CERTIFICATION

To whom it may concern:

I, José Fernando Jaramillo Sanint, certify, under penalty of perjury, that I have translated the foregoing documents, from Spanish into English to the best of my ability. I also declare that I am a competent English-Spanish-English Official Translator and Interpreter, as certified by means of Resolution No. 0499 dated April 02, 2004 issued by the National Ministry of the Interior and Justice of Colombia (now National Ministry of Home Affairs and Justice), duly sworn, appointed and registered before the corresponding authorities in Colombia. As such, I am only liable for those translations of documents bearing my signature and seal on each page.

Issued on March 14, 2019, in Bogotá, Cundinamarca, Colombia.

José Fernando Jaramillo Sanint
Resolution No. 0499 of April 02, 2004
National Ministry of the Interior and Justice of Colombia

JOSÉ F. JARAMILLO SANINT
Traductor e Intérprete Oficial
Inglés Español Inglés
Resolución Nro. 0499
Ministerio de Justicia

INFRARED SPECTROSCOPY, THERMOANALYSIS AND DIFFUSION COEFFICIENTS FOR THE ENDOCARP OF A CASTILLA VARIETY COFFEE SAMPLE

Abstract

This article presents the main results of thermal analytical and drying tests applied to the endocarp of coffee bean samples, in order to analyze their influence on the coffee dehydration process. An infrared analysis, as well as TGA, DTGA and DSC tests, were applied to the parchment of a sample of Castilla variety coffee beans and later compared with similar tests performed on coffee beans of the same variety, upon parchment removal. For analytical tests, the main thermogravimetric transitions are reported up to a temperature of 1000 °C. The study was complemented by drying experiments on samples of beans with and without parchment. The diffusion coefficients were found using Fick's second law and metaheuristic optimization methods (global optimization). On average, the diffusion coefficient of grains without endocarp is 46% greater than that of beans dried with the parchment. The results are considered important for the projection and design of new coffee drying systems and their automatic control.

Keywords

Coffee drying, thermogravimetric analysis, diffusion coefficient, global optimization

1. INTRODUCTION

The drying of the coffee bean is a fundamental stage of its postharvest handling. A good drying guarantees not only an adequate conservation of the grain during the period of storage, prior to roasting, but also a good cup quality [1-4]. In general, the drying of the coffee beans takes place after they are pulped, fermented and washed, in a process known as wet processing. In depulping, the epicarp or skin and the mesocarp or pulp are removed (see Figure 1), and during the wash, the mucilage is removed, too. In this way, the drying of grain involves the removal of the underlying moisture to the grain, and the removal of the endocarp or parchment that still covers it, as well.

Removing the grain parchment before drying is not easy, given its high moisture content (between 55% and 60% b.h., [5] [6]). Besides, producers consider the parchment as protection for dry grains, which must be transported from the production centers to the collection centers, although, it must finally be removed before the product is exported. The components of the dried parchment (or hulls) residue, are mainly cellulose (40-49%), hemicellulose (25-32%) and lignin (33-35%) [7] [8]. The hull is valued for its potential as combustible material, with a 17.9MJ / kg ([9]) report on its calorific value.

The objective of the study reported in this article is to analyze the way in which the endocarp influences the drying process of the grain, through the revision of some of its thermal characteristics. For this, the parchment from a sample of Castilla variety coffee beans collected in the southeastern region of the department of Santander, in Colombia, was subjected to a series of thermo-analytical tests, while experimental curves of drying grain, with and without it, were determined. The thermal analysis was compared with results of similar tests made to the single grain, and the differences between the grain drying kinetics, with and without parchment, were contrasted based on the determination of diffusion coefficients. This exploration is important as a preliminary phase on the study of new drying techniques, which might consider the extraction of moisture in endocarp free seeds. The methodology addressed, as well as the main results obtained, are detailed below.

2. METHODS

2.1 Thermal Analysis

For the thermal analysis, a sample of parchment from Castilla Variety coffee beans was subjected to thermo-gravimetry (TGA), differential thermogravimetry (DTGA) and differential scanning calorimetry (DSC) tests, at a heating rate of 5 °C / min. The analysis was carried out in an inert nitrogen atmosphere, in a NETZSCH device (STA 449 F5 JUPITER). The system was also coupled with an infrared (IR) equipment, brand BRUKER. In the process, the ASTM E1131-08 and ASTM E1269-11 standards were followed. The results were compared with the thermal analysis made by the authors to the grains without endocarp as shown in [10].

2.2 Diffusion Coefficients

Diffusion coefficients were estimated for beans with parchment, without parchment, and for a parchment-only sample, after obtaining the corresponding drying curves. A total of 10 experiments were carried out, 4 for beans with parchment, 4 for parchment free samples and 2 for parchment-only samples. The mass of the samples was 63.5g. The samples were hydrated with distilled water for a period of 48 hours. Subsequently, the already fermented samples were also washed with distilled water, and left to rest for 25 minutes. In this way, the fermentation and washing process carried out by the producers was emulated, prior to drying.

JOSÉ F. JARAMILLO SANINT
Traductor e Intérprete Oficial
Inglés Español Inglés
Resolución Nro. 0499
Ministerio de Justicia

To obtain the drying curves, the samples were dried in a convection electric oven at a controlled temperature of 50 ± 2 °C, arranged in a single layer on an aluminum plate. The moisture loss was calculated based on (1) and (2), with M_t the residual moisture of the grain at time t, m_0 the initial mass of the grain, m_t the mass of the grain at time t, M_0 the initial humidity of the grain, and m_d the dry mass of the sample, which was calculated by drying the samples at a temperature of 104 °C, for one hour. The dry mass for the samples of beans with parchment was calculated at 30.13g; for the parchment free samples in 31.28g; and for the parchment-only samples in 21.01g. The humidity on dry basis M was determined with (4). The first three measurements of mass were taken every 20 minutes, the next two every 30 minutes, and the remaining ones every hour, until obtaining a humidity in the dry basis of less than 12%.

Following the second law of Fick, and the fluid continuity equation [11], the loss of humidity in the grain with respect to time (*M*, in dry basis), is determined using (4).

In equation (4), *D* is the effective diffusion coefficient. Assuming a spherical geometry for the samples, the solution of (4) leads to (5).

In (5), MR is the dimensionless moisture relation, M_0 is the initial moisture in dry basis, M_e the equilibrium moisture, also in dry basis, and *r* the approximate radius of the grains. The time, *t*, was measured in minutes, and the radius, *r*, in millimeters, so *D* contains mm^2 / min units.

A more general solution [12] [13] is detailed in (6), from which the effective coefficient of diffusion *D* is cleared by dividing the first factor, k_i, between π^2 and multiplying it by r^2 (the approximate radius of the grain squared).

With the real data obtained from the drying tests, four global optimization algorithms were experimented (Drone Squadron Optimization, DSO, Trust Region with Dogleg, TRD, Genetic Algorithms, GA, and Particle Swarm Optimization, PSO), to find the parameters A_i , k_i and M_e of (6). The objective function in the algorithms was used (7), in which M^* follows the form (6) and *M* is the vector of the N real data of the drying process. From the preliminary trials the best results were obtained with GA, so this algorithm was used for the calculations in all the samples. The data estimated by the model were compared with the real data through the measurement of the root of the mean square error (8), and the coefficient of determination (9).

3. RESULTS AND DISCUSSION

JOSÉ F. JARAMILLO SANINT
Traductor e Intérprete Oficial
Inglés Español Inglés
Resolución Nro. 0499
Ministerio de Justicia

3.1 Results

Figures 2 and 3 show the thermograms for the parchment and for the coffee bean, respectively. For the parchment, four areas of interest are differentiated, while for the grain, three zones are clearly differentiable. Table 1 summarizes the temperature ranges that identify these zones and the mass losses that occur in them.

The differential thermogram (DTGA) of the parchment is presented in Figure 4. A single transition can be noticed at 346 °C, which corresponds to the loss of cellulose and hemicellulose, its major constituent parts. In the differential thermogram of the grain (see Figure 5), the dynamic range is greater, and the loss of these same components is observed as a transition between 331.4 °C and 358.7 °C. In differential thermogram [10] the range between 219 °C and 254 °C stands out, considering it corresponds to the range where the sublimation of caffeine occurs. This information is relevant for the roasting process.

Figures 6 and 7 correspond to the DSC of the parchment and grain. The most outstanding transitions are summarized in Table 2.

Complementary to the previous analysis, an infrared spectrum was obtained from the IR coupling made in thermogravimetric tests. The acquired spectrum was compared with the Bruker database. Figure 8 shows the spectrum of the parchment at 115 °C, and Figure 9 the grain spectrum without scale at 124 °C.

The drying curves, from which the diffusion coefficients were calculated, are shown in Figures 10 and 11. In Figure 12, a box and whisker diagram of the average dimensionless moisture value is presented (MR), for each type of drying experiment carried out (beans with parchment, parchment free beans, and parchmenet-only samples). Table 3 summarizes the results of the tests with the optimization algorithms used to find the parameters of (6), and table 4 contains the calculation of the diffusion coefficients D and the parameters of (6), for all experiments performed using the GA algorithm.

3.2 Discussion

The drying of the coffee beans is carried out regularly at temperatures between 50 °C and 60 °C. The thermograms and the infrared analysis show that, at these temperatures, the processes in the samples are totally endothermic, and the main component due to mass loss is water. Thus, parchment and grain compete to consume the energy required to dehydrate. In fact, during the drying tests carried out, it is noted how in the beans with parchment assays, it took around 17 hours to reach moisture values in dry basis lower than 12%, while when drying the parchment-free beans that time is reduced to about 13 hours. On the other hand, the diffusion coefficient of grain drying for parchment free beans, is on average 46% higher than that of the dried grain with its endocarp, and the coefficient of the parchment-only samples is 10% higher than that of the parchment free beans. Likewise, the optimization methods rendered very similar results, although GA had the least computation time.

JOSÉ F. JARAMILLO SANINT
Traductor e Intérprete Oficial
Inglés Español Inglés
Resolución Nro. 0499
Ministerio de Justicia

4. CONCLUSIONS

The experiments carried out show that the diffusion of water in the dehydration of coffee beans is notoriously diminished by the presence of its endocarp, while drying without it does not alter its composition, and reduces time and energy. Additional studies are required to determine what type of system can make it feasible for pre-drying to facilitate the early removal of the endocarp, to achieve more efficient drying. Similarly, the optimization algorithms used for the determination of the diffusion coefficient offered quite acceptable results, without having to resort to solving the non-linear equation that defines it.

JOSÉ F. JARAMILLO SANINT
Traductor e Intérprete Oficial
Inglés Español Inglés
Resolución Nro. 0499
Ministerio de Justicia