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Effect of the application of intermittent drying on *Ilex paraguariensis* quality and drying kinetics

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ABSTRACT

The aim of this work was to study the effects of the application of tempering periods on the drying kinetics of yerba maté branches and on the resultant quality parameters of the finished product. Experiments were carried out in a convective pilot plant drier. Air temperature (60, 80 and 100 °C) and tempering time (0, 15 and 30 min) influenced the drying kinetics and the product quality (color parameters *L* and *b*, and the sugar and caffeine contents of an infusion prepared with the material). The influence of tempering time was higher at 60 °C than at the other temperatures. There were no differences between tempering times of 15 and 30 min. The Page model yielded a good fit to the experimental data, where the model parameter *k* varied with drying temperature.

1. Introduction

Currently, there is a growing trend to combine different drying processes according to product characteristics in order to minimize energy consumption (Menshutina et al., 2004). Intermittent drying is a type of discontinuous drying process with tempering periods of heat application in which moisture diffuses from the inner part of the solid to the surface. Consequently, the moisture gradient is diminished, increasing the drying rate (Raisul Islam et al., 2003). This drying technique has been widely researched in the drying of products such as rice, banana, guava, potatoes, soybean, and wheat, reducing in all cases the time of heat application required (Aquerreta et al., 2007; Chua et al., 2000, 2003; Cnossen et al., 2003; Nishiyama et al., 2006; Shei and Chen, 2002; Thomkapanich et al., 2007; Tuyen et al., 2009), but it has not yet been studied in a heterogeneous material like Ilex paraguariensis or yerba maté branches.

Yerba maté is industrially processed as whole branches. Once processed, a very popular tea can be prepared from the leaves and twigs of the plant. There are about 300 factories in Argentina that produce about 220,000 tn/year of the processed product. The processing steps are: (1) heat treatment with hot gases for several minutes to inactivate enzymes which would lead to browning of the leaves; (2) drying, that are usually carried out in two steps in a crossflow drier; (3) grinding, used to prepare the material for the next step, and (4) seasoning, which is carried out in a natural way (storage for 9 months at room temperature) or a controlled way (storage in chambers at 60 °C for 30–60 days).

The irregular shape and the heterogeneous composition of the material (leaves and twigs) make the application of theoretical models, e.g., the integrated Fick 2nd law equation, very difficult. In order to explain the drying kinetics data, empirical or semi-empirical models such as the Page equation (Page, 1949) should be used. From this model, apparent or effective coefficients are obtained in order to compare the different drying kinetics. The Page model has been applied to describe water losses during drying in different food products, e.g., in rice drying (Chandra and Singh, 1984; Ramesh and Rao, 1996), in sunflower seed drying (Syarief et al., 1984) and in the osmotic dehydration of pears (Park et al., 2002). Panchariya et al. (2002) applied different semi-theoretical models, including the Page model, in order to analyze the drying kinetics of green tea (a similar material to yerba maté) at temperatures varying between 80 and 120 °C, but it has not been yet applied to the intermittent drying of a heterogeneous material.

In intermittent drying, heat is applied discontinuously. A material dried in this way therefore has a different heat treatment than one dried in a continuous manner. Consequently, it should have different physical and chemical characteristics. This hypothesis has been demonstrated when this technique was applied to other materials. Chua et al. (2000) found that color variations in banana, guava and potato during drying were influenced by the drying method when heat was applied in a continuous or discontinuous

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Nomenclature

a	color parameter (green–red)
b	color parameter (blue–yellow)
k	constant of Page equation
L	color parameter (lightness–darkness)
MR	$(W - W_e)/(W_0 - W_e)$
n	constant of Page equation
t	drying time (min)
τ	tempering time (min)
W	moisture content at time t (g water/100 g of dry solid)
W_0	moisture content at time $t = 0$ (g water/100 g of dry solid)
W_e	equilibrium moisture content (g water/100 g of dry solid)

way. Thomkapanich et al. (2007) applied two drying methods (superheated steam at low pressure and vacuum drying) to sliced bananas with different air temperatures and tempering times. Both parameters influenced several quality characteristics, e.g., color parameters, vitamin C concentration, hardness, crispness and shrinkage coefficient. Jaisut et al. (2008) applied intermittent drying in a fluidized bed drier to rice, concluding that drying temperature and tempering time influenced rice digestibility properties. In contrast, Madamba and Yabes (2005) found that some physical properties of rice (hardness, germination ratio, whiteness and degree of cracking) were influenced by drying temperature, but not by tempering time.

Traditional drying of yerba maté is carried out in crossflow belt driers. The process takes approximately 5–6 h using air temperatures varying between 80 and 120 °C. Leaves have a higher surface-area-to-volume ratio than twigs (Coelho et al., 2002) and lose water quickly. A large moisture content gradient thus appears between them in the first step of the drying. Thus, Schmalko et al. (2007) found that after an hour of drying, leaves reached a moisture content of approximately 9% (dry basis or d.b.) versus 70% (d.b.) for twigs. The application of a tempering time would allow moisture redistribution between both materials, along with the internal moisture distribution in each one that takes place in most materials. Consequently, the total time of heat application should be reduced.

The aim of this research was to study the effects of the application of tempering periods on the drying kinetics of yerba maté branches and on several quality parameters in the finished product.

2. Material and methods

2.1. Materials

The final stage of yerba maté processing is a heat-treatment step at high temperatures in order to inactivate the enzymes that produce browning of leaves. The test material was obtained after this stage from a factory in Misiones, Argentina. Branches with similar characteristics (i.e., shape and weight) were selected and put into an isolated container before processing. Selected branches of yerba maté had a length of about 25 cm, which have a mean percentage of leaves (d.b.) of 73% and a standard deviation of 2%. Leaves moisture content, before drying had a mean value of 49.28% (d.b.) with a standard deviation of 16.28%, while twigs had a mean moisture content of 108.33% (d.b.) and a standard deviation of 5.33%.

2.2. Drying experiments

Drying experiments were carried out in a pilot plant crossflow drier (Fig. 1). Operating conditions were selected in accordance with standard industrial processing: an air temperature of 60, 80 or 100 °C and an air velocity of 1 m/s. The material was put into baskets in a bed of approximately 10 cm thickness and 250 ± 10 g in weight. During the tempering time, air flux was recirculated (opening valve 1 and closing valves 2 and 3) and the material was maintained in a closed cabinet.

The dry weight of the material and its moisture content were determined by drying the sample in an oven at 103 ± 2 °C until constant weight was achieved (approx. 6 h) (IRAM 20503, 1995). Drying data (moisture content versus time) were obtained from the wet and dry weights of the material at the different times. Tempering times of 15 and 30 min were applied. The time of heat application varied between 90 and 120 min, according to drying temperature. Experiments were carried out in duplicate.

2.3. Drying curves

In order to describe moisture content variation as a function of time, the Page model (Page, 1949) was used. This equation was

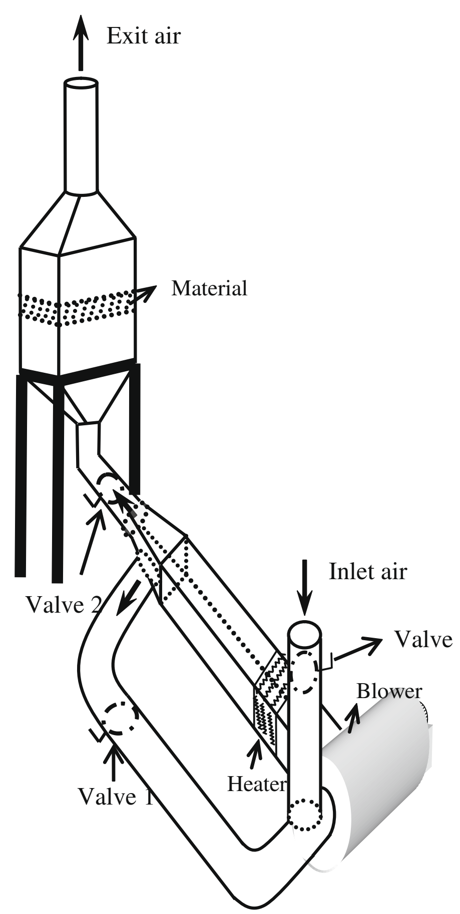

Fig. 1. Schematic diagram of the drier.

developed to explain loss of water during the drying of a thin layer of material (Eq. (1))

$$MR = \frac{W - W_e}{W_0 - W_e} = \exp(-kt^n) \tag{1}$$

where W is the moisture content at time t, W_0 Moisture content at time $t = 0$ and W_e is the equilibrium moisture content. It was obtained from a previous research (Känzig et al., 1987). Values of k and n were calculated by a linear regression technique using Eq. (1).

2.4. Color parameters

Color parameters were measured using a Hunter Lab D25-9 solid colorimeter (Hunter Associates Laboratory). Response parameters on the Hunter Lab scale were as follows: L (lightness or black–white axis), a (green–red axis) and b (blue–yellow axis). Six replicates of each sample were made.

2.5. Sugar and caffeine determination

2.5.1. Sample preparation

In order to determine sugar (glucose and sucrose) and caffeine content, infusion, the traditional method of preparation of yerba maté for consumption, was used. The compounds were analyzed in a water extract prepared in a standardized way (ISO 3103, 1980).

Dried samples were milled in a knife mill with an outlet sieve 1 mm in diameter. Bags with 3 g of the milled samples were prepared using tissue paper. Water extracts were prepared by pouring 150 mL of boiling water into a glass containing the bag and stirring for 6 min (the time and temperature were according to ISO 3103 (1980). The bag was then removed, and the values of the resulting solution were measured.

2.5.2. Sugar (glucose, fructose and sucrose) determination

One milliliter of acetonitrile was added to 2 mL of the water extract, and then the sample was centrifuged. Glucose, fructose and sucrose were determined using an HPLC method (IRAM 20532, 2004). An amino column (Altima; 250 mm × 4.6 mm, Altech Associates Inc., USA) was used with a Waters model 410 differential refraction index detector. The mobile phase was a mixture of acetonitrile/water (70:30 v/v) at a flux of 1.2 mL/min; determinations were made in duplicate.

2.5.3. Caffeine determination

Caffeine was determined using an HPLC technique (IRAM 20512, 2000). A C18 column (Ultrasphere; 250 mm × 4.6 mm, Beckman, USA) with a particle diameter of 5 μm, a mobile phase of methanol:water (30:70 v/v) at a flux of 1.1 mL/min, and a Waters model 481 UV–vis spectrophotometer at a wave length of 280 nm were used; determinations were made in duplicate.

2.6. Statistical analysis

In order to determine parameters of Eq. (1), a linear regression analysis was used. To compare quality parameters (color parameters L, a and b and caffeine and sugar content) an analysis of variance was used. The Stat Graphics (2009) statistical package was used to process the data.

3. Results and discussion

3.1. Drying kinetics

Considering the large differences between moisture contents of leaves and twigs at the end of the drying; the final moisture content of the branches was calculated from the dried weight of both materials and the final moisture content.

Initial moisture contents of the material were 65.23 ± 6.90% (d.b.). The objective of this research was to apply the intermittent drying technique to the industrial drying of yerba maté. Thus, the selected air temperatures in this study were similar to the industrial processes. Selected tempering times, 15 and 30 min, were also selected with this criterion. Drying data with different times (15 and 30 min) and temperatures (60, 80 and 100 °C) are shown in Figs. 2–4, respectively.

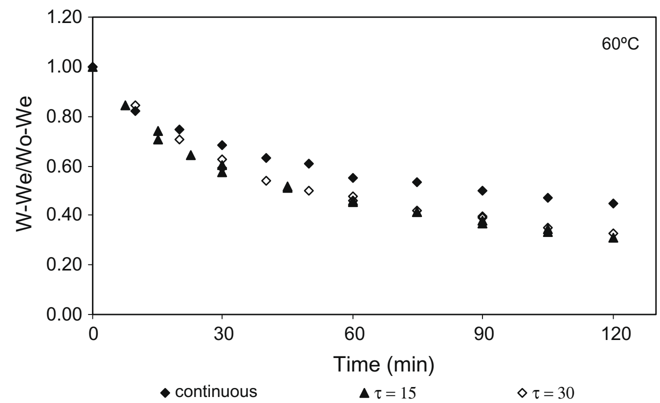

Fig. 2. Effect of intermittency on the evolution of moisture content for yerba maté branches during drying at 60 °C and 1 m/s air velocity.

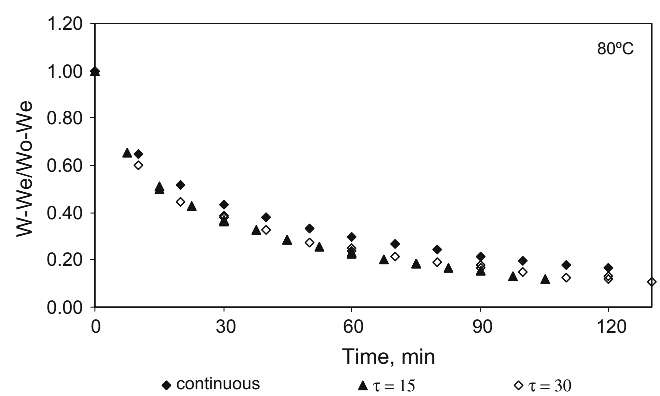

Fig. 3. Effect of intermittency on the evolution of moisture content for yerba maté branches during drying at 80 °C and 1 m/s air velocity.

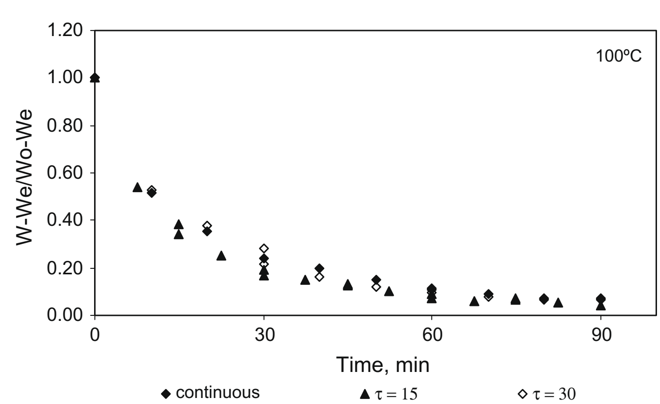

Fig. 4. Effect of intermittency on the evolution of moisture content for yerba maté branches during drying at 100 °C and 1 m/s air velocity.

Fig. 2 shows the drying data at 60 °C. When the tempering time was applied, drying rate increased, consequently decreasing the time necessary to reach a predetermined moisture content. For example, at a drying time of 90 min, the dimensionless moisture content, MR had a value of 0.51 for the continuous drying, while when a tempering time was applied, this value was about 0.40. This difference is probably due to the fact that at 90 min and 60 °C, the moisture content did not reach the equilibrium moisture content. The increase of the tempering time from 15 to 30 min did not clearly affect the drying rate.

The experimental results of drying at 80 °C are shown in Fig. 3. As can be observed here, the application of a tempering time favors drying kinetics. In this case, similar to that at 60 °C, no differences between tempering times of 15 and 30 min were observed. The differences in MR between drying data with or without tempering were diminished when the drying had progressed, so that the moisture content of the material was near the equilibrium moisture content. Thus, it is advantageous to apply tempering time during the first 60 min of drying.

Drying tests at 100 °C had a different behavior (Fig. 4): only some differences were found between the drying with a tempering period of 15 min and the continuous drying curve and 30-min tempering periods. The differences were noticeable only in the first 30 min of drying. In this period, a low value of MR was reached (approximately 0.20). So, when the first tempering period was applied after 30 min of process, the moisture content was so low that the application had no effects. This is probably the reason why no differences were found between experiences without tempering and with a tempering period of 30 min. It should be remembered that time of heat application and tempering time were equal.

Intermittent drying curves using the effective drying time or time of heat application, excluding the tempering time, can be compared with the continuous drying curves. When the experimental data were fitted to Page model, the statistical analysis demonstrated that this model was adequate to describe the drying curves (Table 1). "n" parameter was not found to depend on temperature and tempering time. So, the average value of n (= 0.60) was used to compare k parameter. In the three cases, k parameter depended on drying temperature, increasing its value with the increasing temperature. At 60 and 80 °C, the application of tempering times of 15 and 30 min increased the k value. At these temperatures, no differences were found between the drying kinetics with tempering periods of 15 and 30 min. A different behavior was found at 100 °C, since the k value was only statistically different with the application of a tempering time of 15 min. This difference was appreciable at the first 30 min of drying when the MR value was high.

As in this research, Senadeera et al. (2003) and Simal et al. (2005) found that the parameter n remained constant with temperature during the drying of beans, potatoes and peas (between 1.6 and 2.05) and kiwi (0.796). Abalone et al. (2006) found that the parameter n during the drying of amaranthus seed only depended on the initial moisture of the solid, while the parameter k depended on temperature, initial moisture content and relative humidity. In this study, k increased with air temperature but did not depend on tempering time. Azzouz et al. (2002) also found, during grape drying, that the parameter k only depended on air temperature. Simal et al. (2005) demonstrated a linear dependence of k parameters with temperature during kiwi drying.

Table 1
k parameter of the Page equation (with $n = 0.60$), correlation coefficients and mean percentage errors of the estimations.

T (°C)	Tempering time τ (min)	k (mean value ± 95% confidential limits)	R^2	Mean percentage error
60	0	0.048 ± 0.001	0.996	1.0
	15	0.066 ± 0.002	0.994	1.4
	30	0.064 ± 0.002	0.988	2.0
80	0	0.105 ± 0.002	0.999	0.6
	15	0.129 ± 0.002	0.999	0.6
	30	0.123 ± 0.003	0.999	0.7
100	0	0.181 ± 0.006	0.994	0.7
	15	0.233 ± 0.009	0.988	1.8
	30	0.177 ± 0.011	0.985	1.9

3.2. Color parameters

When an analysis of variance was made considering color parameters of the dried product, the parameters L and b were influenced by drying temperature ($P < 0.004$ and $P < 0.001$, respectively), while the application of a tempering time did not influence them. Fig. 5 shows the influence of drying temperature on L and b. The most important variations took place between 60 and 80 °C. The increase in b with temperature indicates that the product dried at 80 and 100 °C had a more yellow color, while an increase in L indicates that the product less dark. Parameter a was not influenced neither by drying temperature nor tempering time. It had a mean value of −11.65 and a standard deviation of 0.72. Values of b were similar to those obtained by Schmalko and Alzamora (2001) when they measured color parameters in the different steps of yerba maté processing, but the parameters L and a had lower values in this study.

3.3. Caffeine in the water extracts

The application of a tempering time influenced the caffeine concentration in the water extracts of the products ($P < 0.01$), while the drying temperature did not influence it. Differences in caffeine concentration were found between the products without tempering times and those with tempering times (Fig. 6). This variation could be related to the longer drying time applied when tempering periods are used. Samples, during part of the tempering period are at high temperatures and this may cause greater losses. Usually,

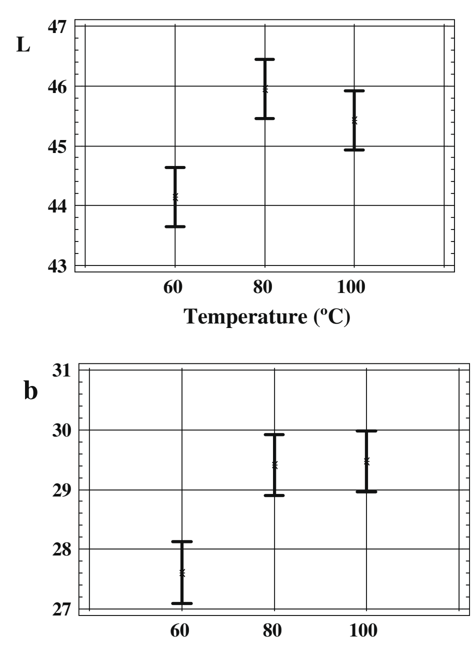

Fig. 5. Parameters L and b variations with drying temperature.

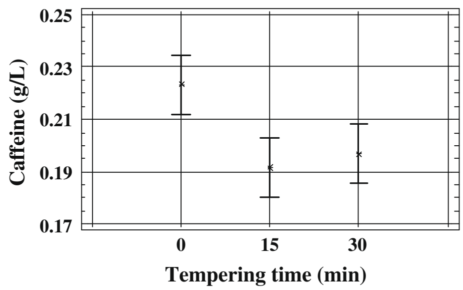

Fig. 6. Caffeine content in the water extracts with tempering time.

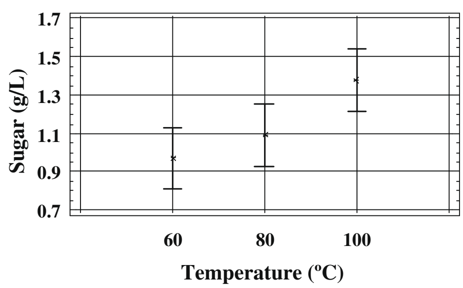

Fig. 7. Sugar content in the water extracts with drying temperature.

high caffeine content is desirable and the application of a tempering time reduces its value in about 10%. It should be remembered that caffeine partially sublimates at these temperatures and some losses of about 30% have been found during yerba maté drying (Schmalko and Alzamora, 2001).

3.4. Sugars in the water extracts

Sugar concentrations in the water extracts of the dried products were found to be influenced ($P < 0.028$) by drying temperature (Fig. 7). During the drying process, changes in the structure of the material took place, and these changes depend on drying temperature. These changes could modify the diffusion rates of some compounds. Different values of sugar concentrations in the water extract may be explained by them. The application of a tempering time did not influence the sugar concentrations in the water extracts. Changes in sugar content during drying were also found by other researchers, like in the convective drying of currants at temperatures of 65–97 °C (Karathanos, 1999). Bondaruk et al. (2007) found changes in the sugar content during drying of potatoes using vacuum-microwave and convective drying. Nevertheless, Borompichaichatkul et al. (2009) and Correia et al. (2009) applying different drying methods, did not find differences in the sugar content of nuts after drying.

4. Conclusions

Intermittent drying curves using the effective drying time, where tempering times are excluded, were compared with continuous drying curves. After 2 h of drying at 60 °C, samples with tempering times had about 10–12% (d.b.) less moisture than those dried continuously. At 80 °C, this difference was about 3–4% (d.b.), and at 100 °C no appreciable differences were found. At this temperature, differences were found only at the beginning of the drying process. At 60 and 80 °C, no differences were found between tempering times of 15 min and 30 min. It should be convenient to apply a tempering time during drying at low temperatures (60 °C) or at the beginning of the drying at high temperatures, when the twig moisture content is still high.

The Page equation gave a good statistical fit to the experimental data, with very low values of mean percentage error, 0.6–2.0%. The model parameter k, considered to be a measure of the drying rate, increased with temperature.

Drying temperature influenced the color parameters L and b and sugar concentrations in the water extracts. In all cases, an increase in drying temperature increased these parameters. Tempering time influenced caffeine concentration in the water extract, decreasing it in about 10%. Consequently, the application of a tempering time during the drying of yerba maté branches had a low influence on the product quality.

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