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THERMAL ANALYSIS OF COFFEE BEANS OF CASTILLA VARIETY GROWN IN COLOMBIA

ANÁLISIS TÉRMICO DE GRANOS DE CAFÉ VARIEDAD CASTILLA CULTIVADO EN COLOMBIA

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Abstract
This article presents the main results achieved after thermally characterizing the coffee beans of the Castilla variety. Such beans were produced in the Southeastern region of Santander, Colombia. Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) tests were carried out. We determined various stages of thermal degradation within the beans, along with their thermal transitions. We also determined the variation of heat capacity throughout the process. Thermal characterization allowed us to carry out an analysis of the resulting gases, using infrared (IR) spectroscopy. We check the resulting experimental data against the Bruker IR database and we were able to detect three sectors within the coffee bean that exhibited a loss of mass, for a heating rate of 5 °C/min, and for temperatures up to 1000 °C. The zones were in the following regions: 58.5 °C and 324 °C, with a mass loss of 50.26%; 324 °C and 432 °C, with a mass loss of 14.67%; and 432 °C and 513 °C, with a mass loss of 4.46%. Residual mass was 24.11%. We obtain similar data with tests at a rate of 10 °C/min. The experiments suggest that temperatures below 60 °C are adequate for grain drying, and that the temperature range between 216 °C and 254 °C is interesting for roasting. The results offer important information about the specific thermic properties of the Castilla variety coffee beans, which until now has not been referenced in the literature consulted.
Keywords: Thermochemical properties of coffee, thermogravimetric analysis, differential scanning calorimetry, infrared analysis.

Resumen
En este artículo se presentan los principales resultados obtenidos de la caracterización térmica de granos de café variedad Castilla, producidos en la región sur oriental del departamento de Santander, Colombia. Se realizaron pruebas de termogravimetría (TGA) y calorimetría diferencial de barrido (DSC). Diferentes etapas de degradación térmica del grano fueron determinadas, junto con sus transiciones térmicas y la variación de la capacidad calorífica durante el proceso. La caracterización térmica permitió también el análisis de la composición de los gases obtenidos, mediante espectroscopia infrarroja (IR) de gases, misma que fue confrontada con la base de datos Bruker de espectros IR. Se pudo observar tres sectores de pérdida de masa para el grano de café, a una velocidad de calentamiento de 5 °C/min y temperaturas de hasta 1000 °C. Las zonas están entre los 58,5 °C y 324 °C, con una pérdida de masa del 50,26%, 324 °C y 432 °C, con una pérdida de 14,67% y, 432 °C - 513 °C, con una pérdida del 4,46%. La masa residual fue del 24,11%. Resultados análogos fueron derivados con pruebas a 10 °C/min. Los experimentos sugieren que temperaturas inferiores a 60 °C son adecuadas para el secado del grano, y que el rango de temperaturas entre 216 °C and 254 °C es de interés para su tueste. Los resultados ofrecen una información importante sobre las propiedades térmicas específicas de los granos de café variedad castilla, hasta ahora no referidas en la literatura consultada.
Palabras clave: Propiedades termoquímicas del café, termogravimetría, calorimetría diferencial de barrido, análisis infrarrojo.

1 Introduction

Colombian coffee is globally known for the quality of its beans, from which its scent and flavor derive. Hundreds of farmers depend on coffee for a living, as it represents one of the highest contribution to the agricultural gross domestic product (GDP) of the country (DANE, 2017). Coffee production spans several stages from recollection to roasting, and it includes pulping, washing, and drying.

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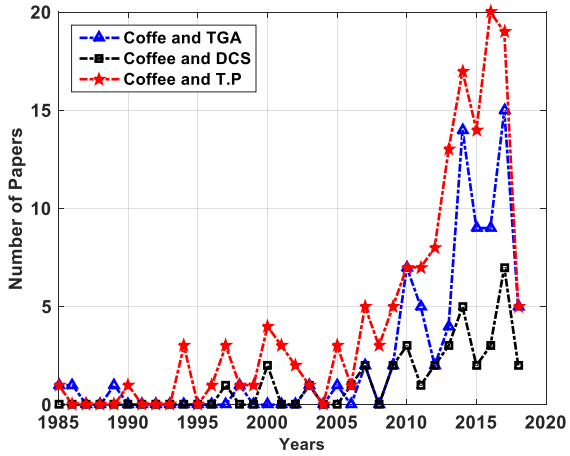

Fig. 1. Registered articles in the SCOPUS database. Blue line: "Coffee and Thermogravimetric". Black line: "Coffee and Differential Scanning Calorimetry". Red line: "Coffee and Thermal Properties".

The latter is a rather delicate process that requires the reduction of its humidity from around 55%, all the way to between 10% and 12% (Boot, 2006). Knowledge about the thermodynamics of the bean is paramount, not only for the drying process, but also for the roasting process. Research about such topic has increased in recent years, as indicated by the number of documents indexed by Scopus for the queries "coffee and thermogravimetric", "coffee and differential calorimetry scanning", and, "coffee and thermal properties", (Figure 1).

As indicated by the figure, before 2005, annual publications regarding these subjects were below five. However, after 2013, related manuscripts were above ten per year. This increment can also be related to experimental techniques that are more accessible. Nonetheless, most works on determining the thermal properties of coffee residue, for the use of their related biomass, or even for the generation of new materials. Thus, the thermochemical analysis for characterizing the bean itself is scarce.

Some initial interesting works, were focused on detecting patterns for establishing differences among different crops, and for determining the relationship between a given crop and the country of origin. Among them, we highlight the works of Dyszel (1985, 1986), which were targeted at characterizing and comparing coffee beans through thermogravimetric analysis (TGA), and atmospheric-pressure chemical ionization mass spectrometry (APCIMS). In the former, the author characterized the Arabica and Robusta varieties from different countries. Temperature sweeps were carried out at 25 °C/min, between 50 °C and 200 °C, and a mass scanning was carried out at 15 and 250 units of atomic mass, once per minute, with the positive ionization mode. Data showed, though not categorically, that the curves of ionic abundance vary less for samples within the same variety and the same country, that for samples from different countries. In the second work, the author presented a similar work but using APCIMS with a negative ionization mode. Through it, it was possible to analyze the abundance of collected mass, from the Robusta and Arabica varieties from different countries. In later works, Tomassetti *et al*. (1989) reported thermogravimetric analyses performed on samples of roasted coffee beans. For such product, mass loss was present at three moments: 24 °C, with a loss of 4.8%; 170 °C, with a loss of 53.6%; and 347 °C, with a loss of 35.5%. Temperature was varied between 20 °C and 700 °C, at a heating rate of 10 °C/min. The authors wrapped up their work by concluding that TGA is appropriate for determining the humidity content of the samples under test. Another work was that of Singh *et al*. (1997), where the authors analyzed the thermophysical properties of ground coffee extracts, both roasted and unroasted, from México and Colombia. The properties that were studied included the specific heat of the samples, which was determined by Differential Scanning Calorimetry (DSC). For temperature variations between 45 °C and 150 °C, specific heat exhibited a marginal increase. In the range between 45 °C and 110 °C, Colombian unroasted ground samples increased their specific heat between 1.77 and 2.13 J/g K, while Mexican samples did so between 1.19 and 1.71 J/g K.

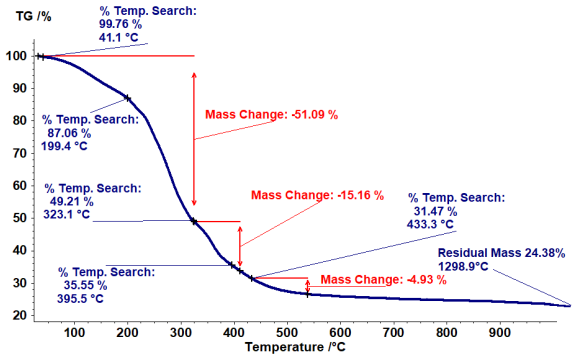

Fig. 2. Thermogram of Castilla variety coffee bean with heating of 5 °C/min. "Temp Search" do reference to mass loss at a specific temperature. In red percentage of mass loss at a specific temperature.

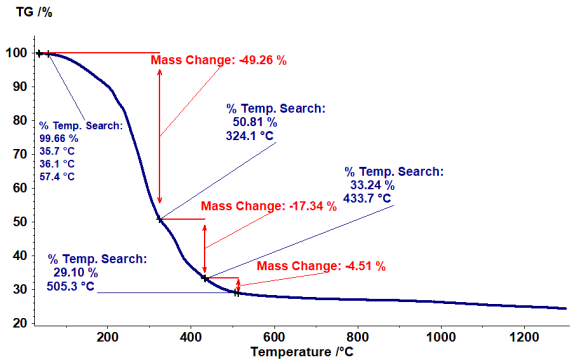

Fig. 3. Thermogram of Castilla variety coffee bean with heating of 10 °C/min. "Temp Search" do reference to mass loss at a specific temperature. In red, delta of mass loss in percentage.

In a comparable way, Ture (2005), studied the thermophysical properties of coffee through TGA and DSC, using a sample of 10 mg of grounded green coffee. Three transition regions were established: 26.4 °C - 194 °C, with a mass loss of 6.96%; 194 °C - 393 °C, with a mass loss of 61%, and 393 °C - 786 °C, with a mass loss of 28%. More recently, Velasco *et al*. (2013) used TGA and Fourier-transform infrared spectroscopy (FTIR) for analyzing ground organic coffee, both green and roasted. They showed an accelerated decomposition between 200 °C and 289 °C. The authors concluded that 218 °C is a critical temperature for roasting. Previously, Rivera *et al*. (2011) studied the effect of roasting on Arabica coffee beans, through TGA, DSC and X-ray diffraction (XRD). They reached a similar conclusion.

Literature about this topic is broader on studies unrelated to coffee beans but related to the thermodynamic analysis of their residue. Such works generally strive to determine the feasibility of using the residue as biomass. Some examples include the work of Chen et al (2012), where the authors focused on using techniques such as TGA and FTIR for estimating the biomass potential of coffee residue as an eventual fuel. Another work is that of Orsini *et al*. (2011), where the authors carried out a thermal study using TGA and DSC on the inner and outer dried husk of coffee, characterizing the amount of water on each sample, as well as their organic and inorganic residuals. The study established an enthalpy of 249 Jg^{-1} for the bean husk. In another work, Ballesteros *et al*. (2014) analyzed the chemical and structural properties of roasted coffee residue, as well as of the bean husk, finding appropriate samples for diverse biotechnological processes.

Earlier, Manals-Cutiño *et al*. (2011) used TGA and DSC on four biomasses, including coffee husk, striving to find the characteristic temperatures of their decomposition procedure. Tsai and Liu (2013) analyzed the effect of temperature on the thermochemical properties and density of coffee residue samples that had been subject to roasting. Li *et al*. (2014) examined the potential of bioenergetic production of coffee residue in pyrolysis processes of 10 and 60 °C/min, through TGA and DTGA; They found points of critical mass loss at 75 °C (due to humidity reduction), 300 °C (due to hemicellulose decomposition), 335 °C (due to cellulose decomposition), and 390 °C (due to lignin decomposition). In the work of Kobelnilk *et al*. (2014), the authors also used TGA and DSC. But, this time they use it for analyzing the lipid profile and for obtaining a thermal model of coffee oil, obtained from roasted and green beans. The work showed that the coffee oil has an exothermal process that is directly related to its oxidation procedure. A similar work is that of Atabani *et al*. (2017). On the other hand, the work of Fermoso and Masek (2018), determined the kinetics of thermal decomposition of ground coffee residue. The experiment included heating rates between 5 and 100 °C/min, up to a maximum temperature of 500 °C. Data showed a linear increase of the maximum decomposition rate with respect to the heating rate, with higher values for more reactive components, and ordered as: hemicellulose, cellulose, and lignin.

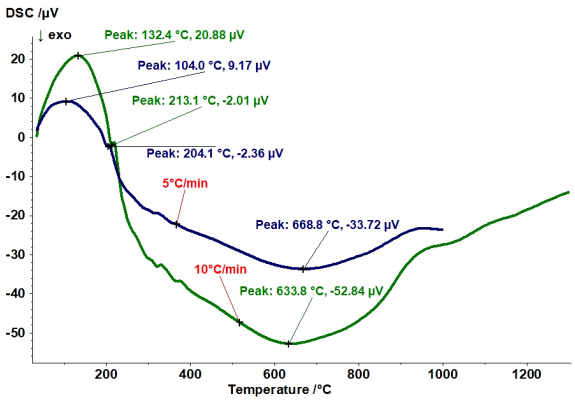

Fig. 4. DSC thermogram of Castilla variety coffee bean to 5 °C/min and 10 °C/min. In blue, DSC values for variation of 5 °C/min. In green, DSC values for variation of 10 °C/min.

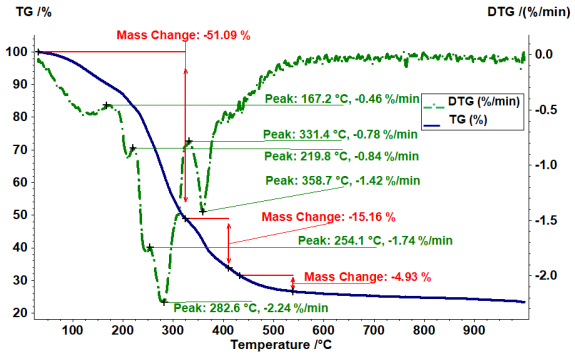

Fig. 5. TGA and DTGA thermograms of Castilla variety coffee bean to 5 °C/min. In red, mass loss percentage in the TGA. In green, peak percentage of mass loss by minutes (DSC), at a specific temperature.

Parallel works for determining tampering of roasted coffee, and the thermal properties of coffee residue mixed with coal can be respectively found in Mhilu and Mashingo (2011), and more recently, in Brondi *et al*. (2017).

Besides this vast literature, it is also known that coffee beans contain minerals such as potassium, calcium, magnesium, and phosphor, as well as a series of organic acids of high interest both, medically and therapeutically, known as caffeoylquinic acids and chlorogenics. Coffee beans also contain alkaloids, the most important one being caffeine, which is usually found in concentrations ranging from 1% to 2.5%, depending on the kind of coffee (Farah, 2012, Abdalla 2015). A composition that can be considered as standard for a coffee bean without contaminants, according to Rojo-Jimenez and Pérez-Urria (2014), is: 34% cellulose, 30% sugar, 11% proteins, between 6% and 13% water (depending on the type of drying process), and between 2% and 15% fat mass. The same authors report that, additionally, minerals such as potassium, calcium, magnesium and phosphor can be found. Even so, throughout the roasting process the bean experiences chemical transformations, in which chlorogenic acids transform in lactones and phenylindanos. Sterols within coffee grains lead to monoterpenes, naphthalene and quinolines. Aliphatic acids such as lactic, pyruvic, glycolic, oxalic, tartaric, and citric, are also a product of roasting.

In this manner, the research we present in this work is focused on increasing the knowledge about the thermal properties of the coffee grain of Castilla variety, which is part of the Arabic species, from samples of beans grown in the Southeastern part of Santander, Colombia. We strive to characterize the various stages of its thermal degradation, which have not been found, in the broad consulted literature, through the use of TGA, DTGA and DSC analysis. These techniques have been widely used for characterization of different and diverse materials besides biomass, as for example, catalysts (Cruz-Ortiz *et al*., 2017), chitosan, algae and zeolite (Pérez-Escobedo *et al*., 2016), excipients (Cassel, Riga and Biddlecom, 2004), or pyrochlore (Torres and Peláiz, 2003). The methodology we follow and the results we achieve are shown below. Then we wrap up the article by presenting the most relevant conclusions.

2 Materials and methods

This work used a sample coffee bean from the Castilla variety, grown in the Southeastern part of Santander, Colombia. After carefully removing the husk, tests with TGA/DSC were carried out at heating rates of 5 °C/min and 10 °C/min, for a sample of 41.7168mg and 49.8005mg of coffee, respectively. Another test was carried at 5 °C/min, for 55.4922mg, but including isothermal periods at 32 °C, 220 °C, 280 °C, and 660 °C. The experiments were conducted under a Nitrogen atmosphere. Moreover, the experiments at 5 °C/min used an IR gas coupling system. The following equipment was used: DSC/TGA-IR, STA 449 F5 JUPITER from NETZSCH, and an IR TENSOR II, from BRUKER with a TGA coupling.

The experiments corresponded to analytic tests, which require the use of sophisticated laboratory equipment, as it has been referred in the above paragraph.

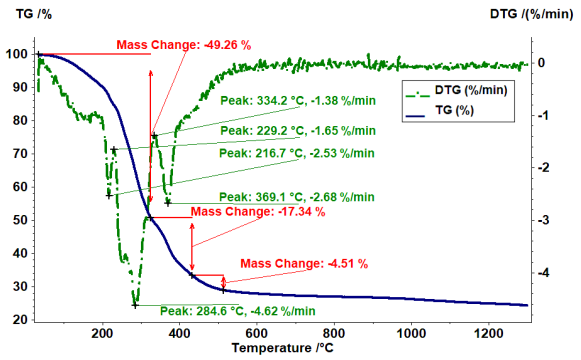

Fig. 6. TGA and DTGA thermograms of Castilla variety coffee bean to 10 °C/min. In red, mass loss percentage in the TGA. In green, peak percentage of mass loss by minutes, at a specific temperature.

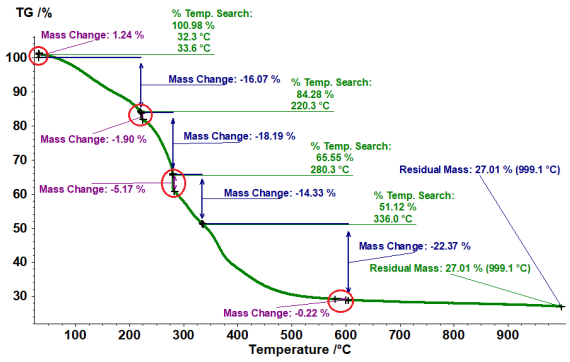

Fig. 7. TGA thermogram of Castilla variety coffee bean to 5 °C/min with isothermal sectors at 32 °C, 220 °C, 280 °C and 600 °C. In blue and purple, delta of mass change. In green, percentage of mass loss at a specific temperature.

The experiments were performed based in the ASTM E1131-08 and ASTM E1269-11 standards. According to this, two test were performed, and both results are reported in the paper

3 Results and discussion

We characterized several stages of thermal degradation for the coffee bean, as well as its transitions and variations in calorific capacity throughout the controlled heating process. Also, we performed a real-time analysis of the composition of the gasses generated throughout the thermal characterization, using Infrared (IR) spectroscopy, and comparing the data to the Bruker IR spectrum database.

3.1 TGA and DSC results

Figure 2 shows a thermogram at 5 °C/min, up to a temperature of 1000 °C, in which three sectors of representative mass loss can be observed. In the first one, located between 58.5 °C and 324 °C, about 50.26% of the coffee bean mass is lost. This represents the highest loss during testing. The second sector ranges from 324 °C and 432 °C, with a loss of 14.67%. The final one is located between 432 °C and 513 °C, with a loss of 4.46%. At the end of the test, the residual mass represented 24.11% and represents the inorganic minerals present within coffee, such as Potassium, Calcium, Magnesium, and Phosphorus. These were not further characterized. Figure 3 shows a thermogram for a heating rate of 10 °C/min. As in the previous case, three main regions of mass loss can be detected. Even though this test was allowed to run until a higher temperature (1200 °C), the residual mass of the sample was similar to that of the previous case.

Figure 4 summarizes the DSC experiments for 5 °C/min and 10 °C/min. The data also shows three processes. The first one, between 32 °C and 180 °C, corresponds to an endothermal process, due to water loss within the coffee bean. There is another transition between 204 °C and 220 °C, also endothermal in nature, and this one corresponding to polysaccharides, sugars, and the fusion of amino acids. From 280 °C onwards, there is an exothermal transition, which corresponds to the degradation and carbonization of the sample.

Figures 5 and 6 show the derivatives of TGA tests at 5 °C/min and 10 °C/min. Here, the transitions shown in Figures 2 and 3 have been overlapped. In the first case (i.e. 5 °C/min), an additional transition can be observed at 254 °C, corresponding to the degradation and sublimation of aromatic organic compounds. It should be taken into account that caffeine sublimates between 219 °C and 254 °C. At this point, is important to note that, the unique difference between Figures 5 and 6, is that the heat rate (5 °C/min and 10 °C/min, respectively), and the samples evaluated are of the same nature. For this reason, the range of sublimation for caffeine is the same. Also, within this range, the thermal transformation of cafestol and kahweol is manifests. Both compounds are known for their importance in coffee scent. The transitions observed between 331 °C and 358 °C are due to hemicellulose and cellulose degradation. Table 1 synthesizes previous results.

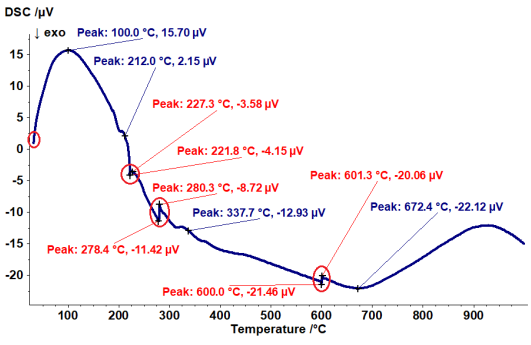

Fig. 8. DSC analysis of Castilla variety coffee bean to 5 °C/min with isothermal sectors at 32 °C, 220 °C, 280 °C and 600 °C. In red and blue, DSC values at a specific temperature: Blue for not isothermal regions, and red for isothermal regions.

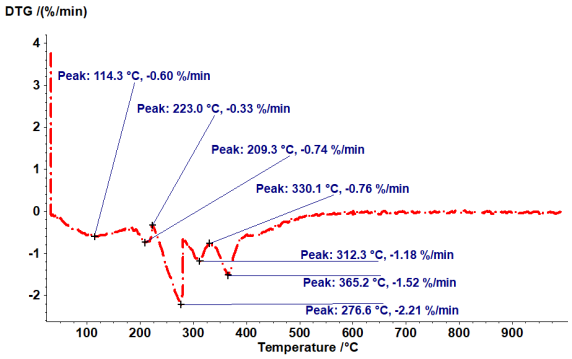

Fig. 9. DTGA thermogram analysis of Castilla variety coffee bean to 5 °C/min with isothermal sectors at 32 °C, 220 °C, 280 °C and 600 °C.

3.2 Results with isothermal sectors

TGA tests with isothermal sectors allowed us to analyze with more detail the mass losses. To do so, we established regions within the degradation process, as shown in Figure 7. The first zone relates to an isothermal of five minutes, at 32 °C. Here, very small mass losses are registered. Afterwards, a region between 32 °C and 220 °C is observed, with a mass loss of 15%. Based on previous tests, this region mainly contains losses due to dehydration, plus some due to sugar and amino acid degradation. Afterwards, an isothermal region at 220 °C appears, where a loss of 1.90% is manifested from the organic compound degradation that may include caffeine and cafestol, among others. From 220 °C to 280 °C there is a region with a mass loss of 17.19%, linked to the start of sample carbonization and to the transformation of cellulose and hemicellulose. The isothermal region at 280 °C exhibits a loss of 5.17%, the biggest one for the isothermal regions. We believe this is due to the fact that, at this temperature, the aromatic compounds with the biggest molecular mass are degraded. Regions between 280 °C, 336 °C, and 600 °C exhibit mass losses of 9.33% and 22.37%, respectively. They are due to big-sized cellulose and polysaccharides degradation. The isothermal region at 600 °C reflects a very small loss, around 0.22%, since most organic compounds have already been degraded or have been removed from the sample. From this point onward, its composition is mainly from inorganic minerals, as was in the residual 27%.

Figure 8 shows the DSC experiments with isothermal sectors. In it, the three transitions mentioned in Figure 4, can also be observed. At 32 °C, there is no transition since there are no changes in the sample. The sector of 220 °C exhibits an endothermal transition, corresponding to sugar and polysaccharides fusion, as was already mentioned. At 280 °C, there is an endothermal transition likely due to degradation of cellulosic derivatives. Afterwards, there is an exothermal reaction due to sample carbonization. At 600 °C, there is a quiet little endothermal transition, generated by the cellulose residue still present within the sample. Note that the peaks details on figures correspond to the analysis point. For example, signal value at 925 °C in Figure 8, correspond to a transition phase for the remaining minerals of coffee residue, for which a representative loss of mass is not generated in this region. In this stage, there is no organic component left in the sample.

Figure 9 shows the derivative of the TGA test at 5 °C/min with isothermal sectors. These data are quite like Figures 5 and 6, which corresponds to the same test but under dynamic conditions. Additional transitions are given in the previously explained isothermal sectors.

3.3 Heat capacity

Using experimental data from the DSC, we calculated the heat capacity (Cp) of coffee beans for a temperature range from 30 °C to 1100 °C, via the standard Sapphire DSC curve (National Bureau of Standards). (See Figures 10 and 11) The heat capacity behavior shows three mayor regions. For temperatures between 30 °C and 500 °C, the values of Cp are near zero, except at 200 °C, where the fusion of sugar and polysaccharides with low molecular weight, generate an alteration.

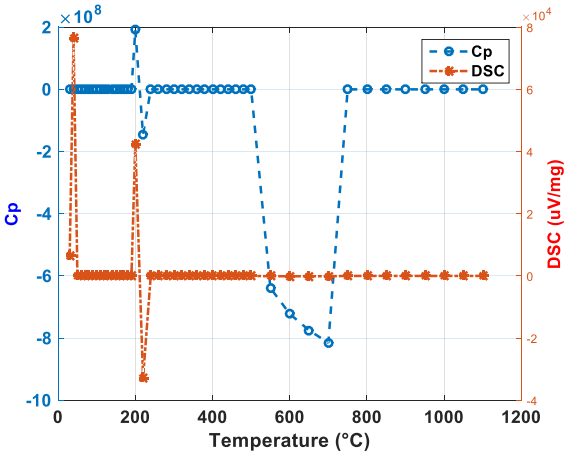

Fig. 10. DSC and Cp of the Castilla variety coffee bean with heating of 10 °C/min.

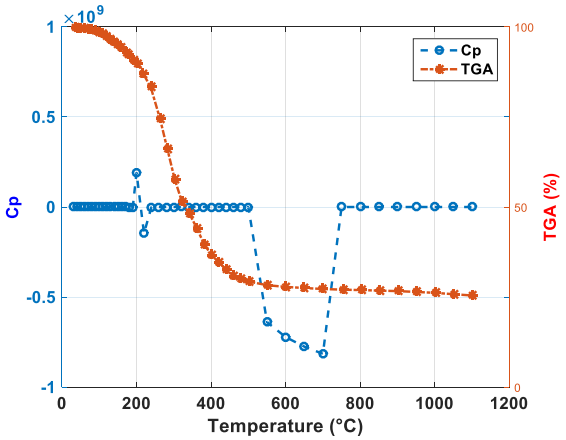

Fig. 11. TG and Cp of the Castilla variety coffee bean with heating of 10 °C/min.

Later, there is a region between 500 °C and 750 °C, where a degradation of big organic molecules, such as, cellulose and its derivatives occurs. Finally, a region between 800 °C and 1100 °C has a similar trend with respect to the first region. At these temperatures, the entire sample is now of inorganic nature, and its composition is made mainly of mineral oxides

3.4 IR Results

The IR coupling during the thermogravimetric tests, allows for capturing the gasses generated at each temperature change and analyzing them through an IR detector. Such gases correspond to mixtures of products from the boiling, sublimation, and degradation of compounds within the bean. Figure 12 shows a 3D plot that summarizes the IR spectra from the test. The X-axis represents the wavelength, the Y-axis corresponds to the absorbance of each signal, and the Z-axis is the temperature of the data sample.

Based on Beer's Law, the absorbance of a signal is proportional to the concentration of components within a mixture. Because of this, it is possible to detect the appearing and disappearing of a compound within a gas mixture, as well as the temperature at which the highest concentration manifests. Nonetheless, since the experiment relates to complex mixtures of gas products, a manual analysis is complex due to a lack of unique signals. To overcome this issue, we used the OPUS Software from Bruker, as well as their IR gas spectra database, so that we could identify signals and compounds within the bean.

Even though IR signals can be observed from the start of the thermogravimetric test, during the initial temperatures, the spectra exhibits low absorbance. This is due to a low gas concentration within the mixture. The spectra throughout the test are quite similar, because the main components overlap the signals of compounds with lower concentrations. We selected the spectra at 124 °C, 165 °C and 300 °C to execute our analysis, since these values exhibited variations in their signals (Figures 13, 14, and 15). Based on previous analyses, coffee bean dehydration starts at around 32 °C, and goes on until approximately 180 °C. This process can be seen through detailed signals of the studied IR spectra. At 124 °C, the gas mixture is mainly composed of water (Figure 13). In the spectrum at 165 °C, however, the absorbance of the signals related to water is diminish, but they are still present (Figure 14) since dehydration is still underway.

Additionally, we observe signals characteristic of compounds similar to naphthalene sulfonic acid. This means that the mixture contains compounds such as aromatic groups, carboxylic acids, nitrogen, and sulfurs. Such functional groups are a product of the degradation and sublimation of compounds such as alkaloids and small aromatic molecules. Within them, can be found cafestol and kahweol, as well as some caffeine. The presence of these functional groups in the 165 °C spectrum complements the information obtained from the thermogram, showing that some compounds within the bean experience transformations before the 200 °C mark.

Going up to 300 °C, on the contrary, shows no presence of water in the gas mixture (Figure 15). But, molecules similar to octanoic acid can be observed. Such acid exhibits a large aliphatic fraction and a carboxylic acid group.

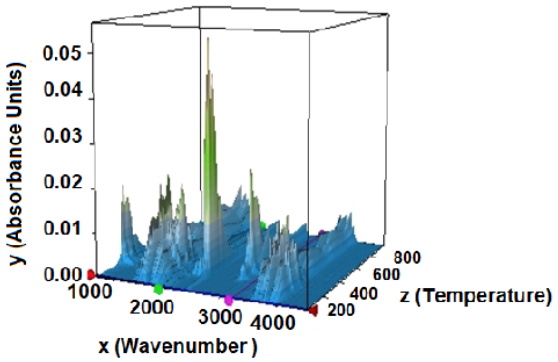

Fig. 12. 3D spectrum collected during a test to 5 °C/min.

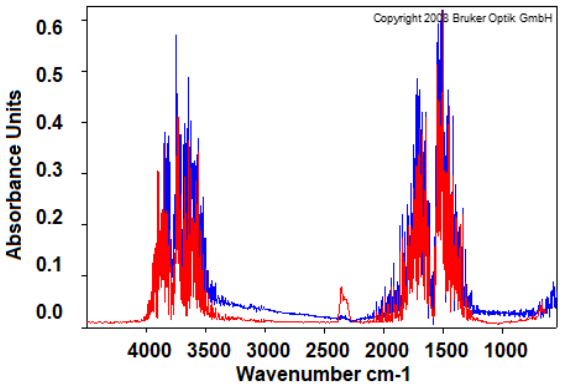

Fig. 13. IR spectrum to 124 °C collected during a test to 5 °C/min (red) and Water IR spectrum from database Bruker (blue).

At this temperature, cellulosic chains exhibit thermal transformations, as was observed in the thermograms. The identified fractions reinforce the possibility of degradation processes, since we are dealing with signals of the linear aliphatic kind, and not of those representing alkane cycles, which are the molecules that make up cellulose and its derivatives. Presence of carboxylic acids at this temperature may be a product of aldehydes oxidation, present in the sugars that make up the cellulosic compounds. Even though testing was carried out under a nitrogen atmosphere, the mixture of coffee bean compounds provided the oxygen required for oxidation of cellulose chains. The peak shown in Figure 15 at 2300 cm^{-1}, corresponds to CO_2. The database eliminates the spectrum treatment for this signal, but, since no modifications were performed in our results analysis, the CO_2 signal is still present in this figure.

4 Discussion of results

Previous results reveal accelerated changes in grain composition, after 200 °C. From DSC we can observe that this changes occur between the ranges of 219.8 °C - 282.6 °C, and 216.7 - 284.6 °C, for variations of 5 °C/min and 10 °C/min, respectively. Analyzing, as it referred, the ranges for degradation of organic aromatic components and caffeine sublimation, a special zone of interest is established for grain roasting: 219 °C - 254 °C. Although we cannot determinate a unique temperatures range for this process, which depends of diverse factors, it is possible state that this results are comparable with the studies of Velasco *et al*. (2013), performed on organic coffee of the Arabic specie, original of the Cauca department of Colombia.

Table 1. Synthesis of results for TGA, DSC and DTGA without isothermal regions.

Type of Analysis	Range of interest	Comments
TGA 5 °C/min	58.5 °C - 324 °C	50,26% of mass loss
TGA 10 °C/min	324 °C - 432 °C	14.67% of mass loss
	432 °C - 513 °C	4.46% of mass loss
	513 °C - 1000 °C	24.11% of residual mass, at the end of the process.
DSC 5 °C/min	32 °C - 180 °C	Endothermal process
DSC 10 °C/min		- water loss
	204 °C - 220 °C	Endothermal process - polysaccharides, sugars, and amino acids fusion.
	280 °C onwards	Exothermal transition, degradation and carbonization of the sample
DTGA 5 °C/min	219 °C - 254 °C	Caffeine sublimes.
DTGA 10 °C/min	331 °C - 358 °C	Hemicellulose and cellulose degradation

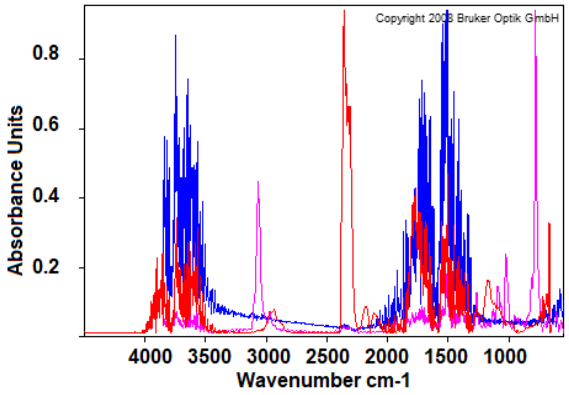

Fig. 14. IR spectrum to 165 °C collected during a test to 5 °C/min (red). Water IR spectrum (blue) and IR spectrum of naphthalenesulfonic acid (purple) from database Bruker.

It explains the importance that it has for roasting at a temperature equal or superior to 218 °C. Mendes *et al*. (2001), for dry coffee of the Robusta specie, fix at a range between 225 °C - 230 °C, as optimum for roasting, and Baggenstoss *et al*. (2008) for Arabic dry coffee, native from Sumatra, also determined, that for roasting, the temperatures above 200 °C are necessary.

With respect to the IR spectrum, the following aspect has been analyzed. The spectrum at 124 °C, established that the first substance that is eliminated of grain composition in the drying process is water, and that it can be tracked through IR spectroscopy, for achieve elimination of percentage necessary for a good composition of dry grain.

Similarly, the spectrum at 165 °C, determine that at this temperatures, characteristics signals correspond to aromatics groups and to some heteroatoms, present in the grain composition. Although at this temperature starts the elimination of some nutritional components, this won't affect the nutritional properties. This technique can become in the future, as an alternative for control of grain composition in the roasting process. From elsewhere, spectrum at 300 °C, details the presence of a derived of a cellulosic thermic transformation. Roasting temperature must be down of this value, because at this heating point, grain don't present high content of aromatic molecules and heteroatoms, which are responsible of its food properties.

Data infers too, that a drying that does not affect grain composition must be performed at temperatures below 60 °C. Indeed, as it has been reported, the IR spectrum at 124 °C shows that until this temperature, the greatest loss of mass is caused by dehydration, and, at the same time, the thermograms at 5 °C/min and at 10 °C/min, reveal that between 50° and 60 °C, the loss of mass does not exceed 0.5%.

Conclusions

This work compiles the main results from a thermochemical analysis of a coffee bean sample of the Castilla variety, grown in the Southeastern region of Santander, Colombia. The results include thermograms obtained via thermogravimetric analysis, differential scanning calorimetry, and gas analysis via real-time infrared spectroscopy. The composition was within the typical ranges for a coffee species. Even so, we observed a particular decomposition range and heat capacity. Three sectors for mass loss were defined, using a heating rate of 5 °C/min, up to 1000 °C. The first zone varied between 58.5 °C and 324 °C, with a mass loss of about 50.3%. The second one did so between 324 °C and 432 °C, with a mass loss of 14.7%. The final one varied between 432 °C and 513 °C, with a mass loss of 4.5%. The residual mass was about 24.1%. We found comparable results with a heating rate of 10 °C/min. For the analysis of the generated gasses, we used the Bruker database for defining the functional groups present at each temperature zone detected, during the controlled heating of the coffee bean sample.

The results provide knowledge about the thermic properties of coffee beans of the Castilla variety that, until now, have not been referred in the consulted literature. From them, we can infer that temperatures below 60 °C, are appropriated for grain drying. Indeed, the IR spectrum indicates that until 124 °C, the biggest mass loss occurs by dehydration, and the TGA thermograms show that until 60 °C, the mass change is less than 0.5%.

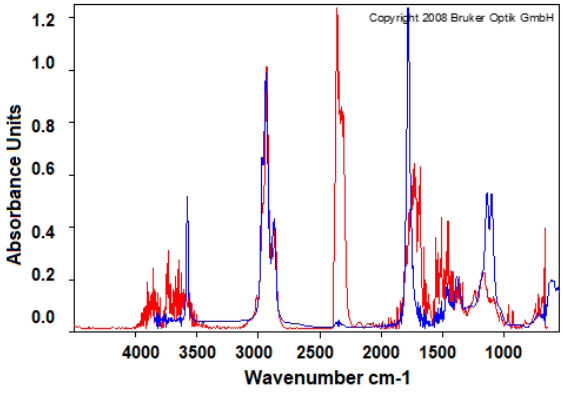

Fig. 15. IR spectrum to 300 °C collected during a test to 5 °C/min (red). Octanoic Acid spectrum from Bruker database (blue).

Likewise, the experiments determine that the range between 216 °C and 254 °C is of interest for roasting, due to the accelerated change in chemical composition that occurs at this interval. To determine an optimal range for this process is out of the scope of this paper, and needs complementary studies.

Nomenclature

Cp heat capacity

Greek symbols

μV micro volts

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