163.2
Maltodextrin	5	4	100	31.5	102.6
Maltodextrin	5	4	120	35.9	126.5
Maltodextrin	5	4	150	41.0	160.2
Maltodextrin	5	8	100	31.1	101.8
Maltodextrin	5	8	120	34.9	121.5
Maltodextrin	5	8	150	40.1	154.4
Maltodextrin	5	12	100	28.3	89.4
Maltodextrin	5	12	120	33.2	113.6
Maltodextrin	5	12	150	38.5	146.1
Maltodextrin	15	4	100	32.1	105.6
Maltodextrin	15	4	120	37.2	134.4
Maltodextrin	15	4	150	44.5	189.0
Maltodextrin	15	8	100	30.5	98.8
Maltodextrin	15	8	120	37.8	140.5
Maltodextrin	15	8	150	43.7	183.9
Maltodextrin	15	12	100	29.9	97.1
Maltodextrin	15	12	120	34.2	119.6
Maltodextrin	15	12	150	40.7	161.5

Results of psychrometric measurements after spray drying of vitamin C and maltodextrin for three inlet temperatures (100, 120, 150°C), two concentration (5 and 15 w/w), and three liquid flow rates (4, 8, and 12 ml/min^{-1}).

- If this moisture content is higher than that required for the end product, the yield will decrease by the removal of the amount of product that does not correspond to the established specifications
- If this moisture content is equal to or lower than that required for the end product, the lower part of the chamber won't be useful for the elaboration of the final product

Thus, psychrometry becomes an interesting tool for the optimization of the yields, but also for the dimensioning of the drying chamber.

The aim of this study is to work out a framework for the use of psychrometry in spray drying of pharmaceutical products. The models mentioned above, taking into account most of spray drying inlet parameters, must necessarily be supplemented by the study of the influence of the other parameters

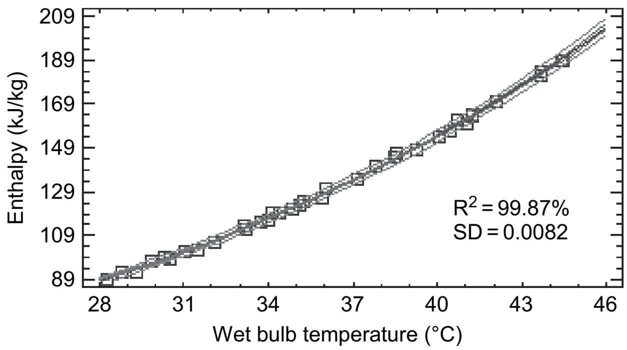

FIGURE 7. Statistical model of regression between the enthalpy and the humid bulb temperature.

(compressed and drying air flow) on the psychrometric variables. Then, after scale up, these models could be use for bulk production dryers.

Control of Mass Balance Related to the Nature of the Product

In order to study the influence of the operational parameters on the effectiveness of drying during spray drying, H_{speP} is compared with the theoretical maximum specific humidity (H_{spemax}). The latter depends on the operational parameters (inlet temperature, liquid flow rate, and concentration of the spray dried product) and corresponds to the specific humidity that would be measured by the sensor after total evaporation of water. The tests were led on aqueous solutions of maltodextrin and vitamin C to 15 w/w. The results presented in Figures 8 and 9, respectively, highlight that, for liquid flows of 4 ml/min^{-1} and 8 ml/min^{-1}, the quantity of water measured by the sensor is higher than that related to the total evaporation of water. In order to consolidate the reality of these results, the same experiments were conducted only on the spray drying of distilled water (Figure 10) for values of liquid flow rate equal to 5, 7.5, and 12 ml/min^{-1} and inlet temperature of 100, 120, 150 °C. The results obtained show that a rise in the temperature involves an increase in specific humidity within the column and that H_{speP} is all the more close to H_{spemax} when the flow is low or the inlet temperature is high. However, at any time, the quantity of steam measured by the sensor is higher than the theoretical maximum quantity.

The phenomenon highlighted during spray drying of product can thus only be explained physically by one phenomenon of accumulation of steam within the spray drying column. This phenomenon of accumulation, appearing only when product is spray dried, is due in part to the creation of turbulences related to the presence of particles moving in the drying chamber (Lo, 2005). These turbulences involve a recirculation of the air, and thus of the steam, which is new once taken into account by the sensor. Nevertheless, one part of the steam accumulation can also be related to adhesion of humid particles on the walls of the drying chamber. Actually, the stuck product will continue to dry and the steam that will evaporate from it will constitute an additional water mass that will be measured by the psychrometer. In this case, it will be shown that accumulation is strongly influenced by the nature of the product to be spray dried.

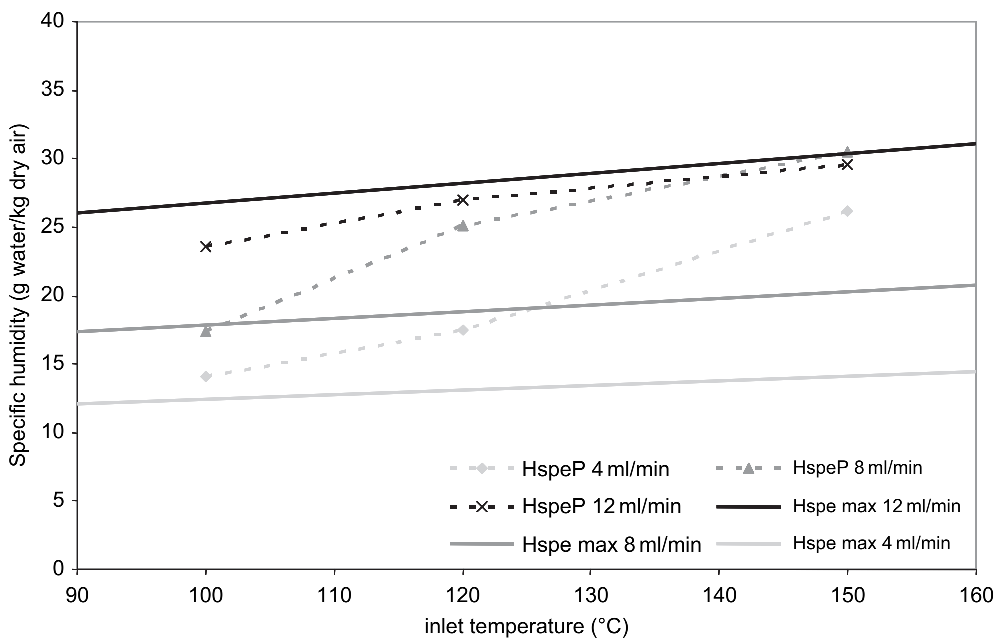

FIGURE 8. Comparison between maximum specific humidity and the real specific humidity at different liquid flow rates for maltodextrin 15 w/w.

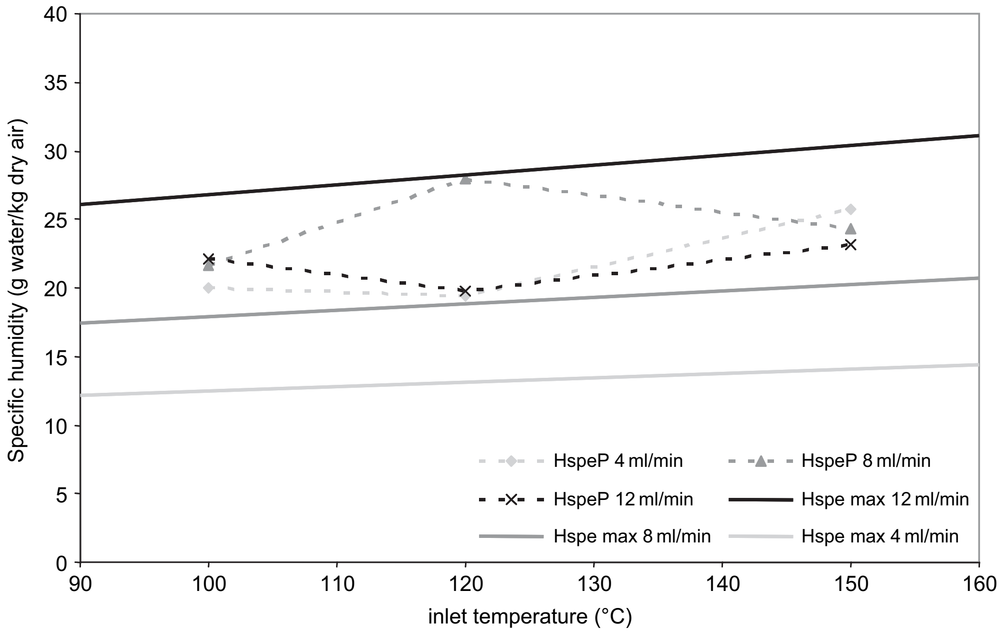

FIGURE 9. Comparison between maximum specific humidity and the real specific humidity at different liquid flow rates for vitamin C 15 w/w.

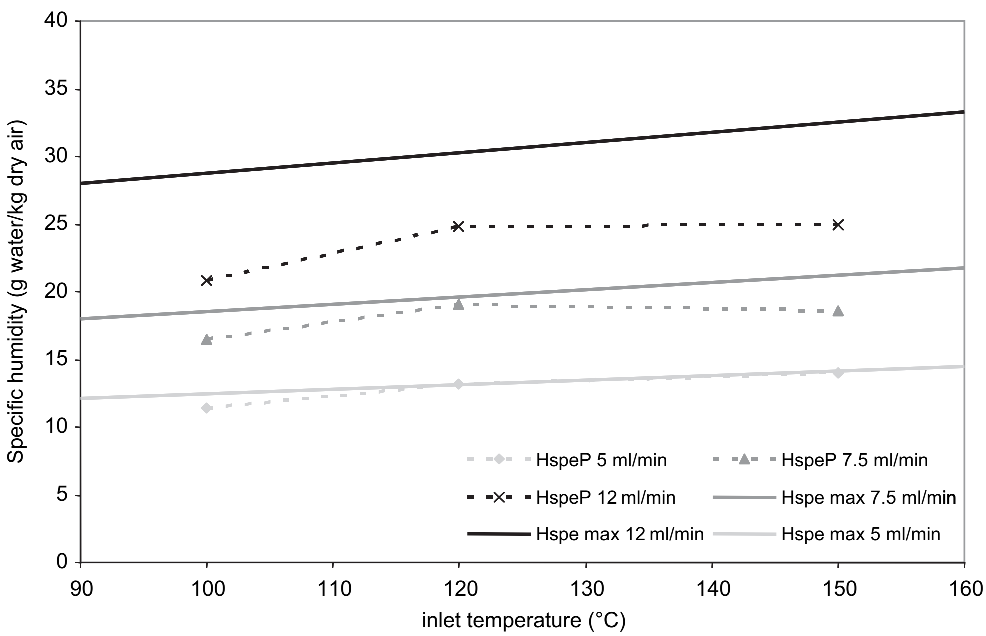

FIGURE 10. Comparison between maximum specific humidity and the real specific humidity at different liquid flow rate for distilled water.

A precise measurement of the quantity of the accumulated steam can be obtained by withdrawing the maximum quantity of theoretical steam (H_{spemax}) from H_{speP}. The incorporation of this variable in the experimental design makes it possible, after analysis, to study the interactions between two different factors (product and liquid flows) on the value of this accumulation. As is it shown in the Figure 11, the quantity of accumulated steam and the influence of the liquid flow rate on the accumulation is not the same from one product to another. For low liquid flow rate (4 ml/min^{-1}), the value of accumulation of steam for the spray drying of vitamin C is higher than that measured for the spray drying of maltodextrin. Nevertheless, an increase in this inlet parameter involves a less marked drop of the value of

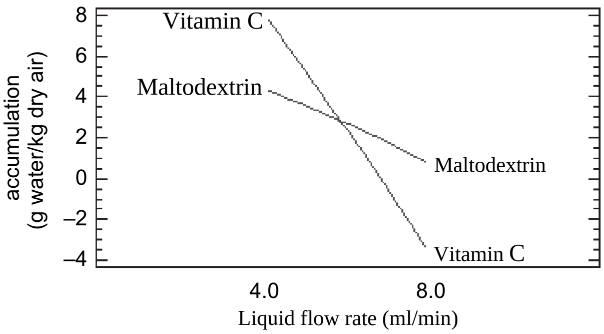

FIGURE 11. Evolution of the phenomenon of accumulation of steam according to the liquid flow rate and the nature of the spray dried product.

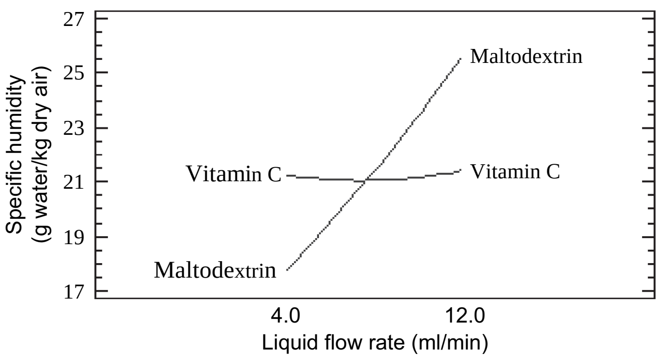

FIGURE 12. Evolution of the specific humidity according to the liquid flow rate and the nature of the spray dried product.

accumulation in the case of maltodextrin. The psychrometric diagram of Figure 5 already showed that an increase in the liquid flow rate always involves a drop in the dry bulb temperature in the chamber. Thus the steam accumulation related to the adhesion of the product to the wall of the drying chamber walls would be influenced by any modification in the dry bulb temperature. This influence is much more marked for vitamin C than for maltodextrin. The difference between the two products should be due to theirmicrostructure: vitamin C is crystalline whereas maltodextrin is partly an amorphous polymer whose glass transition temperature (220 °C) limits wall adhesion.

Concerning the influence of the liquid flow rate on the variation of the specific humidity, the results are quoted on figure 12. They highlight two different drying behaviors for the maltodextrin and the vitamin C, and thus confirm the fundamental role of the nature of the product to be spray dried on its aptitude of undergoing the spray drying process. Theoretically, the specific humidity measured by the sensor must increase with the liquid flow rate. This is the case for the experiments performed on maltodextrin, but with the vitamin C, the specific humidity in the column remains constant whatever the flow. This phenomenon can be explained if these results are coupled with those of Figure 10. For vitamin C, the accumulation of steam strongly decreases when the liquid flow rate increases. This fall in steam accumulation, associated with the increase in the quantity of steam incorporated in the system when the liquid flow rate increases, involves the maintenance of a stable specific humidity, whatever the flow. These results highlight the fact that the nature of the product drastically modifies the quality of the drying medium and show that their influence can modify the control of the process.

CONCLUSION

In order to optimize a spray drying process, it is necessary to closely control the drying processus in the spray drying column. This requires the knowledge of the quality of the drying air whatever the value taken by the operational parameters. Psychrometry constitutes a methodological tool, making it possible to attain this knowledge.

After validation of the sensor allowing the study of the evolution of the psychrometric data, the provided results made it possible to generate a model allowing a better control of the quality of the drying air, and thus of the final product, during spray drying. The relative humidity within the column as well as the enthalpy can be controlled by the determination of the differential of temperature ΔT and the wet bulb temperature.

The analysis of the processus related to the drying during spray drying made it possible to highlight a phenomenon of steam accumulation within the column. This phenomenon is closely related to the nature of the spray dried product. In fact, it is the whole spray drying process which seems to be largely dependent on the nature of the studied product since it interacts with the evolution of specific humidity and the dry bulb temperature according to the liquid flow rate.

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NOMENCLATURE

Constants

A: psychrometric constant—$6.66 \cdot 10^{-4}$ K^{-1} for T > 0°C ; $5.94 \cdot 10^{-4}$ K^{-1} for T < 0°C
d: density of the steam compared to the air—d = 0.62
C_{da}: heat-storage capacity of dry air. $C_{da} = 1.01$ kJ · kg^{-1} · K^{-1}
C_w: heat-storage capacity of steam. $C_w = 1.93$ kJ · kg^{-1} · K^{-1}
L_0: latent heat of vaporization of water with 0°K. $L_0 = 2{,}501$ kJ · kg^{-1}
P_{atm}: atmospheric pressure. $P_{atm} = 101.3$ kPa
R: 8.3145 J · mol^{-1} · K^{-1}
M_w: molar mass of water. $M_w = 0.018$ kg · mol^{-1}
M_{da}: molar mass of dry air. $M_{da} = 0.029$ kg · mol^{-1}

Variables

P_v: steam pressure kPa
P_{vsat}: saturating steam pressure kPa
T_{db}: dry bulb temperature °K
T_{wb}: wet bulb temperature °K
T_{wbP}: wet bulb temperature calculated for RH = 0%, °K
H_{spe}: specific humidity, kg water/kg dry air
H_{spesat}: specific humidity to saturation, kg water/kg dry air
H_{speM}: measured specific humidity, kg water/kg dry air
H_{speA}: ambient specific humidity, kg water/kg dry air
H_{speP}: real specific humidity, kg water/kg dry air
H_{spemax}: maximal specific humidity, kg water/kg dry air
Φ: relative humidity %
ν_m: specific volume m^3/kg
h: enthalpy kJ/kg

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