

ATR-FTIR FOR DISCRIMINATION OF ESPRESSO AND AMERICANO COFFEE PODS

Andrés Felipe Bahamon Monje¹, Lina Ximena Parrado², Nelson Gutiérrez-Guzmán³

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ABSTRACT: Roasted and ground coffee for encapsulation in single-serve capsules compatible with keurig® and coffee powder obtained from Nespresso® commercial capsules were analyzed for pH value, titratable acidity, moisture content, water activity and color (lightness); a data matrix that contains the physicochemical properties and the absorbance measurements using a baseline of 1600 to 1800 cm⁻¹ by FTIR-ATR technique, was evaluated through the combined methods of principal component analysis (PCA) and cluster analysis in order to discriminate between the types of capsules. In the PCA biplot two distinct groups can be identified and in the cluster analysis two groups are that correspond to the two types of capsules. The results reveal that FTIR-ATR based methods seem to be a promising alternative for the discrimination of coffee samples for the pods industry or for the type of consumption.

Index terms: Coffee brewing Single-serve brewer, Coffee pods, Extraction brew physicochemical properties, FTIR spectra.

ATR-FTIR PARA DISCRIMINAÇÃO DE CÁPSULAS DE CAFÉ ESPRESSO E AMERICANO

RESUMO: Café torrado e moído encapsulado para consumo individual compatível com keurig® e pó de café obtido cápsulas comerciais Nespresso®, foram analisadas para valores de pH, acidez titulável, teor de água, atividade de água e cor (luminosidade); uma matriz de dados com as propriedades físico-químicas e medições de absorvância usando uma linha de base de 1600 a 1800 cm⁻¹ pela técnica ATR-FTIR, foi avaliada por meio de métodos combinados de análise de componentes principais (PCA) e análise de clusters a fim de discriminar entre os tipos de cápsulas. No gráfico do PCA podem ser identificados dois grupos distintos, e na análise de clusters, dois grupos que correspondem a dois tipos de cápsulas. Os resultados mostraram que métodos baseados em ATR-FTIR podem ser uma alternativa promissora para a discriminação de amostras de café na indústria de cápsulas ou para o tipo de consumo.

Termos para indexação: Máquina de café em dose única, Cápsulas de café, Propriedades físico-químicas no extrato de café, Espectro de FTIR.

1 INTRODUCTION

Espresso and *americano* coffee are a widely consumed beverages worldwide; while the espresso coffee has been defined as a beverage prepared by submitting ground roasted coffee beans to hot water at a certain pressure for a short time, the *americano* coffee is defined how a diluted espresso, extracted with higher water volume; after the extraction, an *americano* coffee has a similar volume to a filtered coffee.

Traditionally, the espresso and *americano* coffees were extracted in a same big and expensive coffee machines, recently new devices was development to make this beverages based on pods systems, they have gained market share because they are user-friendly and they also make it easy to prepare good-quality coffees through the reduction of uncontrolled preparation variables (PARENTI et al., 2013); although encapsulated coffee is increasingly popular, little research information exists about this product. WANG et al. (2016) evaluated the effects of the capsule parameters on extraction yield and physicochemical properties of

coffee brews prepared using a commercial single serve brewer Keurig®; DESBROW et al. (2017) quantified the caffeine content of the Nespresso® coffee pod and BELCHIOR et al. (2017) evaluated the potential of ATR-FTIR and chemometrics to discriminate espresso coffees with different sensory characteristics described by a panel of coffee tasters.

Some food analysis techniques are complicated instruments and require time and more costs; FTIR- ATR spectroscopy is a simple technique that requires no sample pre-treatment, is fast and provides an overall infrared fingerprint of the specimen; FTIR- ATR have been recently employed for coffee analysis (REIS et al., 2013a); FTIR analysis was used to detect multiple adulterants in roasted and ground coffee CRAIG et al. (2014); to discriminate between defective and non-defective coffee beans prior to roasting (CRAIG et al., 2012) and to evaluate the chlorogenic acid isomer profile and antioxidant capacity of coffee beans (LIANG et al. 2016b).

Since Chlorogenic acids (CGAs) are a major acid group present in Coffee (FARAH &

^{1,2,3}Centro Surcolombiano de Investigación en Café CESURCAFE/Universidad Surcolombiana - Departamento de Ingeniería Agrícola Cx.P. 410001 - Neiva - Colombia - u2010297360@usco.edu.co, u20142131808@usco.edu.co, ngutierrezg@usco.edu.co

DONANGELO, 2006), and caffeine content is relevant in coffee analysis, authors have proposed for the identification and quantification of caffeine and CGAs using infrared spectra finding that peaks are usually located within the wavelength range from 1600 cm⁻¹ to 1800 cm⁻¹ (Table 1).

The objective in this work was to investigate the feasibility of using some physicochemical properties and the bands of the FTIR-ATR spectrum where the CGA's and Caffeine peaks are highlighted (1600-1800 cm⁻¹), in conjunction with PCA and HCA to discriminate between *americano* and espresso coffee pods.

2 MATERIALS AND METHODS

2.1 Samples

Six commercial Nespresso® coffee pods (7g coffee net weight) (EC) and twelve *americano* coffee pods (12g coffee net weight) (AC) of which three commercial Keurig® k-cup coffee pods and nine roasted and grinding coffee samples packed

in the single-serve coffee capsule EZ-CUP^{2.0} compatible with keurig K-cup brewers were used in the analysis; the single origin coffee and encapsulated process is presented in the table 2. The manually encapsulated and sealed process occurred in a laboratory prototype of coffee pod packaging with a single chamber (Figure 1).

2.2 Physicochemical analysis in coffee powder

Moisture content

Before being packed, the roasted and ground coffee for the Keurig® pods and the coffee powder inside of the Nespresso® pods (Factory packed) were evaluated for moisture content with MB45 OHAUS infrared moisture analyzer.

Water activity measurement

3±1 g of coffee powder were placed in the sample chamber of a vapor sorption analyzer, at 25°C. (VSA Aqualab Decagon Device, Inc. Pullman, WA).

TABLE 1 - Reported bibliography of infrared spectral analysis in coffee.

Molecules	Wavelength (W) - peaks (P)	Author(s)/year
Caffeine	W(1660-1600) cm ⁻¹	(KEMSLEY et al, 1995)
	P(1700, 1658, 1600) cm ⁻¹	(DAGHBOUCHE et al, 1997)
	P(1659, 1554, 1710) cm ⁻¹	(BOUHSAIN et al, 1999)
	W(4000-800) cm ⁻¹ , P(1700,1659) cm ⁻¹	(GARRIGUES et al, 2000)
	W(1800-1300) cm ⁻¹	(NAJAFI et al, 2003)
	P(2924, 1591, 1659, 1585) cm ⁻¹	(ESTEBAN-DÍEZ et al, 2004)
	P(1659, 1705) cm ⁻¹	(GALLIGNANI et al, 2008)
	P(1650, 1600) cm ⁻¹	(WANG & LIM, 2012)
	P(1650, 1745) cm ⁻¹	(RIVERA et al, 2013)
	P(2923, 2854, 2925, 2848, 1659, 1655) cm ⁻¹	(REIS et al, 2013b)
	P(1659) cm ⁻¹	(BARBIN et al, 2014)
	W(1700-1600) cm ⁻¹ , P(1642) cm ⁻¹	(ABDALLA et al, 2015)
	P(1625) cm ⁻¹	(CRAIG et al, 2018)
P(1655, 1591, 1072) cm ⁻¹	(BELCHIOR et al, 2017)	
CGAs	W(1450-1000) cm ⁻¹ , P(1300, 1150) cm ⁻¹	(KEMSLEY et al, 1995), (BRIANDET et al, 1996), (REIS et al, 2013b)
	P(1375, 1074)	(BUFFO & CARDELLI-FREIRE, 2004)
	P(1160, 1250, 1380) cm ⁻¹	(RIVERA et al, 2013)
	P(1868, 1741) cm ⁻¹	(BARBIN et al, 2014)
	P(1605, 1627, 1680, 1742) cm ⁻¹	(LIANG et al, 2016a)
	P(809, 1032, 1120, 1165) cm ⁻¹	(LIANG et al, 2016b)
W(1375-1074) cm ⁻¹ , P(1072) cm ⁻¹	(BELCHIOR et al, 2017)	

TABLE 2 - Samples, origin and packed process.

Type of capsule	Origin	Encapsulated
AC	Colombia	Manually packed
AC	Colombia	Manually packed
AC	Bolivia	Manually packed-
AC	Guatemala	Manually packed-
AC	Ethiopia	Manually packed-
AC	Colombia	Factory packed
AC	Colombia	Factory packed
AC	Colombia	Factory packed
AC	Colombia (Huila)	Manually packed-
AC	Colombia (Huila)	Manually packed-
AC	Colombia (Huila)	Manually packed-
AC	Colombia (Huila)	Factory packed
EC	Colombia	Factory packed
EC	Colombia	Factory packed
EC	Colombia	Factory packed
EC	Colombia (Medellin)	Factory packed
EC	Colombia	Factory packed
EC	Colombia	Factory packed

**FIGURE 1**- Laboratory prototype of coffee pod packaging with a single chamber.

Color. CIE L*a*b* coordinates were obtained in the coffee powder directly in the 50 mm (diameter) cell, in triplicate with Minolta CR-410 Colorimeter (Konica Minolta Sensing Inc., Osaka, Japan).

Titrateable acidity. Determined by titration, with the Ph meter (BP-3001 manufactured by Trans Instruments, Singapore), 50 ml of distilled water at 90 ± 2 ° C is filtered with 5 grams of roasted ground coffee. After that, 50 mL of the filtered extract, filtered by waterman qualitative filter paper, was titrated against 0.1 N NaOH solution to pH 6.5 Measurements were taken in duplicate.

2.3 FTIR analysis

A FTIR Spectrophotometer (Cary 630 manufactured by Agilent, EEUU) was used in the ATR-FTIR measurements that were performed in a dry atmosphere and at room temperature (20 ± 0.5 °C). A horizontal ATR sampling accessory (Diamond ATR) equipped with ZnSe cell was employed. Approximately (1 g) of the roasted and ground coffee was placed in the sampling accessory and pressed; the background material was obtained from readings of the accessory without any sample. All spectra were recorded within a range of $1800\text{--}1600$ cm^{-1} where the CGA's and Caffeine peaks are variable importance projection scores (VIP)(BELCHIOR et al., 2017), the resolution used was 4 cm^{-1} with 20 scans and submitted to background subtraction; all samples were analyzed in triplicate. The chlorogenic acid standard SIGMA ALDRICH CAS 327-97-9 purity $\geq 99.0\%$ and caffeine standard CAS 58-08-2 purity $\geq 99.0\%$ was used for determination of spectrum patterns, in the same range defined with VIP scores.

2.4 Coffee brewing

The packed coffee (AC) was brewed using a commercial single serve brewer (K50 Classic Series manufactured by Keurig®, USA), using Lungo method with 177 ml (6 oz) hot water; the Nespresso® pods (EC) were brewed with a commercial single serve (C50-US-CW-NE manufactured by Nespresso®, Switzerland) with pressure of 19 bar and 40 ml (1,35 oz) hot water.

2.5 Coffee beverage characterization

The pH value of coffee AC and EC beverages was measured with pH-meter BP-3001 (Trans Instruments, Singapore); the Refractive index with digital refractometer PR-201 α (Atago, United States) and the beverage lightness (L^*),

was obtained in the coffee beverages, using a CR-A33e Light Protection Tube with Glass of the digital CR410 (Konica Minolta, japan).

2.5 Statistical analysis

All data obtained in this study were analyzed statistically, the results are expressed as the mean \pm standard deviation; differences among average values were estimated. Comparisons between the two groups were made by Student's t test using the statistical package Statgraphics Centurion XVI. Average values were considered significantly different when $P \leq 0.05$. Cluster and PCA analysis was applied to the matrix with the absorbance values of the peaks related to representative functional groups in 1600 to 1800 cm^{-1} region and physicochemical parameters in coffee powder and beverages.

3 RESULTS AND DISCUSSION

3.1 Coffee powder and beverages characterization

Table 3 presents the results of Student's t test for the comparison of the coffee powder contained in EC and AC, with the addition of the comparison of the parameters obtained in the EC and AC beverages. The moisture content, a_w and the degree of roasting in coffee powder from EC presented statistically significant differences ($P < 0.05$) compared to AC; likewise, in the analysis of the beverages, pH, °Brix and beverage lightness (L^*) presented statistically significant differences ($P < 0.05$) in the two types of drinks.

The moisture content and a_w in AC is significantly higher than in EC, this result may be influenced by the storage conditions or because the AC package material allowed vapor exchange between the inside of the pods and the surrounding environment; the color of roasting in the coffee powder of EC and AC showed significant differences ($p < 0.05$), the magnitudes of the coordinate L obtained (23.20 ± 0.53 and 24.79 ± 1.5 respectively) correspond to the degree of dark roasting according to the classification proposed by FRANCA et al. (2009), although the color in samples are statistically different this result is appropriate the preparation of espressos and americano coffee.

The refractive index in the beverages had higher result in espresso than in *americano* coffee as shown in Table 3, similar results were obtained by GLOESS et al. (2013) where he determined that espresso coffee has a refractive index closer to 4.0%.

TABLE 3 - Physico-chemical parameters in EC and AC coffee powder and beverages

Type	Physico-chemical parameters	Type of capsule	
		EC	AC
Roasted and ground coffee	Moisture content (%)	2.71 ± 0.62 ^a	4.51 ± 1.52 ^b
	Water activity (Aw)	0.25 ± 0.11 ^a	0.38 ± 0.11 ^b
	Color (°L)	23.20 ± 0.53 ^a	24.79 ± 1.50 ^b
	Titrateable acidity (0.1 M NaOH / ml)	1.68 ± 0.40 ^a	1.85 ± 0.36 ^a
Beverage coffee	(Ph)	5.21 ± 0.13 ^a	4.92 ± 0.11 ^b
	Refractive index (°Brix)	3.92 ± 0.53 ^a	1.66 ± 0.45 ^b
	Color (°L)	32.04 ± 2.89 ^a	29.80 ± 1.15 ^b

n=3, mean ± SD. Different letters, in the same row indicate significant difference (P<0.05).

The author evaluated these parameters in different machines: a semi-automatic espresso machine, an automatic machine and a Nespresso® brand single-use machine. In the *americano* coffee, the same author determined that the refractive index was slightly higher than 1.0%; finding for the filtered coffee extract a refractive index of 1.03±0.01% and for the French press 1.43±0.01%, these results are similar to those found for the AC. The pH in beverages was similar as those obtained by FUJIOKA & SHIBAMOTO (2008) in seven types of commercial coffees varying between 4.95±0.01 and 5.99±0.01, this author comments that the pH in extracted coffee is related to the presence of chlorogenic acids, although WANG & LIM (2012) comments that high values in the pH of coffee beverages is presents in dark roasted coffee beans and it is associated with the presence of organic acids (citric and malic acids).

3.2 FTIR spectral analysis

Figure 2A shows the CGAs (blue) and Caffeine (red) spectrum patterns; the figure 2B shows the approach of the region between 1600 to 1800 cm⁻¹ in the CGAs standard, three peaks are clearly defined, the carbonyl (C=O) group at 1685 cm⁻¹, the ethylene (C=C) group at 1636 cm⁻¹ and the phenyl ring stretch at 1599 cm⁻¹ according to LIANG et al, (2016a); likewise, the figure 1C presents the Caffeine standard enlargement, two peaks are clearly defined, the carbonyl (C=O) group at 1642 cm⁻¹ and the amines (C=N) group at 1692 cm⁻¹.

Figure 3 presents the *americano* coffee pods AC (red) and espresso coffee pods EC (blue) spectrums in the region 1600 to 1800 cm⁻¹. The spectrums obtained in this research are similar with those reported by LIANG, et al. (2016a) in commercial coffee samples; the figure 3 shows the CGAs and caffeine molecules superimposed with the functional groups located in the corresponding Wavenumber, which were taken to obtain the respective absorbance values. These absorbance values were included in the multivariate matrix for PCA and HCA analysis.

3.3 Cluster Analysis

Figure 4 shows the dendrogram based on similarity of physical-chemical parameters and absorbance values of the functional groups peaks in 1600–1800 cm⁻¹ region. Two clusters are defined for the AC and EC coffee pods samples. All of the americano coffee pods samples AC are included in the Cluster 1, though it is not possible to find a separation pattern for different origins or between commercial or manual encapsulated type. The cluster 2 includes the six espresso coffee pods samples EC.

3.4 Exploratory analysis

The biplot of the PCA on the matrix composed with the absorbance values of the peaks related to representative functional groups in 1600 to 1800 cm⁻¹ region and physicochemical parameters in coffee powder and beverages is shown in figure 5.

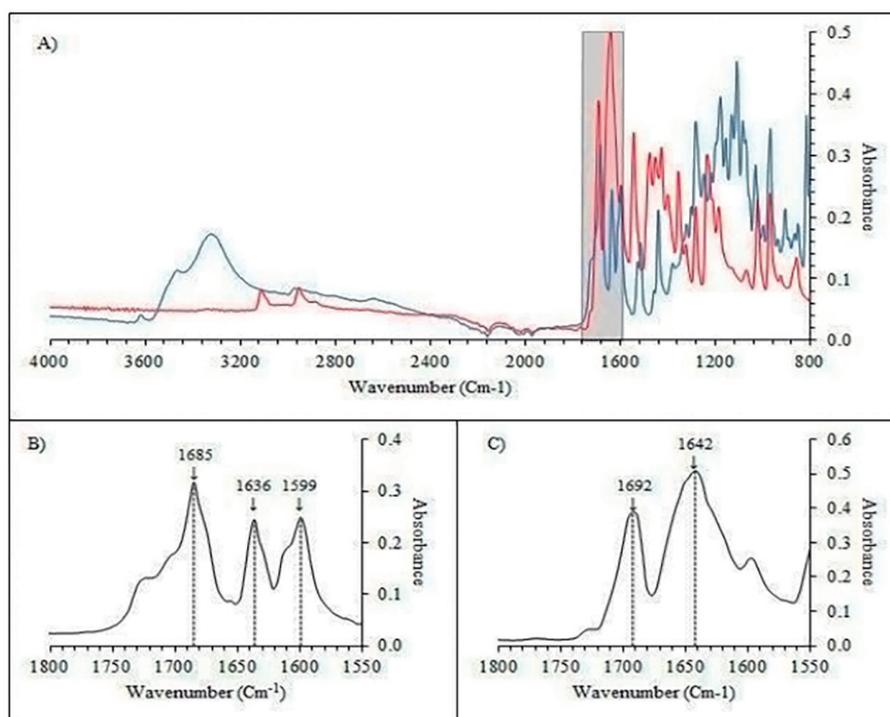


FIGURE 2 - A) FTIR spectral of pure chlorogenic acid and caffeine standards.

B) and C) enlargement of the 1600 to 1800 cm⁻¹ region in CGAs and Caffeine standards

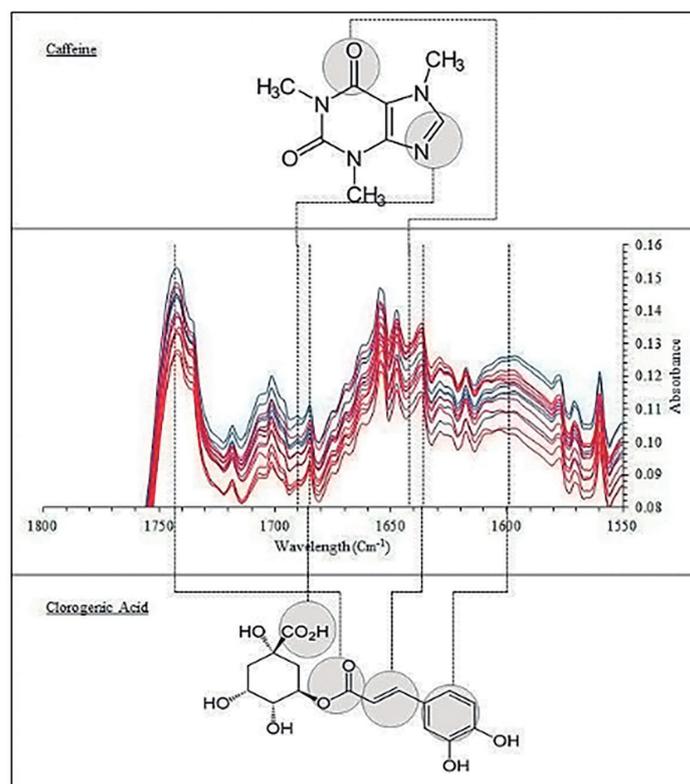


FIGURE 3 - FTIR spectrums for coffee pods AC (red) and EC (blue) samples (CGAs and caffeine molecules superimposed).

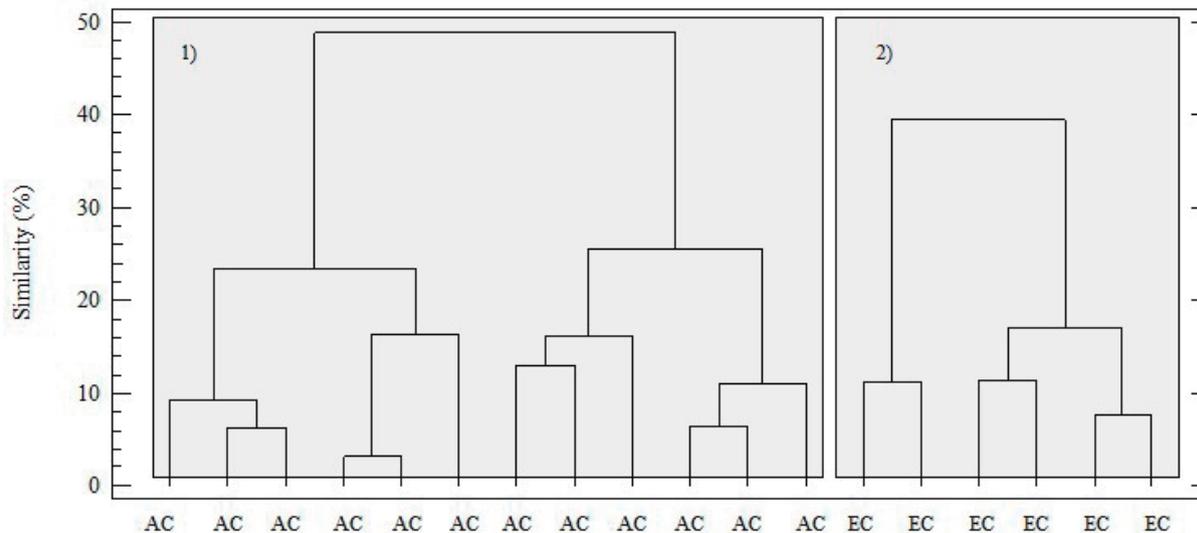


FIGURE 4 - Dendrogram for cluster analysis in americano (AC) and espresso (EC) coffee pods.

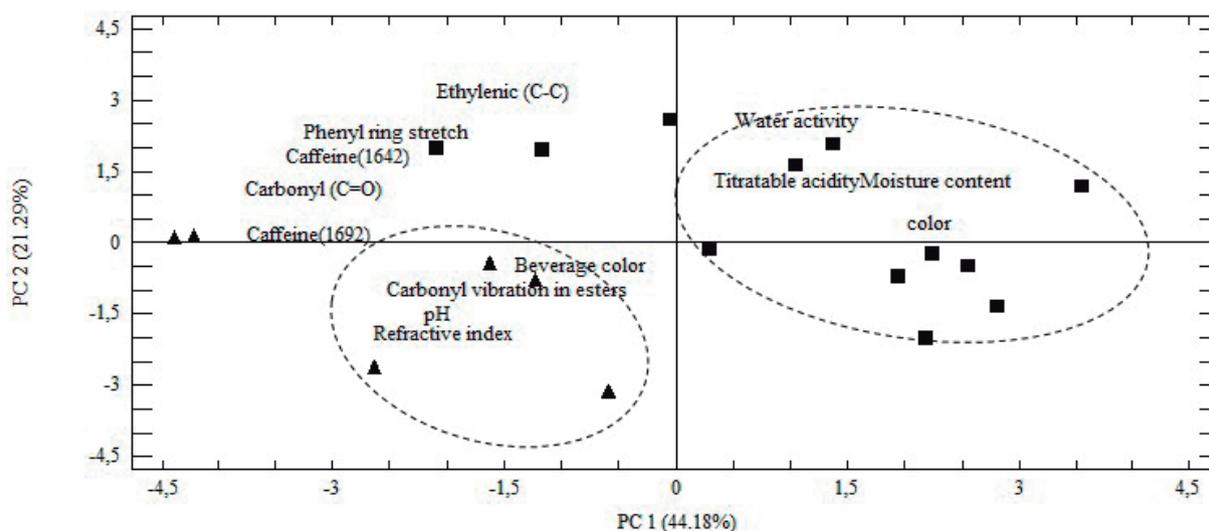


FIGURA 5 - Biplot comparison of espresso coffee pods (▲) and americano coffee pods (■).

The first two principal components, PC1 and PC2, accounted for approximately 65% of the variability. Two distinct groups can be perceived: AC coffee pod samples are located in positive PC1, while EC coffee pod samples are located in negative PC2 quadrant.

These results reveal that FTIR-based methods seem to be a promising alternative for the discrimination of coffee samples for the pods industry or for the type of consumption; in this study, the FTIR technique allow a discrimination between type of capsules for different extraction methods; so, it could be defined the type of capsules (espresso or *americano*) can be elaborated according to the type of coffee available.

4 CONCLUSION

A high moisture content and a_w was found in coffee powder AC. This can significantly affect sensory acceptance since high moisture contents can be indicators of product interaction with the environment given the high degree of hygroscopicity of the coffee.

A differentiation was identified between EC and AC coffee pods, PCA and cluster analysis results indicated that coffee samples could be separated into distinct groups, based on both. The absorbance values of the peaks related to representative functional groups in 1600 to 1800 cm^{-1} region and physicochemical parameters in coffee powder and beverages.

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