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# Effects of pretreatments on convective drying of rosehip *(Rosa eglanteria)*

Alejandra Mabellini[a,c] , Elizabeth Ohaco[a] , Carlos A. Márquez[a] , Antonio De Michelis[b,c,*] , and Jorge E. Lozano [c,d]

[a]Facultad de Ciencia y Tecnología de los Alimentos (Universidad Nacional del Comahue), 25 de Mayo y Reconquista (8336) Villa Regina, Rió Negro, Argentina.
[b] INTA AER El Bolsón, Mármol 1950 (8430) El Bolsón, Río Negro. Argentina.
[c] CONICET-CCT Comahue Quintral 1250 (8400), San Carlos de Bariloche, Río Negro. Argentina
[d]PLAPIQUI (Planta Piloto de Ingeniería Qumica), Camino "La Carrindanga", Km 7, C.C. 717 (8000), Bahía Blanca, Argentina
[*]ademichelis@bariloche.inta.gov.ar

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**Abstract**

he aim of this work was to experimentally determine drying curves for thin layer and bed drying of rosehip fruits, with and without pretreatments, to reduce processing times as a function of drying air operating variables, to propose dehydration kinetics of fruits and to determine its kinetic parameters for further use within drying simulation software. Fruits were pre-treated both chemically and mechanically, which included dipping the fruits in NaOH and ethyl oleate solutions; and cutting or perforating the fruit cuticle, respectively. Simulation models were then adopted to fit the kinetics drying data considering fruit volume shrinkage. These simple models minimized the calculation time during the simulation of deep-bed driers. Results show that pre-treatments reduced processing times up to 57%, and evaluated models satisfactorily predicted the drying of rosehip fruit. Effective mass diffusion coefficients were up to 4-fold greater when fruit was submitted to mechanical pretreatments.

***Keywords:*** Rosehip; drying; pretreatments; effective diffusion coefficients process times

## 1 Introduction

Scientific interest in rosehip fruit has exponentially increased recently due to its high content of vitamin C (Caro, Kesseler, & De Michelis, 2009; Pirone, Ochoa, Kesseler, & De Michelis, 2002, 2007; Mabellini et al., 2009; Ohaco, Pirone, Ochoa, Kesseler, & De Michelis, 2001), carotenoids (vitamin A precursors) (Ohaco et al., 2005), minerals and essential oils. These nutrients are considered very important in the food industry, in medicine and cosmetology. Rosehip also has important potential for agro industries in Argentina. It was introduced many years ago in Argentina and Chile, and its production covers important areas mainly in the Valleys area of south and central Andes of both countries. This pseudo fruit is harvested between March and June. Only processed and conserved fruits are available after that harvesting season.

Heated air convective dehydration appears to be the most viable way to process rosehip (*Rosa eglanteria*) fruit in the mentioned areas. Dehydration of foods, especially fruits, is a very old international tradition. The dried fruits are widely used as ingredients in processed foods, as confectionery, dried soups, ice creams and powders for making juices, fruit infusions, etc. (Barta, 2006). The marketing of fruits of the rosehip (*Rosa eglanteria*), harvested in central and south-

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ern Argentina and Chile, has continuously growth during these last years. Opportunities include the high demand for the dried products on the international market (Márquez, 2003).

The quality of any dehydrated product, of vegetable or animal origin, is directly related to the operative drying conditions. At present, conventional hot air drying of fruits and vegetables is performed quickly, and at temperatures as low as possible, to minimize energy consumption and thermal degradation of nutritional components and other attributes of quality. In order to increase the drying rate of fruits with non-permeate skins, different types of pretreatments (both physical and chemical) are used. The aim of these pre-treatments is to totally or partially remove the non-permeate cuticle, in order to improve water diffusion and reduce the time of processing (Gambella, Piga, Agabbio, Vacca, & D'hallewin, 2000; Erenturk, Gulaboglu, & Gultekin, 2005; Doymaz, 2007; Tarhan, 2007; Jazini & Hatamipour, 2010; Doymaz & Ismail, 2011). Chemical treatments consist of immersing the fruit in aqueous solutions of NaOH, KOH or alkaline ethyl oleate at different temperatures for a certain time, which normally produces a break in the cuticle of the fruit creating microscopic pores that facilitate permeability to moisture. Emulsions of fatty acid esters have long been used as a pretreatment before drying (Petrucci, Canata, Bolin, Fuller, & Stafford, 1973; Doymaz & Ismail, 2011). Immersion of grapes in an alkaline solution of ethyl oleate produces the solubilization of the wax, forming micro pores in the cuticle together with a non-uniform redistribution of components of wax on the fruit surface (Di Matteo, Cinquanta, Galiero, & Crescitelli, 2000).

Other commonly used solutions as a pretreatment before drying of grapes and olives are NaOH or KOH. Physical treatments are based on producing some kind of mechanical damage to the skin of the fruit, fracturing the non-permeable layer and facilitating the flow of water through the surface of the fruit. The method of skin abrasion is one of the most studied physical pretreatments (Di Matteo et al., 2000), but this pretreatment is very difficult to apply to rosehip fruits and little information about superficial cuts and slightly deeper perforations with needles of small diameter is available (Azoubel & Murr, 2003; Grabowski & Marcotte, 2003). Different authors have reported that reductions of drying times for fruits with mechanical pre-treatments range between 15% and 40%. On the other hand, modern methods for design of food dryers are based on the mathematical description of dehydration in beds to estimate drying time as accurately as possible (Giner, 1999; Márquez, De Michelis, & Giner, 2006).

According to the literature, a process as complex as dehydration in deep beds can be analyzed by decomposing it in simpler systems, i.e., drying in deep beds can be evaluated by considering several small beds of height equivalent to a particle diameter (Himmelblau & Bischoff, 1976; Giner, 1999; Ratti, 1991; Márquez et al., 2006). Therefore, the determination of the intrinsic drying properties such as thin layers kinetic parameters becomes an important issue as far as industrial dryer design is concerned. Concerning the thin layer drying problem, numerous studies are available in the literature. They can be classified into three types of solutions: numerical, analytical and approximated. In turn, within the last, semi-empirical and empirical solutions can be distinguished. Moreover, in each category, some contributions take into account product shrinkage. In general, isothermal drying appears as the most common model assumption to solve the variation of dimensionless moisture as a function of time for different air operating conditions: temperature, velocity and relative humidity.

However, in many contributions, only the dry bulb temperature of air drying was varied. It is evident that the complexity inherent to the analysis of drying processes lies in the diversity of biological materials and their shrinkage, so it is very difficult to find a general model. There are several possibilities to model thin layer drying with many different degrees of complexity. As demonstrated by some authors (Giner, 1999; Márquez et al., 2006), kinetic parameters vary substantially according to the method used to evaluate them, and even those obtained by the same method are often dependent on the equilibrium water content used to express the experimental data in dimensionless form (Márquez et al., 2006).

If the objective of the work is to provide the information necessary to simulate food particle beds, an important issue is to find thin layer drying models with good physical background, yet fast to run on the computer to facilitate interactive use, which is essential for equipment design. The thin layer drying equation constitutes the so called "product model", or constitutive equation for mass transfer in individual particles. This equation is useful in two main respects: It permits a study of the way a theory (represented by the equation) can adapt to the drying data of a given food and once the soundness of a theory is verified, it can be used to determine kinetic parameters in operating conditions usual in the drying practice, and then applied within deep bed models, where both product and air conditions vary with space and time, to predict temperature and moisture profiles and calculate drying times for equipment simulation and design.

The aim of this work was therefore to experimentally determine drying curves for thin layer and bed drying of rosehip fruits, with and without pretreatments (with the purpose of reducing processing times and increasing the productivity of industrial driers), as a function of drying air operating variables and to experimentally determine rosehip fruits dehydration kinetics parameters for further use in a dryers simulation model.

## 1.1 Modelling Considerations

Given that dehydration is a coupled phenomenon of heat and mass transfer, it would be necessary to simultaneously solve mass and energy balances, to evaluate dehydration kinetics. However, the literature has shown that as the rate of relaxation of the heat transfer potential is thousands of times faster than that for mass transfer, the temperature profile inside the food can be considered flat, especially if compared with the steep moisture content gradient (Márquez et al., 2006). On the other hand the temperature profile inside the food can be considered flat, especially if compared with the steep water content gradient (Giner & Mascheroni, 2001). In this regard, experiments were carried out to follow temperature variations inside the particle under a range of drying air operating conditions.

In a previous paper, Márquez et al. (2006) found that during rosehip fruit drying, particle temperature rapidly approaches the drying air temperature. So, a possible assumption is to consider a flat temperature profile inside the particles. In turn, in view of the heating rate of fruits, their average temperature becomes very similar to that for air, so this also complies with the isothermal drying assumption. Giner (1999) as well as other researchers (Parry, 1985; Márquez et al., 2006) analyzed the ratio of thermal to mass diffusivities inside the solid as a criterion to guide drying modeling, indicating that a large ratio would suggest an "instant" heat transport, as compared with mass transport. Thermal diffusivity of rosehip fruits varies between $1.96 \times 10^{-7}$ and $2.009 \times 10^{-7}$ m$^2$/s (Márquez, 2003), while mass diffusivities the effective diffusion coefficient in solids according to Zogzas, Maroulis, and Marinos-Kouris (1996) - lie between $10^{-10}$ and $11^{-11}$ m$^2$/s in most foods. Considering the values published by Zogzas et al. (1996), including more than 100 diffusion coefficients from 61 foods with diverse water contents, an average value of $1.45 \times 10^{-10}$ m$^2$/s is found, with a ratio thermal to mass diffusivity in the range 824 - 1386, indicating heat transfer is 1000 times faster than mass transfer. According to Giner (1999) and Márquez et al. (2006), this guarantees heat transfer to be instantaneous against mass transfer, and reinforces the former conclusions of isothermal drying and allows isothermal drying to be used as a reasonable simplification, accepting mass transfer occurs with internal control. Therefore, the analytical solution for unsteady state diffusion with prescribed condition on the surface (Crank, 1975; Bird, Stewart, & Lightfoot, 1960) and diffusion coefficient independent of particle moisture during drying can be used (Crank, 1975; Parry, 1985; Giner, 1999). The analytical solution, obtained after integrating local water content in the particle volume, considered to be spherical for this work, is (Márquez et al., 2006):

$$X^* = \frac{X - X_e}{X_0 - X_e} = \frac{6}{\pi^2} \sum_{n=1}^{n=\infty} \frac{1}{n^2} \exp\left[-n^2\pi^2 \left(\frac{Dt}{R_p^2}\right)\right] \tag{1}$$

where $X^*$ is the dimensionless moisture; $X$, the mean water content of the particle at time t, $X_0$ and $X_e$ the initial and equilibrium particle water content, while $D$ is the diffusion coefficient and

$R_p$ the particle radius. The infinite series of equation 1 could be reduced to only one term for long drying times, but such simplification is valid for $X^* < 0.3$ and not in the practical range for drying of high moisture foods. Then, the complete series would be required for this work, but this includes numerous shortcomings that were previously listed by Giner (1999) and Márquez et al. (2006).

Most commercial software for nonlinear regression often does not allow the use of equations with numerous terms. The minimum number of terms to ensure convergence is unknown and varies with time. A specific computer program is required to minimize residuals between predicted and experimental values, including an error tolerance to achieve convergence for each time. Once the parameters are fitted, drying curve predictions need again a specific computer program. Using the infinite series as a component of a fixed bed of particles increases computing time considerably, since a bed is composed of various thin layers. Therefore it is necessary to have an accurate, simpler and faster equation for use with computers in order to reduce computation times for the simulation of fixed beds without losing the physical meaning of the phenomenon.

A diffusive equation developed first by Becker (1959), and further by Giner (1999) has been used successfully for grain drying. The expression coincides in practice with the infinite series solution from the beginning of drying to dimensionless water contents as low as $X^* = 0.2$ in spherical geometry. Becker (1959) has proposed a prescribed water content of 0.103, on a decimal dry basis, independent of temperature and relative humidity for vacuum drying of wheat. In turn, Giner (1999) has used surface water content obtained from the sorptional equilibrium curve-assuming equilibrium with air, which is dependent on air relative humidity and temperature.

The equation mentioned above takes the following form for spherical geometry (Giner, 1999):

$$X^* = \frac{X - X_e}{X_0 - X_e} = 1 - \frac{2}{\sqrt{\pi}} a_\nu \sqrt{Dt} + 0.331 a_\nu^2 Dt \quad (2)$$

where $a_\nu$ is the area of particle per unit particle volume. In spheres, $a_\nu = 3/R_p$, with $R_p$ representing the particle radius. The radius of the particle in this case is variable, as the rosehip, like other fruits, undergoes significant volume shrinkage during dehydration (Ochoa, Kesseler, Pirone, Márquez, & De Michelis, 2002, 2007; Mabellini, Vullioud, Márquez, & De Michelis, 2010).

Analytical solutions, as well as semi-empirical and empirical expressions, have been used in most cases with constant particle radius. However, in recent works, they were used with variable radii (Thakor, Sokhansanj, Sosulski, & Yannacopoulos, 1999; Di Matteo et al., 2000) in an extended use of integral equations. In the works by Di Matteo et al. (2000), Mabellini et al. (2010), Márquez et al. (2006) and Márquez and De Michelis (2011), the radius of a sphere with the same volume as the particle was used as a variable. To estimate a drying curve for different times, calculation began with the initial radius. The water content obtained at a given time t was used to estimate the volume reduction and then a new radius. An average of both radii is taken and a final calculation of water loss for that interval is carried out with the average radius constant.

In this work, equation 2 will be used, considering the equilibrium water content given by the five-parameter GAB model presented by Vullioud, Márquez, and De Michelis (2006). Particle radius will be evaluated by the volumetric shrinkage equation published by Ochoa et al. (2002), and is presented in equation 3.

$$R_p = R_0 \left[ \left( 0.2124 + 0.7373 \frac{X}{X_0} \right) \right] \quad (3)$$

where $R_0$ is the initial particle radius.

## 2 Experimental

### 2.1 Materials

Rosehip (*Rosa eglanteria*) fruits were harvested in El Bolsón, Province of Río Negro, Argentina. The fruit was kept refrigerated (4 °C, 95.0% relative humidity) for seven days. Water content of the fresh fruit was within 48 and 49.0% expressed on wet basis, which is typical, and the mean diameter varied from 0.014 ± 0.003 m to 0.020 ± 0.004 m

## 2.2 Pretreatments

Drying fruits were pretreated in order to speed up the drying process. Pretreatments were:

a) Chemical pretreatments: Consisted in dipping the fruits in aqueous solutions of (i) 0.01 kg/kg and 0.015 kg/kg NaOH solution at boiling point (100 °C) for 1.5 min; or (ii) 0.02 kg/kg ethyl oleate with 0.025 kg/kg potassium carbonate at 70 °C for 2 min. After treatment fruits were rinsed with tap water for 5 min and dried on tissue paper.

b) Physical Pretreatments: The mechanical pre-treatments applied to the surface of the fruits were: (i) external longitudinal cuts (4 or 6 cuts) on the cuticle, made equidistantly with a scalpel; and (ii) slightly deeper perforations at equidistant points (3, 6 or 12 perforations) along the equatorial plane of the fruit, manually made with a 0.001 m diameter metallic punch. Fruits without pretreatment were also dried as control.

Chemical pre-treatments were selected as the most recommended in the literature. In the case of the mechanical pretreatments, size, number and texture of rosehip fruits was considered.

## 2.3 Drying equipment

Experiments were carried out in a purpose-built pilot scale dryer, consisting basically of a closed system with forced air circulation and appropriate drying variables control, as presented by Ochoa et al. (2002). The relative humidity of the air was controlled by bubbling of the air at 40 °C through a saturated solution of $Cl_2Mg \cdot 6\,H_2O$, and then heating the air up to 70 °C. The experimental equipment allows work on monolayers of fruits and beds with a maximum height of 0.14 m.

## 2.4 Experimental data acquisition technique

Weight loss was controlled with a OHAUS (Ontario, Canada) digital balance (±0.001 g). Air temperature was automatically controlled by software and measured with a copper constantan thermocouple connected to a digital thermometer Digi-Sense (Cole-Parmer Instrument Company, Illinois, USA) with 0.5 °C readability, while air velocity was measured with a hot wire anemometer (Mini Vane CFM Termo Anemometers EXTECH Instruments, Madison, USA). The relative humidity of drying air was determined with a Hygro Palm Hygrometer (Rotronic Instruments, New York, USA). All variables were measured at the drying chamber inlet. Fruits were placed in a single layer on a 0.225 m diameter and 0.14 m high perforated tray. The tray was easily removed or replaced sideways for periodic weighing of the sample. Once replaced, it became sealed by rubber stripping.

With the exception of the initial water content, determined by an oven procedure (AOAC, 1990), all other experimental points of the drying curve were determined by sample weight. This method is based on the constancy of sample dry matter during drying. Each weighing to determine the mass of sample involved some 20 to 30 s. To compare the effectiveness of pretreatments on drying times all pretreated samples were dried under constant conditions (Air at: 70 °C, 5% relative humidity and 5 m/s velocity).

## 2.5 Statistical analysis

Statistical analysis of experimental data was performed using ANOVA (Microcal Origin vs. 4.10)

# 3 Results and Discussion

## 3.1 Influence of pretreatments on drying times

Published results show that the processing times for rosehip fruit, as well as cherries, plums and grapes, are excessively long, a phenomenon attributable to the moisture barrier created by a highly impermeable waxy outer cuticle (Doymaz, 2007; Márquez, 2003). While this outer layer offers advantages such as protecting the fruit from external environmental factors, it is a disadvantage in terms of drying rate. Therefore, it is interesting to study the effect of different pretreat-

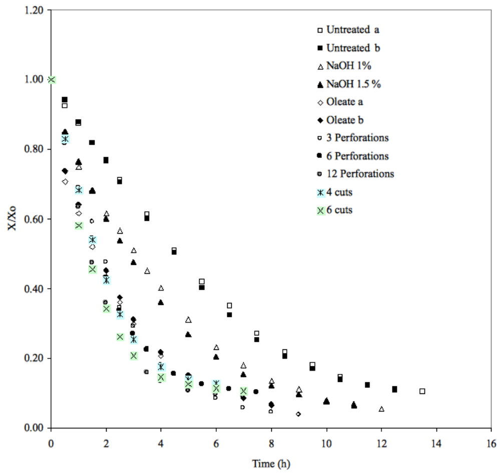


Figure 1: Drying curves of rosehip fruit untreated and pretreated chemically and mechanically (drying conditions: thin layer, air at 70 °C, 5% relative humidity and 5 m/s velocity)

ments to increase the water permeability of the surface cuticle of the fruits of rosehip.

Figure 1 shows the drying curves (relative water content $X/X_0$ vs. Time) in monolayer of pretreated rosehip fruits compared with those without pretreatment. All tested pretreatments significantly reduced drying times, and no significant differences were found on the repetitions of the same pretreatment (ANOVA, $\alpha = 0.01$, $p > 0.67$). Table 1 compares reduction of processing times (drying times for $X/X_0 = 0.15$), when the different drying pretreatment were assayed.

Table 1: Percentage reductions in drying times for the different pretreatments tested

| Pretreatment | Time reduction compared with untreated fruit (%) |
|---|---|
| NaOH 1.0 and 1.5% | 26.2 |
| Ethyl oleate 2.0% and $K_2CO_3$ 2.5% | 48.6 |
| 4 and 6 longitudinal cuts | 51.4 |
| 3, 6 and 12 perforations | 57.9 |

It was observed (Table 1) that drying times were reduced 26.2% and 57.9% for samples pretreated with NaOH solution and mechanically by perforations, respectively, with no significant differences between 3, 6 or 12 punctures per fruit (ANOVA, $\alpha = 0.01$, $p > 0.59$). While the values of % reduction of pretreatments with ethyl oleate and mechanical pretreatments provided comparable drying time reduction, the use of ethyl oleate caused a very dull surface appearance. Doymaz and Ismail (2011) found that the drying times of pre-treated cherries with oleate were 19.5 – 22.6% shorter than those of control samples. On the other hand, mechanical puncture pretreatment was the most practical method to carry out with continuous equipment.

Márquez et al. (2006) presented experimental results of thin layers drying curves of untreated rosehip fruits for different air conditions. As this paper showed, the effect of temperature on drying curves was highly significant. When the water content $X$ was expressed as dimensionless ($X^*$) as in equation 2, no differences between treatments at the same temperature could be found for all experimental data. These results allowed the authors to obtain the diffusion coefficients by fitting the equation 2 to all experimental drying data collected at the same temperature expressed as $X^*$, and the drying kinetic model gave an accurate description of the experimental data, which was corroborated by the statistical indices. These close predictions also implied that the assumption of internal mass transport by liquid diffusion satisfactorily interpreted the results for non-pretreated rosehip drying.

For the purposes of verifying whether the model of equation 2 could also represent the drying curves of the pretreated samples, regressions were carried out under the same conditions as indicated above. As Figure 2 shows, correlation of experimental data with equation 2 was satisfactory including when different pre-drying treatments were applied to rosehip fruits. The diffusion coefficients obtained for the pretreated samples, as can be expected, were higher than those obtained for samples without pretreatment. Particularly, the diffusion coefficient value for samples pretreated by mechanical punctures increased four times, as compared with untreated ones (Table 2).

As observed in table 2 , the drying kinetic

Table 2: Effective diffusion coefficients ($D$) obtained using equation 2 and statistical parameters for goodness of fit

| Pretreatment | $D$ | $R^2$ | Typical error of the estimate (In units of $X^*$) |
|---|---|---|---|
| Untreated | $1.076 \times 10^{-10}$ | 0.976 | 0.009 |
| NaOH 1.0 and 1.5% | $2.417 \times 10^{-10}$ | 0.985 | 0.008 |
| Ethyl oleate 2.0% and $K_2CO_3$ 2.5 | $3.840 \times 10^{-10}$ | 0.997 | 0.014 |
| 4 and 6 longitudinal cuts | $4.090 \times 10^{-10}$ | 0.986 | 0.073 |
| 3, 6 and 12 perforations | $4.580 \times 10^{-10}$ | 0.982 | 0.010 |

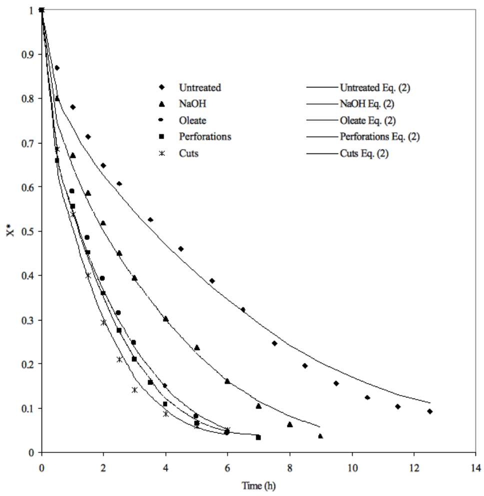


Figure 2: Variation of the experimental dimensionless water content and the estimations with the model of equation 2 for drying of rosehip fruits with different pretreatments at 70 °C, 5% relative humidity and air velocity 5 m/s.

model gives an accurate description of the experimental data, which was corroborated by the statistical indices of coefficient of determination and typical error of the estimate (in units of $X^*$). The confidence interval is the water content value ($X^*$) ± typical error. No curve overlapping was observed, even considering the typical error at every point. Therefore, as diffusion coefficients were obtained by the regression of these humidity values, no diffusion values superposition was supposed. Diffusion coefficients at 70 °C of pretreated samples were, as compared with no treated samples, 2.246 times higher for NaOH; 3.570 times higher for ethyl oleate; 3.730 times higher for cuts; and 4.256 times higher for perforations.

Figure 3 shows water content, on decimal dry basis, as a function of time during rosehip fruit drying, both experimentally and predicted by equation 2, for pretreated rosehip with NaOH and punctures. As Figure 3 shows, the model satisfactorily interprets the experimental behavior; therefore, the selected model is adequate for further use in drying simulation of thick layers of untreated and pretreated rosehips, such as those appearing in commercial scale batch and continuous dryers.

### 3.2 Influence of pretreatments on drying times for beds

Figure 4 presents, as an example, experimental curves for drying of pretreated and untreated fruits, in beds of 0.068 m in height under the same operational conditions (air at 70 °C, 5% relative humidity and 5 m/s velocity) used for thin layer drying. As shown in Figure 4 , the effect of pretreatments reduced drying times by the same order of magnitude as those obtained during thin layer drying (57.7%).

## 4 Conclusion

Air dehydration curves of rosehip fruits, with and without pretreatments, were experimentally determined both in thin layer and bed methods. As one of the objectives of this work was to determine the drying kinetics for further use in simulation of commercial drying equipment, a simple, yet physically well founded model was selected to evaluate the drying curves. This diffusive model, though valid in all the practical drying range, was used in conjunction with a sorptional equilibrium

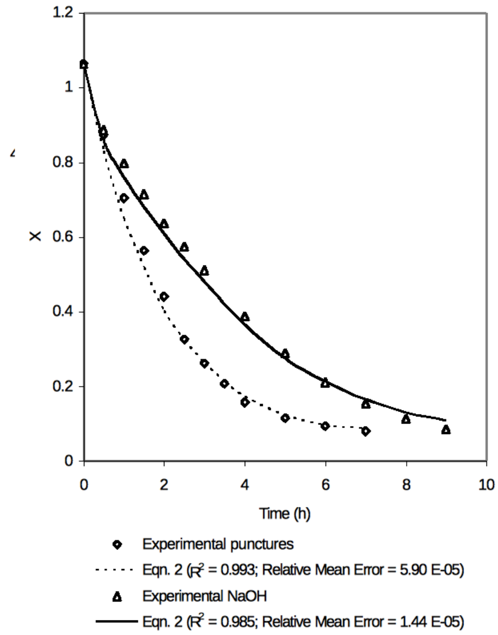


Figure 3: Dimensional water content (kg/kg dry matter) as a function of drying times for rosehip fruit samples pretreated with perforations and NaOH; operational variables of the drying air: 70 °C, 5% relative humidity and 5 m/s.

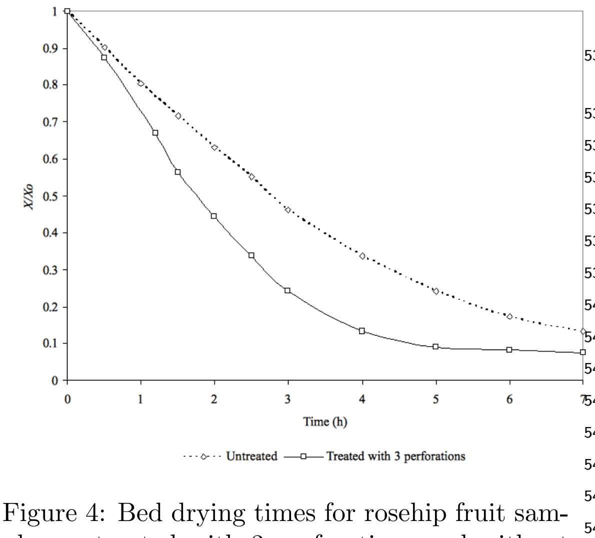


Figure 4: Bed drying times for rosehip fruit samples pretreated with 3 perforations and without pretreatment. Experimental bed height: 0.068 m; operational variables of the drying air: 70 °C, 5% relative humidity and 5 m/s.

and a volumetric shrinkage correlation. When applied to the data, this kinetic model allowed the determination of the effective water diffusion coefficient inside rosehip fruits. Also, different pretreatments to reduce processing times were evaluated. The most suitable was the mechanical perforations of the fruits with three holes sufficient to get an effective drying reduction time. The diffusive model chosen provides good results in predicting the drying kinetics both in the case of pretreated and untreated fruits, and proved to be fast to run when used in bed simulation and design of commercial dryers. It has also been experimentally verified that pretreatments reduce drying time of the fruits in deep beds in the same order of magnitude as the reductions achieved in thin layer.

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