



DOI: <http://dx.doi.org/10.1590/1807-1929/agriambi.v21n1p56-60>

## Simultaneous optimization of coffee quality variables during storage

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### Key words:

*Coffea arabica* L.  
processing  
hulling

### ABSTRACT

The objective of the study was to use methodology of simultaneous optimization of multiple responses applied to an experimental design to determine the best combination of storage period and conditions for preservation of coffee beans. *Coffea arabica* L. fruits were harvested in the ripe stage of maturation, processed using wet and dry methods, and dried to 11% (wet basis) moisture content. Part of the beans was hulled, while the other part was hulled only after the beans were stored under two different environmental conditions: cooled air at 10 °C with 50% relative humidity; and at 25 °C without controlling the relative humidity. Samples were taken at 3, 6, and 12 months intervals in order to evaluate quality. The data were submitted to the simultaneous optimization of responses for each processing and hulling condition separately, in a completely randomized design and 2 x 3 factorial scheme (two storage conditions and three storage periods). In conclusion, the use of the simultaneous optimization of responses is viable to be applied for determining the ideal storage conditions in a refrigerated condition.

### Palavras-chave:

*Coffea arabica* L.  
processamento  
beneficiamento

## Otimização simultânea de variáveis da qualidade do café durante armazenamento

### RESUMO

Objetivou-se utilizar a metodologia de resposta simultânea aplicada a um delineamento experimental para determinar a melhor combinação de tempo e das condições de armazenamento para a conservação de grãos de café. Frutos de *Coffea arabica* L. foram colhidos no estágio de maturação cereja, processados por via úmida ou por via seca e secados até atingir 11% de teor de água. Parte dos grãos foi beneficiada e parte não foi beneficiada antes de ser armazenada em duas condições de ambiente: em ar refrigerado a 10 °C e umidade relativa de 50% e em 25 °C sem controle da umidade relativa do ar. Nos períodos de 3, 6 e 12 meses, foram retiradas amostras para avaliação da qualidade. Os dados foram submetidos à técnica de otimização de resposta simultânea para cada processamento e beneficiamento separadamente, em delineamento inteiramente casualizado e esquema fatorial 2 x 3 (duas condições de armazenamento e três períodos de armazenamento). Conclui-se que a utilização da metodologia de resposta simultânea é viável para ser aplicada para determinação das condições ideais de armazenamento em condição refrigerada.



## INTRODUCTION

Coffee grains are conventionally hulled and stored in sites without any control of temperature and relative humidity, favoring the deterioration, with losses in the sensory quality (Ribeiro et al., 2011; Borém et al., 2013; Rendón et al., 2013). Thus, grain mass cooling can be an alternative to prolong the storage period maintaining the quality, since favorable air conditions are important to reduce the deterioration process in stored grains (Guo et al., 2008; Rigueira et al., 2009; Quirino et al., 2013; Saath et al., 2014).

However, it is difficult to analyze the individual or joint effects of the factors involved in the quality of stored grains with the commonly used statistical techniques, because each variable may represent different scales and objectives.

The use of the desirability function employed in the technique of simultaneous optimization of responses (Derringer & Suich, 1980) is an alternative, because it determined the best combination of the studied factors (Carneiro et al., 2005). This technique considers the relative importance of each variable in relation to various intrinsic criteria, as well as the conditions of operation and/or restrictions on the responses.

Hence, this study aimed to use the methodology of simultaneous optimization of responses applied to an experimental design to estimate the best combination of storage time and conditions to preserve coffee grains.

## MATERIAL AND METHODS

The evaluations were performed in grains of *Coffea arabica* L. cv. 'Catuaí Amarelo', obtained in production fields of the Procafé Foundation Experimental Farm, Varginha-MG, Brazil. The raw material was obtained from a coffee plantation located at an altitude of 940 m, in the 2012/2013 agricultural season. The coffee fruits were harvested at the cherry stage with water content around 55% (wet basis) and part of the fruits was processed through the dry method (natural coffee), while another part was processed through the wet method (pulped coffee).

The natural coffee was dried to 30% (wet basis) and the pulp coffee to 25% (wet basis), on screens suspended on a wooden structure. From the half-dry on, the grains were transferred to mechanical fixed-bed dryers, where grain mass temperature was maintained at 35 °C until the grains reached the water content of 11% (wet basis). After drying, part of the grains was mechanically hulled to remove skin and parchment (exocarp and endocarp) using the PA-DESC coupled huller device for cleaning, processing and ventilation, from the company Palini & Alves®, while another lot was stored without hulling.

The grains were placed in polypropylene bags (permeable) and stored in two environments, cold chamber (10 ± 2 °C; 50% ± 5 RH) and in an environment at 25 ± 2 °C with no control of relative air humidity, which was monitored during the storage period, with mean value of 67.8%. In each evaluation period, the coffee grains stored without hulling were manually hulled to avoid damages. The grains were subjected to sensory, physiological and chemical evaluations at 3, 6 and 12 months of storage.

**Sensory analysis:** performed by evaluators certified by the Specialty Coffee Association of America (SCAA), according to the methodology proposed by Lingle (2011).

**Electrical conductivity and potassium leaching:** performed by adapting the methodology proposed by Malta et al. (2005), expressing the electrical conductivity in  $\mu\text{S cm}^{-1} \text{g}^{-1}$  of grains and the leached amount of potassium in ppm.

**Germination test:** performed in four replicates of 50 grains without the parchment, on germitest-type paper sheets moistened with distilled water, in a germinator regulated at temperature of 30 °C. The evaluation was performed at 30 days after sowing, expressing the results in percentage of normal seedlings, based on the recommendations of the RAS (Brasil, 2009).

**Tetrazolium test:** performed in four replicates of 25 embryos. The results were expressed in percentage of viable embryos. The methodology was adapted because of the size of the sample for the test, using half the amount of grains necessary in the test according to the methodology of Clemente et al. (2012).

**Total titratable acidity:** determined through titration with 0.1 N NaOH, adapting the methodology of the AOAC (1990). The result was expressed in mL of 0.1 N NaOH, per 100g of sample.

**Total sugars:** determined through the Antrona method (Dische, 1962) with results expressed in percentage.

**Polyphenoloxidase enzymatic activity:** determined using the second method adapted by Carvalho et al. (1994), with results expressed in  $\text{u min}^{-1} \text{g}^{-1}$  of sample.

**Statistical methodology:** The data were subjected to the technique of simultaneous optimization of responses for each condition of processing (natural and pulped) and hulling (hulled or not) separately, in a completely randomized design with three replicates in a 2 x 3 factorial scheme, with two storage conditions (10 °C and 50% of RH and 25 °C with no control of relative humidity) and three storage periods (3, 6 and 12 months). A quadratic regression model was fitted to the mean responses of three replicates. With these specifications, the model's equation is given by the parametric values to be estimated, represented by the coefficients  $\beta$ , according to Eq. 1.

$$y_j = \beta_0 + \beta_1 E_i + \beta_2 T_k + \beta_{11} E_i^2 + \beta_{22} T_k^2 + \beta_{12} E_i T_k + \xi_{ik} \quad (1)$$

where:

i - 3, 6 and 12 months (levels of the factor storage time);

k - 10 and 25 °C (levels of the factor temperature); and,

$y_j$  - mean response of three replicates for the j-th dependent variable, described in Table 1.

Table 1. Description of the variables and objectives to be achieved through the simultaneous optimization of responses

Variables ( $y_{ij}$ )	Objectives
Percentage of viable embryos (Tetrazolium)	Maximum
Electrical conductivity	Minimum
Potassium leaching	Minimum
Final score of the sensory analysis	Maximum
Polyphenoloxidase activity	Maximum
Total titratable acidity	Minimum
Total sugars	Maximum

Based on the model (Eq. 1), the best combination between the levels of storage time and temperature that achieved the desired objectives (Table 1) was fitted to each variable, using the procedure of simultaneous optimization of responses proposed by Derringer & Suich (1980). In this procedure, the “desirability” function is represented by D (Eq. 2), obtained by the geometric mean of the estimates  $d_j$  ( $j = 1, \dots, p$ ) (Eq. 3), in which  $p$  is the total number of variables.

$$D = \left( \prod_{j=1}^p d_j \right) \tag{2}$$

$$d_j = \begin{cases} 0, & \hat{y}_j < y_{jL} \\ \frac{\hat{y}_j - y_{jL}}{y_{jT} - y_{jL}}, & y_{jL} < \hat{y}_j < y_{jT} \\ \frac{y_{jU} - \hat{y}_j}{y_{jU} - y_{jT}}, & y_{jT} < \hat{y}_j < y_{jU} \\ 0, & \hat{y}_j > y_{jU} \end{cases} \tag{3}$$

where:

- $\hat{y}$  - predicted value of the  $j$ -th response;
- $y_{jT}$  - specific value for the  $j$ -th response of interest;
- $y_{jL}$  - lowest value that the desirability function will assume ( $y_{jL} < y_{jT}$ ); and,
- $y_{jU}$  - highest value that the desirability function will assume ( $y_{jT} < y_{jU}$ ).

This function aims to convert a multiple-response into a single-response problem using a normalization procedure. Thus, the minimum value assumed by  $d_j = 0$  allows to conclude that the optimal point studied is undesirable. In the case of  $d_j = 1$ , the optimum is considered desirable or satisfactory.

## RESULTS AND DISCUSSION

### Optimization of the combination of factors considering the variables for pulped coffee

In agreement with the methodology of simultaneous optimization of responses, the limits of specification were determined for each dependent variable based on the maximum and minimum values obtained in the conduction of the experiment with stratification in three levels of values (high, medium and low).

Depending on the objective of each variable (Table 1), the desirable levels were selected, whether they are high or low. Hence, Table 2 shows the description of the limits and the estimates of the determination coefficients of the model fitted to each variable, applied to pulped coffee stored with and without hulling, considering the effect of interaction between storage time and temperature.

The variables that showed values below 40% for the quality of fit, measured by the coefficient  $R^2$ , were disregarded in the optimization procedure (Table 2), except for the sensory variable, since it is the main reference in coffee quality evaluation. Regardless of the values of the coefficients  $R^2$ , the quadratic model was maintained because it describes a curvature, suggesting a point of maximum or minimum so that the gradient method shows a convergence that results in a combination that meets all the objectives (maximum and/or minimum) imposed on the variables. In addition, the criterion of selecting the same variables for pulped coffee stored with and without hulling was adopted.

The optimal combination between the levels of storage time and temperature, obtained with the application of the technique, occurred so that the fitting statistics mentioned in Eq. 2 and Eq. 3 showed results close to the optimal value (1), considered desirable. Thus, the statistical procedure was applied evaluating various scenarios between the variables with good indication of fit (Table 2), for pulped coffee stored with and without hulling. The results that led to maximum value of the global fit index, denoted by the statistics  $D$  (Eq. 2) are described in Table 3.

According to the results in Table 3, for pulped coffee stored without hulling, there are no statistical evidences to recommend

Table 3. Summary of the results referring to the desirable functions for each variable ( $d$ ) and the global desirability function ( $D$ ) obtained with the simultaneous optimization procedure applied to the dependent variables related to the pulped coffee

Variables	With hulling		Without hulling	
	$\hat{y}$	D	$\hat{y}$	D
Electrical conductivity	73.22	0.90	18.71	0.33
Total sugars	7.01	0.75	7.96	0.38
Sensory analysis	80.27	0.81	79.52	0.50
Optimal levels				
	With hulling (D = 0.82)		Without hulling (D = 0.40)	
Storage time	3 months		(6.7) 7 months	
Temperature	10 °C		10 °C	

Table 2. Limits of specification and determination coefficients of the quadratic regressions ( $R^2$ ) used in the simultaneous optimization procedure, fitted to each variable measured in the evaluation of pulped coffee

Variables ( $y_j$ )	With hulling			Without hulling		
	Limits		$R^2$	Limits		$R^2$
	Minimum ( $y_{jL}$ )	Maximum ( $y_{jU}$ )		Minimum ( $y_{jL}$ )	Maximum ( $y_{jU}$ )	
Germination percentage	0.00	0.00	-	62.33	93.50	-
Percentage of viable embryos	20.00	29.00	-	70.30	99.00	-
Electrical conductivity <sup>(1)</sup>	70.50	100.00	0.9903	15.16	20.54	0.4487
Potassium leaching (ppm)	19.66	34.54	0.8752	8.80	10.18	0.8718
Final score of sensory analysis	78.62	81.25	0.7004	79.90	81.38	0.2510
Polyphenoxidase activity <sup>(2)</sup>	36.34	37.35	0.8924	40.97	43.13	0.8194
Total titratable acidity <sup>(3)</sup>	185.00	193.30	0.7335	130.00	143.30	0.9143
Total sugars (%)	6.40	7.15	0.9657	7.84	8.16	0.8082

<sup>(1)</sup> $\mu\text{S cm}^{-1} \text{g}^{-1}$  of grains; <sup>(2)</sup> $\text{U min}^{-1} \text{g}^{-1}$  of dry mass; <sup>(3)</sup> $\text{mL NaOH (100 g)}^{-1}$  sample

optimal levels of storage time and temperature, since the statistics D was equal to 0.40. Such fact is justified by the lack of fit of the quadratic model to the sensory variable (Table 3).

For pulped coffee stored after hulling, the combination between the levels of storage time and temperature that meets the simultaneous optimization was estimated at 3 months and temperature of 10 °C, indicating that the cooling conditions in the storage are adequate for the optimization of the sensory, physiological and chemical quality of pulped coffee grains at 3 months of storage.

It is known that, in the case of specialty coffees, it is adequate a storage of at least 30 days before the product is roasted, ground and consumed. This period of rest provides greater harmonization of the chemical compounds precursors of the coffee flavor and aroma, also reducing the unpleasant, herbaceous flavor and aroma, characteristic of grains consumed without rest. Thus, one of the hypotheses for this result is that the cooling of the air down to 10 °C, for the preservation of raw coffee grains at 3 months of storage, causes the chemical compounds to be harmonized more slowly, since the low temperature reduces the speed of the reactions, causing the quality to be more well preserved under this condition.

The mechanical hulling method caused damages to the grains, reducing the initial quality before storage and contributing to the shorter time of safe storage for the coffee stored after hulling.

It has been demonstrated through research that, after the hulling of coffee grains, there is a faster reduction of viability in comparison to grains stored intact and protected by the parchment (endocarp) (Selmar et al., 2008; Rendón et al., 2013). One of the hypotheses that these authors used to explain this result is the mechanical stress to which the grains are subjected during the hulling, leading to injuries that are responsible for the more rapid decrease in grain viability. Hence, the storage in parchment is recommended to preserve the quality of the coffee processed through the wet method.

However, it is important to point out that the cited studies analyzed the variables independently and did not aim to optimize the results using statistical tools such as the procedure of simultaneous optimization of responses adopted in the present study.

### Optimization of the combination of factors considering the variables for natural coffee

The limits of specification of the analyses of the variables and the estimates of the quadratic regression model for natural

coffee stored are described in Table 4, considering the effect of interaction between storage time and temperature adjusted to each variable measured in the analyses. Again, the variables that showed low quality indices were not considered in the optimization procedure. In spite of that, the criterion of selecting the same variables for natural coffee with and without hulling was adopted.

The combination of the levels of storage time and temperature that meets the simultaneous optimization for natural coffee stored with and without hulling corresponds to 7 months of storage at temperature of 10 °C (Table 5). It is noted that the determination coefficient of the quadratic regression model, fitted to the variable Sensory Analysis (Table 4) of the coffee without hulling, showed a reasonable value, considering the criterion of R<sup>2</sup> higher than 40%. However, since this value represents an individual fit, such result did not interfere with the estimation process, because the optimal fit identified the fit of the controllable factors and that minimizes the variability of the response variable, regardless of whether the model is significant or not.

It should be pointed out that the statistics D (Eq. 2) considers the geometric means that contemplates, in a certain way, possible tendencies detected individually for each variable. Thus, in the optimization procedure in the presence of the variables (Table 5), the statistics D showed values close to the maximum, justifying its presence in the determination of the optimal condition for the natural coffee.

The optimal combination of the levels of factors was the same for the natural coffee, regardless of whether the grains were stored with or without hulling. The results, for both

Table 5. Summary of the results referring to the desirable functions for each variable (d) and the global desirability function (D) obtained with the simultaneous optimization procedure applied to the dependent variables related to the natural coffee stored

Variables	With hulling		Without hulling	
	$\hat{y}$	D	$\hat{y}$	D
K leaching	21.72	1.00	12.49	1.00
Total sugars	7.38	0.93	8.47	1.00
Sensory analysis	78.22	0.77	79.55	0.51
Optimal levels				
	With hulling (D = 0.82)		Without hulling (D = 0.40)	
Storage time	6.7 months		6.8 months	
Temperature	10 °C		10 °C	

Table 4. Limits of specification and determination coefficients of the quadratic regressions (R<sup>2</sup>) used in the simultaneous optimization procedure, fitted to each variable measured in the evaluation of natural coffee

Variables (y <sub>i</sub> )	With hulling			Without hulling		
	Limits		R <sup>2</sup>	Limits		R <sup>2</sup>
	Minimum (y <sub>l</sub> )	Maximum (y <sub>u</sub> )		Minimum (y <sub>l</sub> )	Maximum (y <sub>u</sub> )	
Germination percentage	0.00	0.00	-	38.00	57.00	-
Percentage of viable embryos	48.00	58.00	-	64.00	96.00	-
Electrical conductivity <sup>(1)</sup>	64.12	84.47	0.8874	33.80	42.22	0.8308
Potassium leaching (ppm)	21.83	28.71	0.9089	12.50	15.11	0.9477
Final score of sensory analysis	78.41	80.50	0.8806	79.91	82.50	0.5824
Polyphenoloxidase activity <sup>(2)</sup>	35.31	36.13	0.4647	38.66	39.93	0.7858
Total titratable acidity <sup>(3)</sup>	175.00	188.30	0.7243	155.00	165.80	0.8004
Total sugars (%)	6.93	7.83	0.8594	7.35	8.26	0.9814

<sup>(1)</sup>μS cm<sup>-1</sup> g<sup>-1</sup> of grains; <sup>(2)</sup>U min<sup>-1</sup> g<sup>-1</sup> of dry mass; <sup>(3)</sup>mL NaOH (100 g)<sup>-1</sup> sample

natural and pulped coffee, agree with those found by other authors who confirmed that the storage in cooled environment is effective to preserve the initial quality characteristics of coffee (Afonso Júnior et al., 2006; Rigueira et al., 2009).

However, the storage time for the optimization of the quality characteristics of natural coffee is longer in comparison to that of pulped coffee. A possible explanation for this result is the fact that the natural coffee is more susceptible to deterioration compared with pulped coffee (Taveira et al., 2012; Saath et al., 2014). Because of that, there is a more rapid reduction of its quality characteristics in the storage, compared with pulped coffee, increasing the period for the optimization of the variables.

Since the beginning of the storage, the quality of the natural coffee showed a sharp decrease, reaching a level at which all values of the quality attributes are so low that the maximum values for all variables coincide, leading to an optimized combination.

## CONCLUSIONS

1. The use of the simultaneous response methodology is viable to determine the ideal storage conditions under cooling, for being flexible and contemplating the experimental model in different objectives of optimization.

2. Temperature of 10 °C and storage period of seven months for natural coffee and three months for pulped coffee constitute the combination obtained by the simultaneous optimization aiming at quality preservation.

3. Regardless of the processing method and hulling condition, the temperature of 10 °C is favorable to the best preservation of coffee grains.

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