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Comparative Study of the Drying Kinetics of Citrus Sinensis, Reticulata, Paradisi and Aurantium Seeds from Congo

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Abstract This work aimed to model the kinetics of drying Citrus seeds in an *oven. C. sinensis* (orange), *C. reticulata* (mandarin), *C. paradisi* (pomelo) and *C. aurantium* (bigarade). The fruits of the species studied from the Plateaux des cataractes were purchased in Brazzaville, specifically at the Plateau des 15 ans and PK markets. Drying was carried out at temperatures of 40, 70 and 105°C in a Memmert brand UN30 oven. Mass measurements were taken every 30 minutes for several hours until the moisture content was almost constant. The modeling of the drying kinetics of citrus seeds was carried out by the Origin Pro 2018 software while following seven models, namely: the models Diffusional, Modified Page I, Lewis, Henderson and Pabis, Page, Avhad and Marchetti, Peleg. The observations of this study on the drying kinetics of seeds highlighted two distinct phases: a rapid temperature increase phase and a gradual decrease phase. Temperature impacted the drying kinetics. As the temperature increases, the drying period decreases. Between 40 and 70 °C, bitter orange seeds are the first to dry out, followed by pomelo seeds, and finally mandarin and orange seeds. For each of the temperatures examined, the Avhad and Marchetti model was the most appropriate to represent the experimental drying data of the different citrus seed species studied.

Keywords: *Citrus, Thermal energy, speed, mathematical model*

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1. Introduction

The plant world is full of resources and virtues from which man draws not only his food but also active substances which often provide benefits to his organism sometimes affected by insidious disorders [1].

Moreover, it has been established that fruit by-products such as seeds and peels constitute a source of biologically active compounds, including vitamins, dietary fibers. These enhance their use as health-promoting supplements and enable the development of functional nutraceuticals [2].

Recent research on citrus by-products has revealed the presence of these bioactive compounds, which are promising in the food industry and have been identified as an excellent candidate for the development of functional foods intended to prevent or reduce diseases caused by oxidative stress [3].

Drying is a unit operation consisting of partially or totally eliminating the water present in a humid body by supplying thermal energy. It is a very old process for preserving agricultural and food products. It helps to obtain a dry and homogeneous product at the end of drying [4], which allows to considerably reduce the mass and volume of the product, thus facilitating its transport, storage and handling [5]. Its importance lies in the conversion of perishable goods into stabilized products by lowering the water activity to a very negligible value (< 0.5) in order to allow their storage at room temperature [6]. Also, it allows to greatly reduce the reactions that can accelerate the decomposition of the food by extraction of water contained in the product. This water is eliminated by evaporation into the air surrounding. For this, it is necessary to provide energy (activation energy), causing this water present in the product to migrate to the surface and transform into water vapor carried by the air outside the plant organ. It requires perfect control of three fundamental parameters, in particular, the drying time and the thermal energy provided which heats the product and causes the migration of water towards the surface and its transformation into water vapour; the capacity of the surrounding air to absorb the water vapour released by the product. This capacity depends on the percentage of water vapour already contained in the air before it arrives in the dryer and on the temperature to which it has been brought and, the speed of this air at the level of the product which, especially at the beginning of drying, must be high (up to a certain limit) so as to accelerate the entrainment of water vapour.

The use of drying in the food industry has multiple advantages which are as follows: Increase the shelf life of products (meat, fish, fruits, seeds, pasta, spices, tea, mushrooms) [7]; improve extraction yield; promote the transformation of products of biochemical or biological reactions; prevent the growth of microorganisms by reducing water activity on the one hand [8] and enzymes responsible for the deterioration of polyphenols such as polyphenoloxidases and prevent bacterial proliferation on the other hand [9,10] and, stabilize agricultural products and cushion the seasonal nature of certain activities.

There are two types of drying for food preservation which are divided into several modes or methods. Conventional drying which includes solar, oven and freeze drying and, non-conventional drying such as microwave drying and osmotic drying. [11]

The choice of drying method depends, among other things, on the type of product to be dried, the climatic circumstances at the time of harvest and the materials available. [12]

Oven drying This is a faster method of dehydration than sun drying, but can only be used on a small scale. An ordinary kitchen oven can be used. The temperature setting of the oven, the residence time and the size of the sample to be tested must be specified. Although this size is not generally critical, the residence time in the oven must be adapted to the surface/volume ratio. The best time is "to constant weight" to reach the dry mass. The weight loss is calculated by the difference in weight before and after drying. There are two types of ovens. For particularly rapid drying or sterilization, natural convection ovens are ideal. They guarantee rapid and uniform drying. Ovens are used up to 300°C for routine drying and sterilization tasks and are suitable for a wide range of applications. In a forced convection drying oven, all thermal processes are carried out very efficiently thanks to the high air exchange. Thanks to the homogeneous heat distribution and the fast dynamic response, this heating oven saves valuable working time. It must also be equipped with a powerful fan.

Drying kinetics translates the evolution of the rate of water elimination in a wet product in order to describe and/or predict the behavior of this product during drying. [13,14,15,16].

Modeling, on the other hand, is an abstract representation or interpretation of a reality or a physical phenomenon, which is accessible to analysis and calculations. The models are in the form of equations with a single or multiple variables. Modeling consists of developing a set of equations or rules to describe a phenomenon in a reproducible and simulable way. [17,18]

This work aimed to model the kinetics of oven drying of *Citrus sinensis seeds.* (orange), *C. reticulata* (mandarin), *C. paradisi* (pomelo) and *C. aurantium* (bigarade).

2. Materials and Material

Plant material

For the realization of this work, the shelled seeds of *Citrus* of four species namely C. *sinensis* (the orange), *C. reticulata* (mandarin), *C. paradisi* (pomelo) and *C. aurantium* (bigarade) were used. The fruits of the species studied were all purchased in Brazzaville, specifically at the Plateau des 15ans market in the "4" Moungali district for oranges and mandarins, and at the PK market in the "7" Mfilou district for grapefruit and bitter orange. These fruits all originate from the Plateaux des cataractes (Pool and Bouenza).

Drying kinetics

In order to perform the drying kinetics of *Citrus seeds*, a sample of seeds from each of the species studied was dried in a Memmert oven of type UN30 at different temperatures such as 40, 70 and 105 °C. To do this, 5 g of the *Citrus seeds* studied were placed in petri dishes and then placed in the oven. Mass measurements were taken every 30 minutes for several hours until the moisture content was almost constant. The weighing was carried out using an analytical balance of the Constant brand and resolution d = 0.01 g.

Modeling of seed drying kinetics

To predict the drying kinetics of the studied plant materials, a mathematical model of the seed moisture evaporation process was necessary [19,20,21,22].

As a result, seven (07) mathematical models were retained for modeling the drying kinetics of the seeds of the citrus fruits studied (Table 1).

The data predicted by these mathematical models were fitted to the drying curves of the experimental data to select the models that best describe the drying process of the leaves studied.

Table 1. Mathematical models of drying kinetics used

Model	Equation
Diffusional	X*= a*exp(-k*t)+(1-a)*exp(-b*k*t)
Page modifié I	X*=exp(-(k*t)^n)
Lewis	X*=exp(-k*t)
Henderson et Pabis	X*=a*exp(-k*t)
Page	X*=exp(-k*t^N)
Avhad et Marchetti	X* =a* exp (-k* t^N)
Peleg	X* = 1-t /(a+b *t)

With k, n, a, b and N are the drying coefficients

In this study, mass loss data collected from the studied seed samples at different time intervals were transformed into mass loss data. moisture loss. From the initial water content of the plant material, water content data at different time intervals and equilibrium moisture content for different temperatures, reduced water content without dimension (X^*) was determined. Then, this reduced water content as a function of time was used to adjust the mathematical models. The expression used to calculate X^* is expressed by the following equation:

$$X^* = \frac{m_t - m_e}{m_i - m_e}$$

With: Mt: water content at time t; me: water content at equilibrium; mi: initial water content.

Statistical analysis

The modeling of citrus seed drying kinetics was performed by Origin Pro 2018 software while following the models chosen in Table 1.

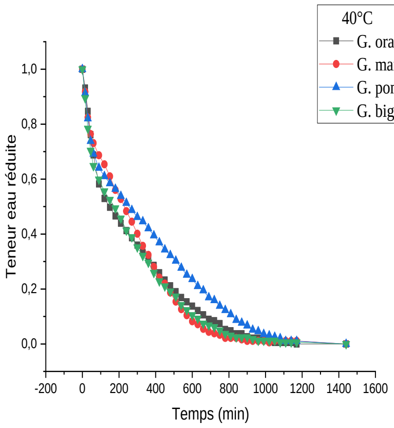
Chi-square ($\chi2$), coefficient of determination (R2), are statistical parameters used to determine the degree of fit of the model. The highest R2 value, close to 1, and the lowest $\chi2$ value, close to 0, represent the best fit. [23]

3. Results and Discussion

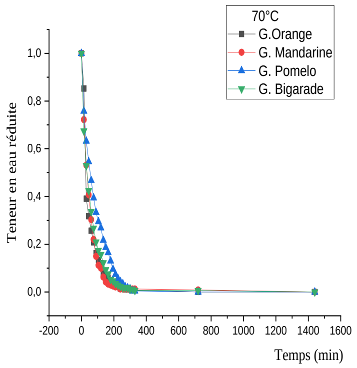
Influence of the time-temperature couple on the evolution of water loss

Drying kinetics tells us about the loss of water in a body as a function of time at different temperatures.

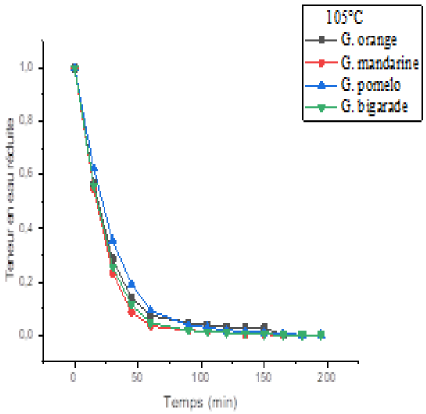
Drying Citrus *Seed Samples sinensis, reticulata, paradisi* and *aurantium* was carried out using a natural convection oven at temperatures of 40, 70 and 105°C or 312, 343 and 378 K. The water loss as a function of time at varying temperatures is shown in curves (a), (b) and (c) of the following figure 1:



Drying seeds at 40°C



Drying seeds at 70°C



Drying seeds at 105°C

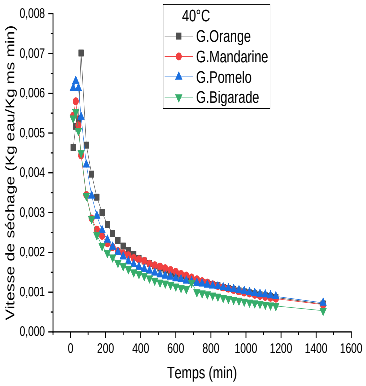
Figure 1. Citrus seed drying kinetics curves studied at different temperatures

The curves shown in Figure 1 show the evolution of the reduced water content as a function of the drying time at different temperatures. Examination of curve (a) whose seeds were dried at 40°C shows that the water loss of the samples studied is significant during the first 700 minutes and begins to stabilize from 800 minutes. During the first 80 minutes all the seeds have the same drying characteristics and each species releases almost 30% of moisture. After 80 minutes of drying, the moisture evaporation behavior changes according to the species. However, practically the same behavior was observed on the drying of bitter orange and orange seeds from the beginning to the end of drying at 40°C but this does not mean that these seeds contain the same amount of water, rather it could be due to the fact that these seeds have practically the same moisture evaporation capacity. At 400 minutes, bitter orange, mandarin and orange seeds released nearly 80% of moisture, while grapefruit seeds had to wait 700 minutes to evacuate this amount of water. From these observations, we say that the seeds of the citrus studied do not have the same capacity for evaporating moisture. Grapefruit seeds seem to lose less water during drying compared to the other three species studied because their moisture evaporation time is longer to evacuate the same level of moisture. As for the analysis of curve (b) whose seeds were dried at 70°C, we observe a considerable decrease in water during the first 200 minutes and begin to stabilize from 300 minutes. At around 40 minutes, all the seeds studied have lost 40% of the moisture they contained. After this time, the moisture evaporation in the grapefruit seeds becomes slower which justifies the distance of the grapefruit seed drying curve. From this observation, we say that the moisture evaporation process in grapefruit seeds is slower than in other seeds for a certain time. However, at around 380 min, all the seeds of each of the species studied become dry. For curve (c) whose seed drying was carried out at 105°C, a considerable loss of moisture was observed in the first 60 minutes and begins to stabilize after this time. A The same observation was made with grapefruit seeds, its characteristic curve is always further away from the others at all temperatures at certain drying times. This leads us to believe that the moisture evaporation process in grapefruit seeds is more difficult or slower than in the other seeds studied at a given time; at least it should be noted that at around 100 min, grapefruit seeds dry after those of mandarin and bitter orange. In view of these observations, we say that the reduced water content decreases with the increase in the Time-Temperature couple, that is to say, the more the time increases, the less the plant material loses moisture for the same temperature. Similarly, the more the temperature increases, the more the plant material loses moisture in a short time. Clearly, the higher the drying temperature, the shorter the drying time; the lower the drying temperature, the longer the drying time. In other words, we say that the drying time is inversely proportional to its temperature. From these results, we adhere to the idea of [24], which states that temperature has an influence on the evolution of water content during drying. The influence of temperature on drying kinetics is important, and an increase in temperature results in a decrease in water content. Drying these samples at 40°C does not promote rapid moisture evacuation from the seeds studied to reach the desired moisture content [25]. It emerges from this experiment that the seeds of *Citrus sinensis, reticulata, paradisi* And *aurantium* can be dried for 3, 7 and 20 hours at 105, 70 and 40°C respectively.

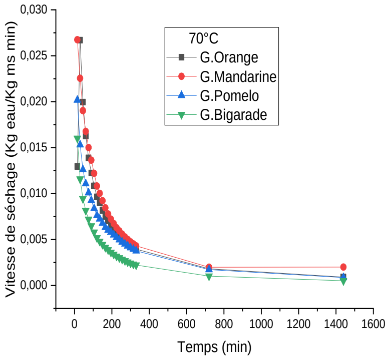
Influence of the time-temperature couple on the evolution of the drying speed

It characterizes the decrease in water content in a product over time.

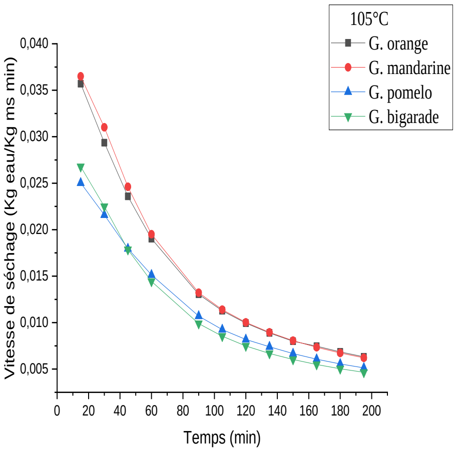
Curves **(a)**, **(b)** and **(c)** in Figure 2 describe the evolution of the drying speed as a function of the Time-Temperature couple.



Drying speed at 40°C



Drying speed at 70°C



Drying speed at 105°C

Figure 2. *Citrus* seed drying speed curves as a function of the Time-Temperature couple

The results show that the drying speed decreases at all the fixed temperatures. This speed varies depending on the temperature and the drying time. The analysis of curves (a) and (b) whose seeds were dried at 40 and 70°C respectively shows that the drying speed decreases considerably during the first 100 minutes for curve (a) and for almost 50 minutes for curve (b). We note the formation of three groups in each of the curves which show that these seeds do not have the same drying speed. We note that the bitter orange seeds dry first followed by the grapefruit seeds and finally the other two (mandarin and orange) which have almost the same drying speed. As for curve (c) whose drying was done at 105°C, we note the formation of two groups which show a rapid decrease in speed during the first 90 minutes. For the first group, the drying speed of grapefruit seeds is higher than that of bitter orange seeds during the first 45 minutes. After this time, the bitter orange seeds take over until complete drying, which shows that the drying speed of the bitter orange seeds is more accelerated than that of the grapefruit seeds. For the second group, the orange seeds dry faster than the mandarin seeds during the first 87 minutes. After this time, the same drying speed was observed until the total exhaustion of moisture in the orange and mandarin seeds.

From this experiment, we note that only the drying temperature significantly influences the drying speed of citrus seeds. For this, we say that the drying speed is a function of the temperature for a given time. Also, an increase in temperature leads to a decrease in the drying time of a product. Indeed, when the temperature of the drying air increases from 40 to 105°C, there is a decrease in the drying time of citrus seeds from 20 h to 3 h respectively.

Assessment of the fit of mathematical models

Drying is a complex thermal process in which unstable heat and moisture transfer occur simultaneously [26]. It is important to better understand this complex process in engineering. Therefore, mathematical modeling plays a vital role [27].

In order to assess the fit of the mathematical models which best describe the drying curves, the calculated values of the statistical parameters used are reported in

Table 2, Table 3 and Table 4 below.

The selection of the best-fitting mathematical model is based on the values of coefficient of determination (R^2) and reduced ki -square ($\chi2$) [28] (Doymaz,. In modeling the drying kinetics of castor beans, [29] Perea *et al* in 2012 accepted mathematical models with R2 values greater than 0.97 as models fitted to experimental data; similarly, this theory was validated by [30] Meziane in 2013 on the modeling of the drying kinetics of olive pomace, and confirms that a model is adopted when R2 values are greater than 0.97.

The different models are compared based on the values of the coefficient of determination and $\chi2$. The higher the coefficient of determination R^2 is close to 1, the better the model fits. Also, the closer the ki -square is to zero, the better the model fits.

As part of this study, a modeling of the drying kinetics of *Citrus sinensis, reticulata, paradisi and aurantium seeds* was carried out using the *Origin Pro 2018 software* with the selection of seven (07) mathematical models namely: the Diffusional model, the modified Page I model, the Lewis model, the Henderson and Pabis model, the Page model, the Avhad and Marchetti model and the Peleg model. The data predicted by the mathematical models used were fitted to the drying curves of the experimental data of *Citrus* seeds to finally select the model that best validates the drying process of these seeds. The statistical parameters resulting from the modeling of the drying processes at 40°C of the different citrus seeds studied are presented in the following Table 2.

Examination of Table 2 shows that apart from the Diffusional and Lewis models which do not validate the drying kinetics of *Citrus paradisi seeds* (0.95654), all the models selected for modeling the drying kinetics of citrus seeds studied at 40 °C are suitable with $R^{2\ \mathrm{values}}$ ranging from 0.9677 to 0.99026 and ki ^{2} values ranging from 33.9 E-4 to 7.79003E-4. Although the Peleg model is slightly the best fitted than that of Avhad and Marchetti with a difference of 0.00212 for the drying of ***C. sinensis*** seeds at 40 °C, the best fitted model is that of Avhad and Marchetti with a coefficient of determination of 0.99026; 0.97568 and 0.98679 respectively *for* drying seeds of *C. reticulata*, C. *paradisi* and *C. aurantium.*

The analysis of Table 3 whose seed drying was carried out at 70°C shows that except for the Diffusional and Peleg models with the respective coefficients of determination of 0.72334 and 0.95976 for the drying of *C. sinensis seeds* and that of Peleg with a coefficient of determination of 0.94471 for the drying of *C. reticulata seeds* which do not validate the drying kinetics of the seeds of these species, all the kinetic models selected for the mathematical modeling of citrus seeds studied at this temperature are suitable with the coefficient of determination values ranging from 0.9714 to 0.99911. However, the Avhad and Marchetti model proved to be the best adjusted to the drying process of all species with the respective coefficients of determination of 0.97328; 0.99911; 0.99449 and 0.99884 for drying seeds of *C. sinensis, C. reticulate, C. paradise* and *C. aurantium.*

Table 2. Values of statistical parameters of seeds dried at 40°C

	Diffusional	Modified Page I	Lewis	Henderson and Pabis	Page	Avhad and Marchetti	Peleg
C. sinensis							
R^2	0.97421	0.98754	0.97421	0.98459	0.98754	0.98787	9.68452E-4
$\chi2$	20.6E-4	9,70308E-4	19.6E-4	12 E-4	9,70313E-4	0,98999	7,79003E-4
C. reticulata							
R^2	0,9839	0,98415	0,9839	0,98523	0,98411	0,99026	0,98252
$\chi2$	15,1 E-4	14,5 E-4	14,4 E-4	13,5 E-4	14,5 E-4	9,1316E-4	16 E-4
C. paradisi							
R^2	0,95654	0,9677	0,95654	0,97549	0,96769	0.97568	0.97069
$\chi2$	33.9 E-4	24.6 E-4	32.3 E-4	18.7 E-4	24.6 E-4	19 E-4	22.3 E-4
C. aurantium							
R^2	0.97234	0.98401	0.97234	0.98643	0.98401	0.98679	10.3 E-4
$\chi2$	21.7 E-4	12.2 E-4	20.6 E-4	10.4 E-4	12.2 E-4	0.98453	11.8 E-4

Table 3. Values of statistical parameters of seeds dried at 70°C

	Diffusional	Modified Page I	Lewis	Henderson and Pabis	Page	Avhad and Marchetti	Peleg
C. sinensis							
R^2	0.72334	0.97257	0.9714	0.97152	0.97256	0.97328	0.95976
$\chi2$	198.5 E-4	18.8 E-4	18.8 E-4	19.5 E-4	18,8 E-4	19,2 E-4	27,6 E-4
C. reticulata							
R^2	0,99898	0,99911	0,99893	0,99899	0,99911	0.99911	0.630599E-4
$\chi2$	0.726106E-4	0.60376E-4	0.69712E-4	0.68733E-4	0.60379E-4	0,94471	9,42434E-4
C. paradisi							
R^2	0,98382	0,99408	0,99071	0,99364	0,99408	0.99449	0.99408
$\chi2$	0.00128	4,47588E-4	6,728E-4	4,80317E-4	4,47607E-4	4,3483E-4	4,47607E-4
C. aurantium							
R^2	0.9883	0.99881	0.9883	0.99221	0.99881	0.99884	0.98936
$\chi2$	7,89617E-4	0.76909E-4	7,23816E-4	5,02958E-4	0.76913E-4	0.7797E-4	6,87069E-4

Table 4. Values of statistical parameters of *C seeds. sinensis* dried at 105°C

	Diffusional	Modified Page I	Lewis	Henderson and Pabis	Page	Avhad and Marchetti	Peleg
C. sinensis							
R^2	0.99621	0.99633	0.99621	0.99624	0.99633	0.99633	0.98471
$\chi2$	3.97261E-4	3.5E-4	3.31051E-4	3.58192E-4	3.50174E-4	3.8433E-4	14.6 E-4
C. reticulata							
R^2	0.99651	0.99955	0.99536	0.99559	0.99955	0.99955	0.97486
$\chi2$	3.74079E-4	0.43484E-4	4.1491E-4	4.2971E-4	0.43498E-4	0.4783E-4	0.00245 E-4
C. paradisi							
R^2	0.9979	0.99934	0.9979	0.99808	0.99934	0.99934	0.9837
$\chi2$	2.36738E-4	0.67779E-4	1.9728E-4	1.9727E-4	0.67817E-4	0.7442E-4	16.7E-4
C. aurantium							
R^2	0.99719	0.99967	0.99719	0.99736	0.99967	0.99967	0.97913
$\chi2$	3.02826E-4	0.31996E-4	2.5236E-4	2.5898E-4	0.32011E-4	0.3518E-4	20.4E-4

The statistical results recorded in Table 4 whose drying of seeds of all the species studied was carried out at 105°C show that the seven models are suitable for the mathematical modeling of this drying kinetics. However, the models of avahd and machetti, Page and modified Page I proved to be the best-fit models when drying all the species at this temperature and give the coefficients of determination very close to 1 in this case 0.99633; 0.99955; 0.99934 and 0.99967 respectively for the drying of seeds of *Citrus sinensis, C. reticulata, C. paradisi* etc. *aurantium.* Although the Avahd and Marchetti, Page and modified Page I models give the same coefficients of determination for each of the species at 105°C, the modified Page I model proved to be the best adjusted to the experimental data of our samples because it presents the value of $\chi2$ the lowest therefore closer to zero. Also, the temperature of 105°C is the best to model the drying of citrus seeds.

4. Conclusion

At the end of this research which focused on the modeling of the drying kinetics of the seeds of *Citrus sinensis, reticulata, paradisi* and *aurantium,* it should be noted that the drying kinetics of the seeds of *Citrus* studied were followed by producing characteristic curves of moisture evaporation. The curves made it possible to differentiate two phases, namely the very short heating phase and the decreasing speed phase. This drying kinetics was influenced by the temperature. The higher the temperature, the shorter the drying time. For all temperatures considered, the Avhad and Marchetti model frequently proved to be the best adjusted. Also, the temperature of 105°C is the best to model the drying of citrus seeds.

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