



# Analysis of coffee thermophysical changes during roasting using differential scanning calorimetry

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## Abstract

The main objective of the present study was to evaluate the variation of dependency between physical green coffee beans characteristics and thermal effects during roasting. In this work was analyzed coffee roasting process, used differential scanning calorimetry able to describe thermophysical processes inside a coffee bean during the roasting process was developed. For the analysis of thermophysical effects inside the coffee bean were explored six samples of coffee beans with different origins. The scanning calorimetry data obtained showed differences in thermal effects, the transition from an endothermic process to an exothermic one, as well as the amount of heat required for the roasting process. The analyzed humidity and density curves were then compared with the observed data. The analysis showed a direct dependency between the physical characteristics of the grain and the thermal effects during the roasting process. The present study concludes that green coffee with different properties requires the use of different strategies for the use of energy to obtain a quality product.

**Keywords:** coffee; heat effects; DSC; Maillard reaction.

**Practical Application:** This present work has such practical applications as: the use of differential scanning calorimetry as a new technique for the study of coffee; provide knowledge and understanding of thermophysical processes during heating of coffee beans; study of the influence of the physical properties of coffee on the roasting process.

## 1 Introduction

Coffee is a commodity that competes for position in the world market. Its production and commercialization is a constant increase during the last years to the point of being located as one of the most tradable goods in the world (Celso, 2022). Consumers, who drink coffee more often showed high expectations for sensory analysis (Bressani et al., 2021).

The taste and distinctive organoleptic qualities of coffee vary greatly around the world due to the influence of genetic strains, geographical location, climatic conditions, various agricultural practices and variations in post-harvest processing methods, making the flavor or aroma of coffee possibly its most important component (Sunarharum et al., 2014). Functional and sensory properties are the two most important factors that influence consumer attractiveness to coffee drinks. Strictly controlled changes in the post-harvest processing of coffee beans can affect the taste characteristics of roasted coffee (Pereira et al., 2021a). Post-harvest processing affects the physicochemical characteristics of coffee beans. Physical characteristic of green bean, especially bulk density, color, are affected by postharvest processing (Yulianti et al., 2022).

However, after post-harvest process have been completed, the coffee needs to be roasted. This process proceeds at an initial temperature of 120 °C and ends from 180 to 200 °C. In the course of this relatively simple process, a series of events occur in line with the roasting process, causing the green bean to completely change its structure to release the coffee-forming compounds in the cup (Pereira et al., 2021b). It creates the organoleptic characteristics

of coffee, which is the most important factor influencing the consumer appeal of a coffee drink (Santoso et al., 2022).

Roasting is a crucial stage in the coffee processing, that is aimed to change markedly the chemical, physical, structural and sensorial properties of the green beans by heat induced reactions. In this way roasting process makes coffee beans suitable for brewing. The green beans are, in fact, characterized by only a weak and greenly aroma and a hard texture hinders their use as food (Fabbri et al., 2011a)

Roasting is a complex process involving both energy (from the roaster to the bean) and mass (water vapor and volatile compounds from the bean to the environment) transfer implied in the main changes of the coffee beans in terms of weight, density, moisture, color and flavor (Eggers & Pietsch, 2001).

The physical and chemical properties of roasted coffee are highly dependent on the conditions of the roasting process, in particular temperature and time conditions inside the coffee bean depending on the heat transfer (Baggenstoss et al., 2008). Different time-temperature led to distinct aroma compounds in the coffee profiles industry (Schenker et al., 2002). But excessive roasting generally takes to decreasing or stable amounts of volatile substances, except for hexanal, pyridine, and dimethyl trisulfide, whose concentrations continued to increase during over-roasting (Baggenstoss et al., 2008). Events such as chlorophyll breakdown and water evaporation occur in the early stages of roasting, while unwanted exothermic pyrolysis of saccharides can occur in the later stages of roasting (Fabbri et al., 2011b).

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Previous studies show that the first phase occurs at temperatures below 160 °C, and the second phase occurs up to 250...260 °C. At the last stage, pyrolysis inevitably begins. Pyrolytic processes occur at 190 °C and are accompanied by oxidation, reduction, hydrolysis, polymerization, decarboxylation and many other chemical changes leading to the formation of substances that, among other things, are necessary to give the taste of coffee. After this second step, the beans must be cooled quickly to stop the reactions (using water or air as a cooling agent) and prevent over-roasting, which changes the quality of the product (Hernández et al., 2007).

There are various models that describe the thermophysical changes in the grain during coffee roasting. These models rely on variables such as the density and consumption of the product, as well as the physical and chemical properties of the coffee. The above variable mechanisms change depending on the origin and mode of post-harvest processing. At the same time, the same models can give different degrees of error, and also have different predictive power, which depends on a high estimate of the studied physical properties (humidity, density, etc.).

Differential scanning calorimetry (DSC) is an effective analytical tool for characterizing the physical properties of a polymer. DSC enables determination of melting, crystallization, and mesomorphic transition temperatures, and the corresponding enthalpy and entropy changes, and characterization of the glass transition and other effects which show either changes in heat capacity or a latent heat. In food products, due to the heterogeneous nature of the products, it is difficult to perform a mathematical description of phase transitions (Grajales-Lagunes et al., 2018). The Flash DSC 1 enables to mimic realistic conditions of practice and to measure (meta)stability and reorganization phenomena of substances and materials (Pyda et al., 2011). Dynamic effects are due to the formation of a temperature profile during scanning (Schawe, 2015).

DSC belongs to a group of thermal analysis based on measuring the heat flux difference between the sample and the reference substance, that is, the energy required to equalize the temperature between the sample and the reference substance, during heating or cooling of the sample, under controlled conditions (Grujić & Savanović, 2019).

## 2 Materials and methods

### 2.1 Materials

In present work there was twelve different green coffee samples from various origins: Brazil, Colombia, Ethiopia, Thailand. Samples includes two types of coffee: *C.Arabica* and *C.Conilon*. Each sample coffee has a number of International

Coffee Organization (ICO) for traceability. All of samples was coded for present work as well (Table 1).

### 2.2 Proximate analysis

Analysis of the density and humidity of the samples was carried out by the express method using the equipment Lightells MD-500. The measurement error is not more than 0.5% of humidity measuring and 1.2 g/L density measuring.

### 2.3 Differential Scanning Calorimetry (DSC)

DSC studies were carried out on a NETSCH-204 calorimeter. Nitrogen was used as a purge gas. The instrument was calibrated for heat flow, temperature and baseline using standard Tzero technology. The calibration in temperature was carried out using standards such as sapphire, and the calibration in energy was carried out using standard like sapphire. To improve the accuracy of the measurements, Tzero aluminum pierced lead with the same mass ( $\pm 0.01$  mg) have been used in this work.

Each measurement uses two lidded crucibles made from the same material and of approximately identical masses. One crucible serves as an empty and inert reference and the other holds the sample. The crucibles sit on separate sensors, connected by a thermocouple. When heated in a shared environment any difference in heating rates produces an electrical signal via the thermocouple which is captured by the instrument, is shown in Figure 1.

Each sample was prepared by cross sectioning a 10 mg embryo and was heated from 20 to 250 °C, which corresponds to the temperature range during the coffee roasting process. Heating was carried out at a constant rate of 10 k/min.

## 3 Results and discussion

The research results physical properties and differential scanning calorimetry with samples heating from 20 °C to 250 °C, are given in Tables 2-3 and Figures 2-3.

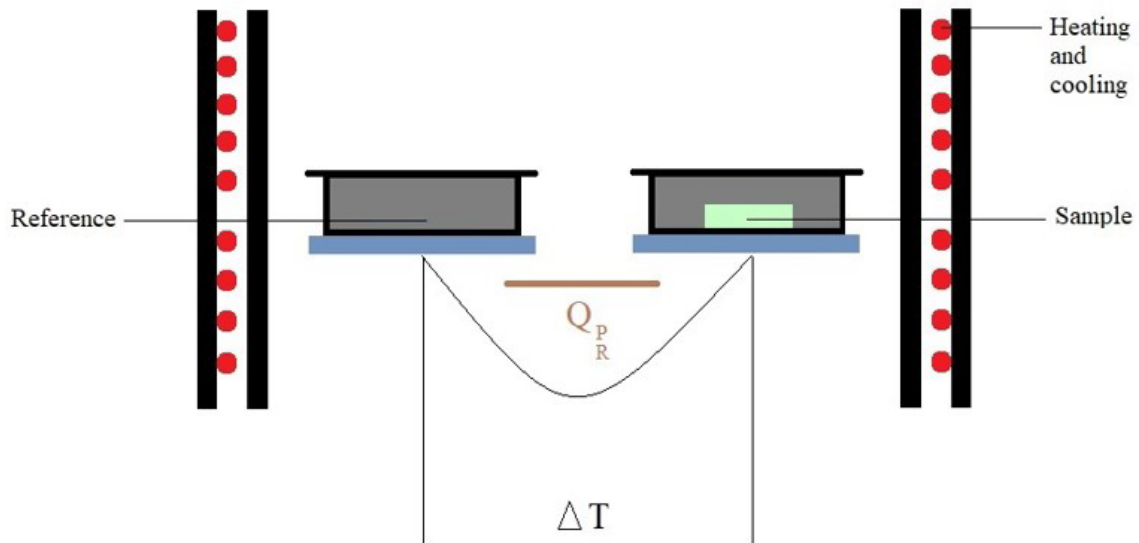
The correlation between density and humidity of coffee samples, is shown in Figure 1.

Moisture and density analysis showed significant differences between samples of different origins. The curves show a correlation between humidity and density of different green beans.

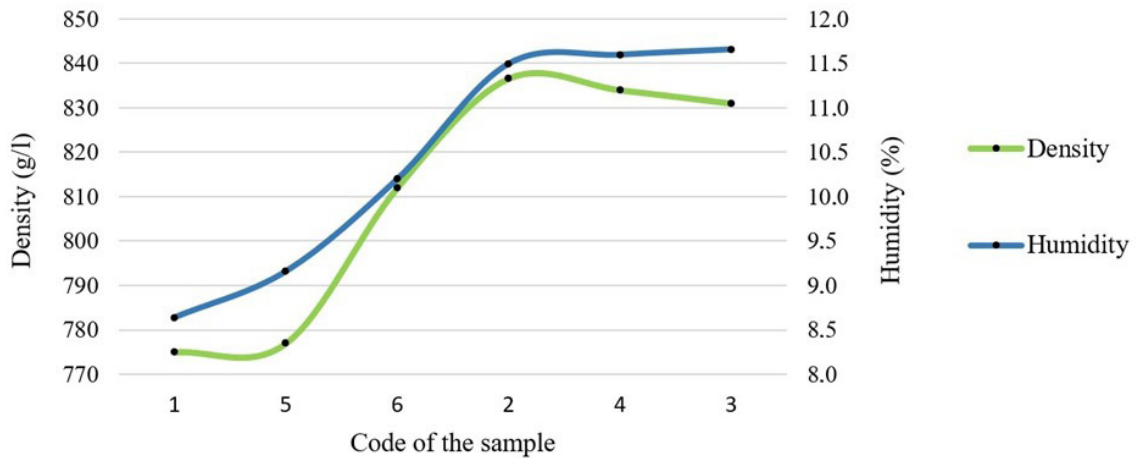
Sample 3 and sample 4 showed the highest density (*C. Arabica* from Colombia), as well as the Sample 2 (*C. Canephora* from Brazil), which may be due to the characteristics of the genetic variety. These samples have the highest moisture content. The Sample 1 (Brazilian *C. Arabica*) has the minimum indicator of humidity and density.

**Table 1.** Coffee samples background.

Code	Nº ICO	Country	Region	Variety	Postharvest process
1	002/1671/0001	Brazil	Serra do Cabral	C.Arabica	Dry
2	002/2131/0014	Brazil	Caparaó	C.Canephora	Semiwashed
3	3/0265/00099	Colombia	Huila	C.Arabica	Wet
4	3/0265/00207	Colombia	Nariño	C.Arabica	Wet
5	010/0590/0057	Ethiopia	Gelana Abaya	C.Arabica	Wet
6	140/6022/088	Thailand	Chiang Rai	C.Arabica	Black Honey



**Figure 1.** Principle of measurement on a differential scanning calorimeter.



**Figure 2.** Correlation between density and humidity of coffee samples.

**Table 2.** Physical properties of coffee samples.

Code	Density, g/L	Humidity, %
1	775 ± 1.2	8.6 ± 0.5
2	836 ± 1.2	11.5 ± 0.5
3	831 ± 1.2	11.7 ± 0.5
4	834 ± 1.2	11.6 ± 0.5
5	777 ± 1.2	9.2 ± 0.5
6	812 ± 1.2	10.2 ± 0.5

**Table 3.** DSC values at control points.

Code	Onset (°C)	Peak (°C)	End (°C)	Area of the peak (μV/mg)
1	44.6 ± 0.1	207.4 ± 0.1	157.3 ± 0.1	625.5 ± 0.5
2	39.2 ± 0.1	218.7 ± 0.1	148.5 ± 0.1	691.4 ± 0.5
3	44.9 ± 0.1	211.8 ± 0.1	158 ± 0.1	730.7 ± 0.5
4	42.8 ± 0.1	209 ± 0.1	156.3 ± 0.1	708.7 ± 0.5
5	49.8 ± 0.1	208.7 ± 0.1	153.7 ± 0.1	635.7 ± 0.5
6	45 ± 0.1	211.1 ± 0.1	153.6 ± 0.1	642.7 ± 0.5

The DSC thermogram shows the thermal effects of the samples from 20 °C to 250 °C, obtained by heating at a rate of 10 °C/min in a constant nitrogen atmosphere.

Thermograms obtained for all samples (Figure 3) showed the following events: an initial endothermic phase followed by an exothermic phase. In this case, the change in the nature of the process from endothermic to exothermic occurs at different temperatures for each sample.

Any anomaly thermophysical effect of DSC can be fully described the following parameters: the temperatures of onset of the peak (Onset), which characterizes the beginning process, and the end of the peak (End); temperature of the maximum (minimum) peak which characterizes the end of the process; the peak area, which determines the enthalpy (specific heat) of the process. This indicator is characterized by the ratio amount absorbed/released heat to the mass of the test substance. At these points, values were found in the analysis of DSC (Table 3).

For samples 2 and 4, an early onset of an active endothermic process was observed with peaks at 148.5 °C (sample 2) and 156.3 °C (sample 4). The peak as an event is associated, among other things, with the onset of the Maillard reaction. The reaction is a form of non-enzymatic browning that usually occurs between 140 and 165 °C and is dependent on many factors.

The data obtained were also compared with the physical characteristics of green coffee. As a result, a correlation was found between density and the amount of heat required for the process to proceed (peak area), as evidenced by Figure 4.

The establishment of such a correlation between physical characteristics suggests that green coffee, which has a higher density, requires the use of more heat during the roasting process.

This study shows that the dynamics of the physical characteristics of coffee changes significantly during the roasting process, which is due to the endothermic and exothermic transformations that occur inside the coffee bean. As a result, these changes directly affect the formation of the organoleptic characteristics of coffee.

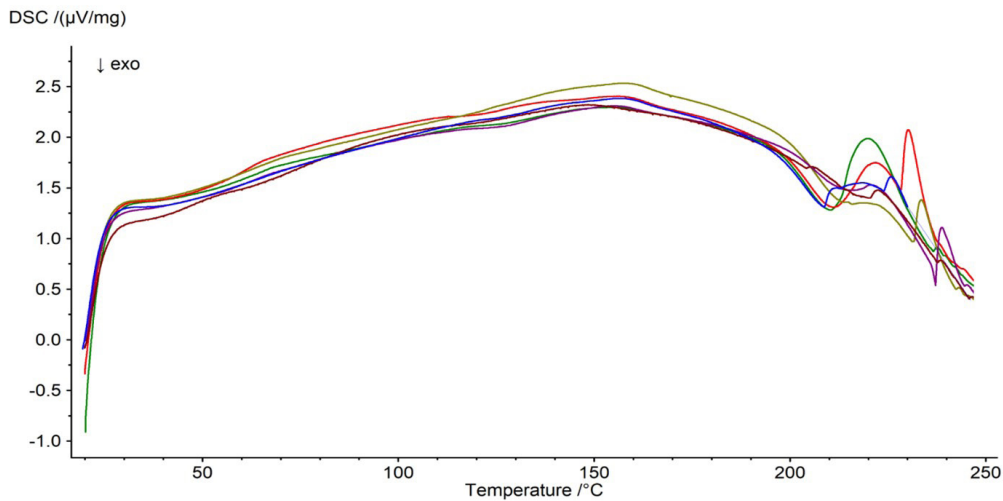


Figure 3. Thermograms of differential scanning calorimetry of coffee samples.

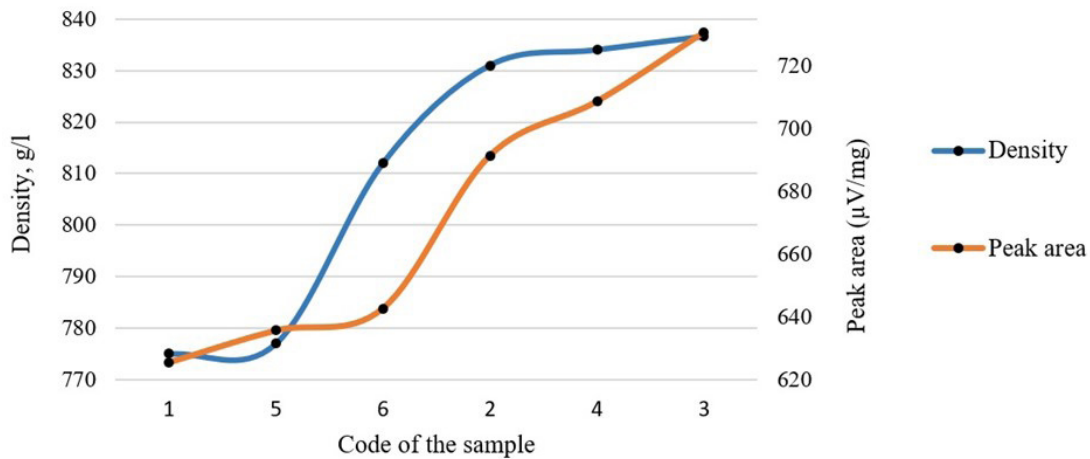


Figure 4. Correlation between density and peak area of coffee samples.

## 4 Conclusion

The main objective of the present study was to evaluate the variation of dependency between physical green coffee beans characteristics and thermal effects during roasting. The evaluated characteristics were: density, humidity, heat effects. The results show that heat quantity needs for heating as more, as higher a density and humidity of samples. Moreover, a point of changing from endothermal to exothermal character of process changed depends of beans physical parameters in the same correlation.

The present study concludes that green coffee with different characteristics requires the use of different strategies for the use of energy to obtain a quality product. Due to the nature of the chemical composition of green coffee, which also has a direct impact on the characteristics of the roasting process, the next step for further research is to look for relationships between the chemical components of coffee and the characteristics of thermal effects when heated. The results of this study can significantly contribute to the development of food science in the field of coffee processing.

This work is in line with the direction of further research and development in this area.

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## References

- Baggenstoss, J., Poisson, L., Kaegi, R., Perren, R., & Escher, F. (2008). Coffee roasting and aroma formation: application of different time-temperature conditions. *Journal of Agricultural and Food Chemistry*, 56(14), 5836-5846. <http://dx.doi.org/10.1021/jf800327j>. PMID:18572953.
- Bressani, A. P., Martinez, S. J., Batista, N. N., Simão, J. P., & Schwan, R. F. (2021). Into the minds of coffee consumers: perception, preference, and impact of information in the sensory analysis of specialty coffee. *Food Science and Technology*, 41(Suppl. 2), 667-675. <http://dx.doi.org/10.1590/fst.30720>.
- Celso, R. R. (2022). Competitiveness of the Mexican coffee in international trade: a comparative analysis with Brazil, Colombia, and Peru (2000-2019). *Análisis Económico*, 37(94), 181-199.
- Eggers, R., & Pietsch, A. (2001). Roasting. In R. J. Clarke & O. G. Vitzthum (Eds.), *Coffee: recent developments* (pp. 90-107). London: Blackwell Science. <http://dx.doi.org/10.1002/9780470690499.ch4>.
- Fabbri, A., Cevoli, C., Alessandrini, L., & Romani, S. (2011a). Numerical modeling of heat and mass transfer during coffee roasting process. *Journal of Food Engineering*, 105(2), 264-269. <http://dx.doi.org/10.1016/j.jfoodeng.2011.02.030>.
- Fabbri, A., Cevoli, C., Romani, S., & Rosa, M. D. (2011b). Numerical model of heat and mass transfer during roasting coffee using 3D digitised geometry. *Procedia Food Science*, 1, 742-746. <http://dx.doi.org/10.1016/j.profoo.2011.09.112>.
- Grajales-Lagunes, A., Rivera-Bautista, C., Loredó-García, I. O., González-García, R., González-Chávez, M. M., Schmidt, S. J., & Ruiz-Cabrera, M. A. (2018). Using model food systems to develop mathematical models for construction of state diagrams of fruit products. *Journal of Food Engineering*, 230, 72-81. <http://dx.doi.org/10.1016/j.jfoodeng.2018.02.025>.
- Grujić, R., & Savanović, D. (2019). Thermal analysis of food products using differential scanning calorimetry (DSC). *Contemporary Materials X-2*, 10(2), 176-181. <http://dx.doi.org/10.7251/COMEN1902175G>.
- Hernández, J. A., Heyd, B., Irlés, C., Valdovinos, B., & Trystram, G. (2007). Analysis of the heat and mass transfer during coffee batch roasting. *Journal of Food Engineering*, 78(4), 1141-1148. <http://dx.doi.org/10.1016/j.jfoodeng.2005.12.041>.
- Mathot, V., Pyda, M., Pijpers, T., Poel, G. V., van de Kerkhof, E., van Herwaarden, S., van Herwaarden, F., & Leenaers, A. (2011). The Flash DSC 1, a power compensation twin-type, chip-based fast scanning calorimeter (FSC): first findings on polymers. *Thermochimica Acta*, 522(1-2), 36-45. <http://dx.doi.org/10.1016/j.tca.2011.02.031>.
- Pereira, L. L., Cherkasova, E. I., Golnitskiy, P. V., Toygambaev, S. K., & Mutovkina, E. A. (2021a). Processing modes influence on the sensory profile of various types of coffee. *IOP Conference Series. Earth and Environmental Science*, 677(5), 052036. <http://dx.doi.org/10.1088/1755-1315/677/5/052036>.
- Pereira, L. L., Debona, D. G., Pinheiro, P. F., Oliveira, G. F., ten Caten, S., Moksunova, V., Kopanina, A. V., Vlasova, I. I., Talskikh, A. I., & Yamamoto, H. (2021b). Roasting process. In Pereira, L. L., & Moreira, T. R. (Eds.), *Quality determinants in coffee production* (pp. 303-308). Cham: Springer. [http://dx.doi.org/10.1007/978-3-030-54437-9\\_7](http://dx.doi.org/10.1007/978-3-030-54437-9_7).
- Santoso, B., Wijaya, A., & Pangawikan, A. D. (2022). The addition of crude gambir extract in the production of functional robusta coffee powder. *Food Science and Technology*, 42, e55721. <http://dx.doi.org/10.1590/fst.55721>.
- Schawe, J. E. K. (2015). Measurement of the thermal glass transition of polystyrene in a cooling rate range of more than six decades. *Thermochimica Acta*, 603, 128-134. <http://dx.doi.org/10.1016/j.tca.2014.05.025>.
- Schenker, S., Heinemann, C., Huber, M., Pompizzi, R., Perren, R., & Escher, R. (2002). Impact of roasting conditions on the formation of aroma compounds in coffee beans. *Journal of Food Science*, 67(1), 60-66. <http://dx.doi.org/10.1111/j.1365-2621.2002.tb11359.x>.
- Sunarharum, W. B., Williams, D. J., & Smyth, H. E. (2014). Complexity of coffee flavor: a compositional and sensory perspective. *Food Research International*, 62, 315-325. <http://dx.doi.org/10.1016/j.foodres.2014.02.030>.
- Yulianti, Y., Andarwulan, N., Adawiyah, D. R., Herawati, D., & Indrasti, D. (2022). Physicochemical characteristics and bioactive compound profiles of Arabica Kalosi Enrekang with different postharvest processing. *Food Science and Technology*, 42, e67622. <http://dx.doi.org/10.1590/fst.67622>.