

Vacuum drying of peeled coffee cherry beans: drying kinetics and physiological effects

Hully Alves Rocha¹, Jefferson Luiz Gomes Correa², Flávio Meira Borém¹

¹Universidade Federal de Lavras/UFLA, Departamento de Engenharia/DEG, Lavras, MG, Brasil

²Universidade Federal de Lavras/UFLA, Departamento de Ciência dos Alimentos/DCA, Lavras, MG, Brasil

Contact authors: hully_alvesrocha@yahoo.com.br; jefferson@ufla.br; flavioborem@ufla.br

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ABSTRACT

The drying process is a very important step for a large part of the grains, because, in general, these products are harvested with high moisture contents, which favors rapid deterioration. Several physical, physiological and biochemical changes can occur in the grains during drying. Physiological analyzes have been used as indicators of this quality. Alternatives that allow reducing the drying time without changing the quality of the coffee are required. Considering that vacuum drying provides smaller exposure times, by reducing the vapor pressure, this study aimed at assessing the drying kinetics and the physiological effects caused in peeled coffee beans, when subjected to vacuum drying. The fruits were harvested at their maximum maturation potential and submitted to the removal of the peel by a mechanical process. The peeled fruits were dried in a vacuum oven under absolute pressures of 147, 447 and 747 mmHg (at the local atmospheric pressure of 747 mmHg) at 40 °C, until reaching 11 kg of water/kg of material. After drying, the grains were stored under controlled conditions of refrigerated air (10 °C and 50% relative humidity) for 30 days, standardizing drying. Coffee grains were evaluated by scanning electron microscopy and drying kinetics. For the adjustment of the mathematical models, a non-linear regression analysis was performed using the Quasi-Newton method. It was concluded that the use of vacuum significantly reduced the drying time. The model that best fit was Page Modified. Drying under absolute pressure of 447 mmHg was an interesting alternative to the conventional one, as it did not damage the cellular structures of the grain, which was similar to that obtained at the local pressure of 747 mmHg. However, the lower pressure, 147 mmHg, led to significant changes in grain quality.

Key words: Absolute pressures; Mathematical models; Scanning Electron Microscopy (SEM).

1 INTRODUCTION

In general, coffee fruits are harvested with varying moisture content between 30 kg of water 100 kg of material⁻¹ and 65 kg of water 100 kg of material⁻¹, depending on its maturation stage, being subject to rapid deterioration. Therefore, before being stored, the coffee must necessarily be dried. Although there are other methods for food preservation, drying is the most used for coffee. Among the various stages of post-harvest coffee, drying is the most relevant stage, in relation to energy consumption, processing costs and also the maintenance of product quality. (Borém; Andrade; Isquierdo, 2014; Borém; Reinato; Andrade, 2008; Dong et al., 2017). Drying air temperatures above 40 °C causes thermal damage that detracts from the quality of the coffee (Isquierdo et al., 2013; Taveira et al., 2012).

Vacuum drying is an alternative to hot air drying. It allows the evaporation of water at lower temperatures with an increase in the drying rate and a consequent reduction in drying time. On the other hand, the atmosphere in a vacuum drier presents low oxygen concentration, what reduces oxidative processes (Orikasa et al., 2014). During post-harvest, the grains are subject to deteriorating actions that compromise the quality and physiological performance, the loss of the integrity of the membranes is one of the main changes related to the deterioration process (Borém et al., 2013; McDonald, 1999;

Saath et al., 2010). The temperature of 40°C is recurrent in vacuum drying publications (Orikasa et al., 2014) and hot air drying of coffee (Alves et al., 2013, 2017), because at that temperature, the cells membrane are not degraded (Isquierdo, 2013; Livramento et al., 2017; Oliveira et al., 2013; Taveira et al., 2012).

The interest in the development of drying systems with a high drying rate, lower temperatures, which do not cause damage to the cellular structure of the beans, proposes a study of the drying of the peeled coffee submitted to vacuum and its effects, based on physiological analyzes and on the drying kinetics, as quality indicators. In this context, this work was carried out with the goal of determining the effect of the vacuum on the drying of peeled cherry coffee; evaluate the drying kinetics at the studied pressures; evaluate the fit to the drying curves with empirical models from the literature and analyze the effect of the absolute pressures of 147, 447 and 747 mmHg during drying in maintaining the integrity of the cell wall and plasma membrane of the peeled coffee.

2 MATERIAL AND METHODS

Coffee fruits (*Coffea arabica* L. cv Topázio MG 190), from the experimental crop of the Federal University of Lavras (UFLA), were harvested manually and selectively at the cherry stage and were mechanically peeled.

After harvest, the fruits were separated, in water, by density difference and the fruits with lower specific mass (dried, insect damage and mal-formed) were removed. After the hydraulic separation, a new manual selection was carried out to ensure that the samples consisted only of ripe fruits. The selected ripe fruits were mechanically peeled (wet processing). The initial water content of the grains was carried out according to the Rules for Seed Analysis - RAS (BRASIL, 2009).

Knowing the mass and the initial moisture content of the peeled coffee cherry, the drying was monitored by the gravimetric method until reaching the desired moisture using an analytical balance with a resolution of 0.01 g, according to Equation 1.

$$M_t = \frac{m_{ai} - (m_{ti} - m_{tt})}{m_{ms}} \quad (1)$$

Where M_t is the moisture over time t , m_{ai} is the mass of moisture in the fresh fruit, m_{ti} the initial total mass, m_{tt} the total mass over time t and m_{ms} the dry matter mass.

After processing, knowing the mass and the initial moisture of the peeled coffee cherry, the beans were placed in *Petri* dishes and taken to a vacuum oven (SOLAB, SL-104/40, Piracicaba, São Paulo) at a temperature of 40 °C in absolute pressures: 147, 447 and 747 mmHg, the last being the local atmospheric pressure, the drying was monitored by the gravimetric method, until the desired moisture was reached, using an analytical balance with 0.01 g resolution. After drying, the samples were stored at 10 °C and 50% relative humidity, for 30 days to perform the scanning electron microscopy analysis. The moisture ratio (M_r) was calculated by Equation 2. For all conditions tested, the values of M_r depending on the drying time, they were adjusted to describe the drying kinetics of the peeled coffee cherry.

$$M_r = \frac{M_t - M_{req}}{M_0 - M_{req}} \quad (2)$$

Where M_r is the moisture ratio, M_t is the moisture content of the product at each moment,

M_0 is the initial moisture content of the product and M_{req} , the equilibrium moisture content.

The hygroscopic equilibrium moisture was calculated by Equation 3, for peeled coffee cherry (Afonso Junior, 2002):

$$M_{req} = (2,9636 + 0,0530 * T - 10,7837 * RU^{4,5136})^{-1.6503} \quad (3)$$

Where M_{req} is the equilibrium humidity of the product, T is the temperature of the drying air and RU is the relative humidity of the drying air. Although such an equation was not obtained for vacuum drying, it is considered that its use does not affect relevant errors. The mathematical models used to adjust the drying kinetics are shown in Table 1.

The adjustment of the mathematical models was performed with nonlinear regression analysis by the Quasi-Newton method, using the STATISTICA 8.0® software (Statsoft, 2004). Among the used models, the one of Wang Singh is empirical based in a quadratic equation and all the other ones are semitheoretical and came from the general series of Fick's second law (Ertekin; Firat, 2017). The parameter k corresponds to the drying constant (time^{-1}) and n is a dimensionless empirical constant to the time term.

To verify the degree of fit of each model, the value of the coefficient of determination (R^2), root mean square error (RMSE) and chi-square (χ^2) represented by Equations 13, 14 and 15 were used:

$$R^2 = \frac{\sum_{i=1}^n (M_{rpred, i} - M_r)^2}{\sum_{i=1}^n (M_{rpred, i} - M_r)^2} \quad (13)$$

$$RMSE = \left[\frac{1}{n} \sum_{i=1}^n (M_{r\text{expi}} - M_{r\text{predi}})^2 \right]^{1/2} \quad (14)$$

$$\chi^2 = \left[\frac{\sum_{i=1}^n (M_{r\text{expi}} - M_{r\text{predi}})^2}{(N - n)} \right] \quad (15)$$

Table 1: Mathematical models used to adjust coffee drying kinetics.

Model	Equation	Reference	
Henderson and Pabis	$M_r = ae^{(-kt)}$	Henderson and Pabis (1961)	(4)
Logarithm	$M_r = ae^{(-kt)} + c$	Doymaz (2007)	(5)
Midilli	$M_r = ae^{(kt^n)} + bt$	Midilli. Kucuk and Yapar (2002)	(6)
Newton	$M_r = e^{(-kt)}$	Callaghan. Menzies and Bailey (1971)	(7)
Wang Singh	$M_r = 1 + at + bt^2$	Wang and Singh (1978)	(8)
Page Modified	$M_r = e^{-(kt)^n}$	Özdemir and Onur Devres (1999)	(9)
Diffusion approach	$M_r = ae^{(-kt)} + (1 - a) e^{(-kbt)}$	Kassem (1998)	(10)
Two Exponential Terms	$M_r = a.e^{(-kt)} + (1 - a).e^{(-kat)}$	Henderson (1974)	(11)

a, b, c, k and n are adjustment parameters and t, the time, in minutes.

For the SEM, fragments of the coffee endosperm, cut longitudinally, were used. These were placed in 1.5 ml Eppendorf tubes immersed in fixative solution (modified Karnovsky), pH 7.2 and stored in a cold chamber for 24 h. Subsequently, the samples were washed in 0.05M cacodylate buffer solution (three times of 10 min to remove glutaraldehyde residues that can reduce osmium tetroxide), later transferred to a 1% osmium tetroxide solution (3 drops) and water to cover them for one hour, they were washed three times in distilled water and subsequently dehydrated in a series of acetone (25, 50, 75, 90 and 100%, the last concentration being repeated three times) and then taken to the critical point device. The specimens obtained were mounted on aluminum supports "stubs", using carbon tape placed on an aluminum foil film, covered with gold and observed in a scanning electron microscope LEO EVO 40 XVP. An image was generated and digitally recorded for each treatment, under working conditions of 20 kV and working distance between 7 and 15 mm. The generated images were recorded and opened in the Photopaint Software of the Corel Draw 12 package, where they were selected and prepared. Based on the generated images, it is possible to observe the filling of intracellular spaces, the extravasation of the cell lumen and the formation of dots in the form of drops, to characterize the drying effect used. The adjustment of the mathematical models was performed with nonlinear regression analysis by the Quasi-Newton method, using the STATISTICA 8.0® software.

3 RESULTS

Fresh coffee fruits presented moisture of 61.38 ± 0.8 kg of water per 100 kg of sample. After drying in an oven, the grains presented moisture of 11.33 ± 0.01 kg of water 100 kg of material-1. The Figure 1 shows the drying curves of coffee beans submitted to absolute pressures 147, 447 and 747 mmHg and drying air temperature of 40 °C, depending on the moisture ratio (M_r) with time.

For the graphical representation of the drying curves in Figure 2, the Modified Page model was used because it presents a lower number of coefficients compared to the other models and mainly for obtaining a better result.

It is verified in Figure 2, by the correspondence between the experimental and estimated values, satisfactory adjustment for the description of the drying of peeled coffee cherry.

The changes caused by the drying effects on the cell membrane structures of the coffee endosperm and, observed in the digital images, are presented in the sequence. The results of the analysis of the peeled coffee endosperm after drying are shown in Figures 3, 4 and 5.

Table 2 presents the results of the adjustment parameters of the 9 models to the experimental data, as well as the determination coefficients (R^2), root mean square error (RMSE) and chi-square (χ^2).

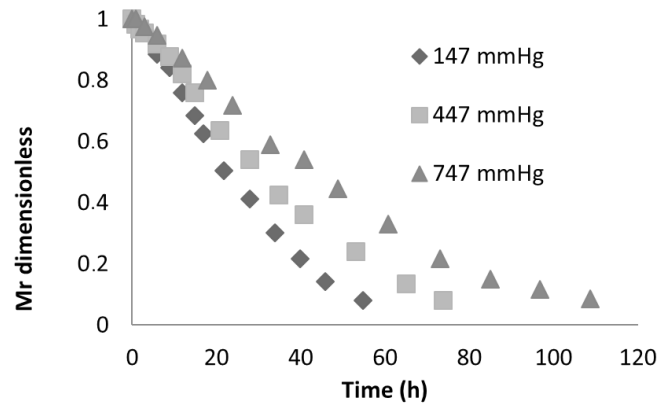


Figure 1: Drying kinetics of coffee beans using an air temperature of 40 °C.

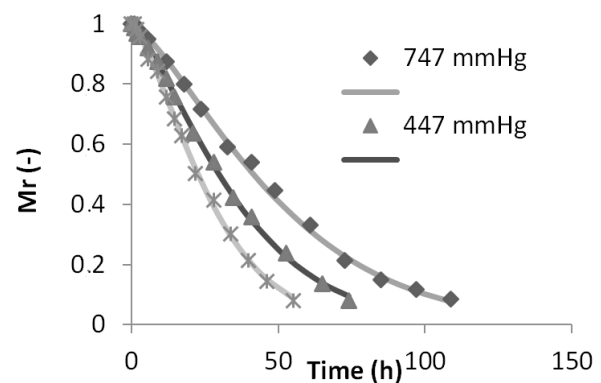


Figure 2: Experimental values of the moisture ratio (M_r) observed and estimated by the Modified Page model for drying coffee beans using the air temperature of 40 °C.

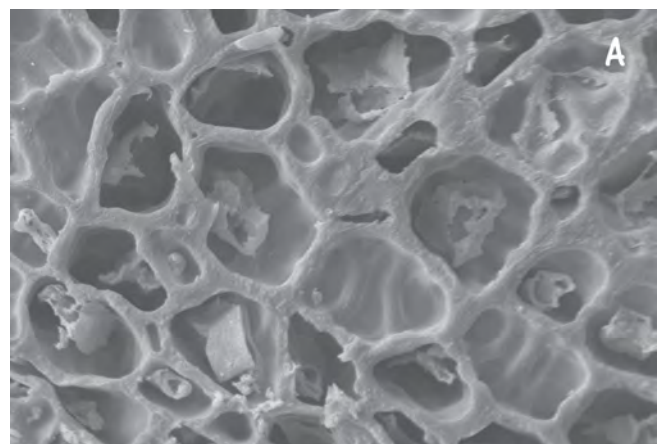


Figure 3: Scanning electromicrograph of the endosperm of the coffee (A) peeled coffee cherry after drying at an absolute pressure of 747 mmHg.

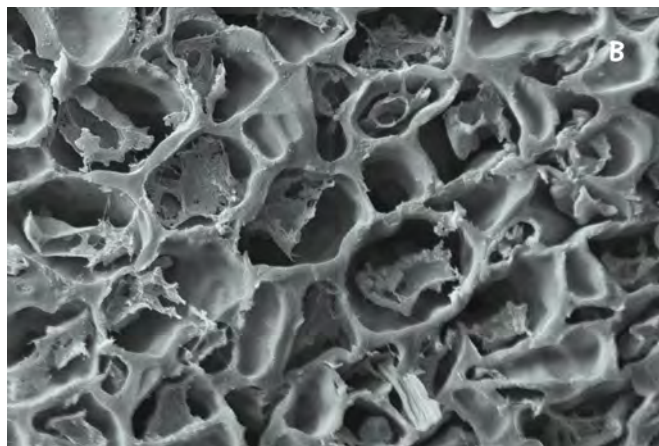


Figure 4: Scanning electromicrograph of the endosperm of the coffee (B) peeled coffee cherry after drying at an absolute pressure of 447 mmHg.

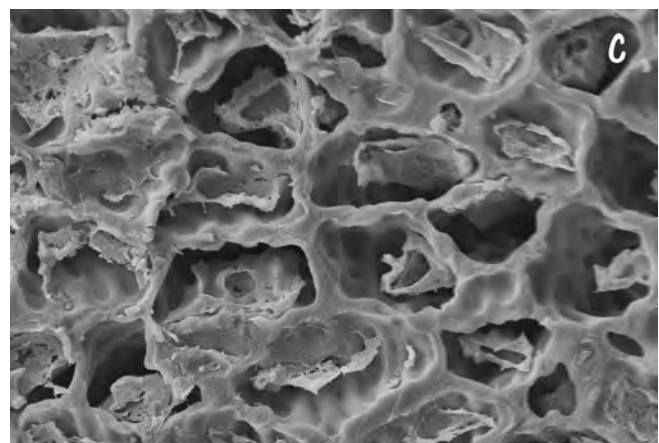


Figure 5: Scanning electromicrograph of the endosperm of the coffee (C) peeled coffee cherry after drying at an absolute pressure of 147 mmHg.

4 DISCUSSION

The decrease in absolute pressure (Figure 1) provided an increase in the drying rate and, consequently, a decrease in the drying time. The drying times were 55, 74 and 109 hours for the absolute pressures of 147, 447 and 747 mmHg (local atmospheric pressure of 747 mmHg). The reduction in drying time was 32% at a pressure corresponding to 60% of the local pressure and up to 50% at a pressure of approximately 20% of the local pressure.

The reduction in drying time when using a vacuum was also observed by Kayacan, Sagdic and Doymaz (2018) when they dried bee pollen. They compared vacuum drying with hot air drying and the reduction in vacuum drying time was significantly greater. This fact was also observed by

Horecki et al. (2018) when they compared lyophilization with vacuum drying of Cornelian cherries.

The use of reduced pressures makes it possible to decrease the drying time due to the reduction of the ambient pressure differential and the water vapor pressure (Lee; Kim, 2009). As drying is a high energy cost operation, reductions in drying time can mean a reduction in the final cost of the product.

Analyzing the values obtained regarding the determination coefficient (R^2), root mean square error (RMSE) and chi-square (χ^2), it is observed that the Modified Page model (Table 2) is the one that presents the best adjustment to the experimental drying data, with $R^2 > 0.99$, RMSE below 0.01 and χ^2 below 1.1×10^{-3} allowing its use in the prediction of the vacuum drying kinetics of peeled coffee cherry. However, all other models had a good adjustment and can also be used for this drying condition.

Siqueira et al. (2017) observed that empirical models can be used to predict coffee drying kinetics and that the Midilli model was the one that best adjusted for drying coffee, the authors also observed for those conditions that there was an increase in the drying rate at an air temperature of 40 °C. The Modified Page model was the one that best fit the vacuum drying of peeled coffee cherry. This model was also selected to predict the behavior of other products such as lemongrass (Martinazzo et al., 2010), pineapple (Waughon; Pena, 2009), smell pepper (Pontes et al., 2009), grape bagasse (Ferreira et al., 2012) and orange bagasse (Fiorentin et al., 2012).

The absolute drying pressures had an influence on the resistance and structural predisposition of the cell wall of the endosperm. The degradation of the cell membrane is more accentuated with the decrease of the pressure. However, the cell wall was kept integrated with the empty intercellular spaces. The same was verified by Saath et al. (2010) in convective drying of washed coffee with drying air temperature of 40 °C. In that work, drying peeled coffee cherry at a temperature of 40 °C did not cause any damage to the cells' cytoplasm. However, with an increase in temperature to 60 °C, a compromise was observed in the cellular structures of the grains.

The difference between dryings can be associated with the pressure to which the peeled cherry coffee fruits were subjected. It appears that, with that, the vacuum drying took place in safe conditions, preserving the integrity of the cells according to the results of (Saath et al., 2010).

In a study on different methods of drying and extracting linseed oil with vacuum drying, observed by electromicrographs, it presented a small contraction in the cell wall of the cells, however, it maintained the integrity of the same (Zhang; Liu; Che, 2018).

Table 2: Adjustment parameters of the kinetic drying models for peeled coffee cherry and determination coefficient values (R^2), root mean square error (RMSE) and chi-square (χ^2).

Model	Pressure [mmHg]	$k \times 10^3$	n	a	b	c	$R^2(\%)$	RMSE $\times 10^2$	$\chi^2 \times 10^3$
Diffusion approach	147	1.06	-	5.15	1.21	-	99.88	1.08	0.15
	447	0.21	-	5.00	0.83	-	99.09	3.00	1.13
	747	0.14	-	4.94	0.79	-	99.3	2.78	0.96
Two-exponential terms	147	0.88	-	1.95	-	-	99.85	1.22	0.17
	447	0.64	-	1.88	-	-	99.87	1.10	0.14
	747	0.46	-	1.90	-	-	99.77	1.57	0.03
Henderson and Pabis	147	0.59	-	1.07	-	-	97.96	4.53	0.24
	447	0.43	-	1.05	-	-	98.41	3.99	1.83
	747	0.32	-	1.05	-	-	98.39	4.20	2.04
Logarithm	147	0.38	-	1.35	-	-0.30	99.43	2.38	0.71
	447	0.14	-	2.19	-	-1.18	99.27	2.68	0.90
	747	0.11	-	1.93	-	-0.92	99.25	2.87	1.03
Midilli	147	0.62	0.93	1.04	-8×10^{-5}	-	99.43	2.38	0.78
	447	0.19	1.06	1.02	-3×10^{-5}	-	99.73	1.62	0.36
	747	0.61	0.86	1.04	-4×10^{-5}	-	99.3	2.77	1.04
Newton	147	0.50	-	-	-	-	96.48	5.95	3.80
	447	0.40	-	-	-	-	97.68	4.81	2.48
	747	0.03	-	-	-	-	97.70	5.04	2.72
Page modified	147	0.57	1.41	-	-	-	99.91	0.92	0.098
	447	0.42	1.34	-	-	-	99.90	0.97	0.11
	747	0.42	1.34	-	-	-	99.90	0.97	0.11
Wang Singh	147	-	-	-4.01×10^{-4}	3.4×10^{-8}	-	99.41	2.42	0.68
	447	-	-	-3.1×10^{-4}	2.3×10^{-8}	-	98.09	1.75	0.35
	747	-	-	-3.1×10^{-4}	1.3×10^{-8}	-	98.09	1.75	0.35

5 CONCLUSION

There is a vacuum effect in the drying of peeled cherry coffees. The use of vacuum reduced drying time by up to 50%. The physiological quality of grains subjected to absolute pressure of 447 mmHg was maintained, as was the use of local pressure (747 mmHg).

The modified Page model had a better fit to the drying curves; however, the other models also had an acceptable fit to the drying curves. This work can be considered introductory in the analysis of coffee drying at reduced pressures.

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