

Research Note

A comparative evaluation of methodologies for water content determination in green coffee

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Abstract

The main objective of this study was to compare methods for mass loss evaluation in green coffee to water content determination by Karl Fischer titration (KFT). The following methodologies were tested: (i) ISO 6673 (oven drying at 105 °C for 16 h); (ii) the reference method employed by the Brazilian Agriculture Ministry (oven drying at 105 °C for 24 h)—BRAMw, employing whole beans and BRAMg, employing ground beans; and (iii) infrared drying (IRD). Reference oven drying methodologies ISO 6673 and BRAMw presented results statistically equivalent ($p > 0.05$) to those from KFT in the moisture content range that is of interest for green coffee commercialization (8–13 g/100 g), whereas IRD results were lower than those for KFT. ISO 6673 and BRAMw also presented the highest values of correlation coefficients to KFT. Differences in moisture content determination became more significant for lower moisture content values (4–7 g/100 g), probably due to loss of organic volatile substances during drying and occurrence of moisture loss during sample grinding.

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1. Introduction

Water content determination is the most frequent analysis performed in food products and it is quite significant in many aspects (Isengard, 2001). Nearly every food product contains water and this parameter affects many others, both of physical and chemical nature. Evaluation of most chemical parameters is based on dry mass and therefore water content must be measured. Also, water content affects microorganism growth and enzymatic activity, affecting the stability and shelf-life of foodstuffs. As different methods are available for water determination, the question to which one is more appropriate still remains. The problem becomes more difficult due to the facts that water in food is distributed in different bonding states and that both the product itself (dry matrix) and its water content affect method performance (Isengard, 2001; Yazgan, Bernreuther, Ulberth, & Isengard, 2006).

Water content determination is critical for evaluation of green coffee quality. It affects mold growth, mycotoxin production, fermentation, physical, chemical and sensory parameters. As water is quite cheap compared to coffee, its amount is also interesting from a commercial point of view. Thus, a precise knowledge of water content in green coffee is fundamental to ensure quality, as it should be in the range of 8–13% to allow for safe transportation and storage (Clarke, 1985; Reh, Gerber, Prodolliet, & Vuataz, 2006).

Reference methods for water content determination in green coffee are based on oven drying. Such methods do not measure water content itself, but the mass loss under the heating conditions employed, which includes other volatile substances. The term moisture content is usually employed for this type of measurements, even though the term mass loss should be most accurate (Isengard, 2001). The major drawback associated to oven drying methods is that they usually require long measuring times, which can be overcome by using infrared dryers. However, depending on heating conditions, some level of decomposition and

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water formation due to Maillard reactions should occur (De Caro, Aichert, & Walter, 2001; Reh et al., 2006).

There are three standards issued by ISO International Standard (1978, 1983, 2001) for water content determination in green coffee: 1446 (slow drying at 48 °C under phosphorous pentoxide up to constant weight); 1447 (two step drying at 130 °C) and 6673 (16 h drying at 105 °C). A recent study by Reh et al. (2006) presented a comparison of those methods and showed they were highly correlated to each other (R^2 values above 0.99). They also showed that none of the procedures allowed complete drying of green coffee and concluded that ISO 6673 was appropriate for routine analysis since it was the one that required the least input of labor (one step of drying, no grinding required) and was found to be independent of climate conditions and the presence of forced ventilation. Therefore, ISO 6673 is one of the methods evaluated in the present study.

In Brazil, the reference method for water content determination in green coffee is based on oven drying at 105 °C for a 24 h period (Ministério de Agricultura e Reforma Agrária, 1992). Cabrera and Taniwaki (2003) compared this methodology to ISO 1447 and reported an average difference of approximately 1% between them. They also evaluated a rapid method (infrared drying (IRD) at 130 °C for 15 min) and found out that it underestimated moisture content in approximately 2% compared to ISO 1447.

The Karl Fischer titration (KFT) is the most important chemical direct method for the determination of water content in food products. It is based on the reaction of water with iodine in an alcoholic solution. Its major advantage with respect to weight loss techniques is the high selectivity for water coupled to the fact that it does not require sample heating (De Caro et al., 2001). However, its application as a routine method is limited due to its higher costs in comparison to oven drying methodologies and it is usually recommended for calibration purposes. It has not been employed as a reference for water evaluation in green coffee on the basis that it requires sample grinding and thus some water could be lost during the grinding step (Clarke, 1985; Reh et al., 2006). A collaborative study on this

method was performed in 1963 and differing results were obtained by the laboratories involved (Clarke, 1985). However, it is noteworthy mentioning that at that time automatic titration was not available.

In view of the above, the objective of this study was to compare KFT to mass loss methods for water determination in green coffee. Also, the effect of sample grinding was evaluated.

2. Methodology

2.1. Samples

High quality Arabica green coffee (Viçosa, MG, Brazil) previously classified by cup as soft (Farah, Monteiro, Calado, Franca, & Trugo, 2006; Franca, Mendonça, & Oliveira, 2005) was employed in all tests. The coffee was placed in shallow recipients and allowed to thermodynamically equilibrate for a 48 h period (Lot 0). The recipients were then closed until the beginning of the tests. The relative humidity was monitored and varied from 63% to 66%. In order to cover a wider range of water content, some coffee samples were submitted to drying and humidification procedures. The humidification procedure consisted on spraying distilled water over the coffee beans. The amounts employed were 1 g (Lot –1), 2 g (Lot –2) and 5 g (Lot –3) per 100 g coffee. Drying was performed in a convective oven with forced ventilation (model 400/3ND, Nova Ética, Brazil) at 200 °C for 30 (Lot 1), 60 (Lot 2) and 90 s (Lot 3). In all cases samples were let to equilibrate for 24 h in sealed containers prior to the analyses.

2.2. Water content determination

The present work aimed at a comparison of the drying methods presented in Table 1 to KFT. The methodologies evaluated were ISO 6673, the reference method employed by the Brazilian Agriculture Ministry (BRAM), using both whole and ground coffee beans, and IRD. All methods employed the same drying temperature (105 °C). Oven-based drying analyses were terminated at a specified drying

Table 1
Water content determination methodologies based on drying

Method	Equipment	Sample	Test conditions	Reference
ISO 6673 (oven drying)	Convective oven with forced ventilation (model 400/3ND, Nova Ética, Brazil)	5 g whole beans	105 °C, 16 h	ISO (1983)
BRAMw (oven drying)	Convective oven with forced ventilation (model 400/3ND, Nova Ética, Brazil)	5 g whole beans	105 °C, 24 h	Ministério de Agricultura e Reforma Agrária (1992)
BRAMg (oven drying)	Convective oven with forced ventilation (model 400/3ND, Nova Ética, Brazil)	5 g ground beans	105 °C, 24 h	Ministério de Agricultura e Reforma Agrária (1992)
IRD (infrared drying)	Infrared dryer (Mark 160 Top Ray, Bel Engineering)	5 g ground beans	105 °C, 2–5 min	^a

^aTest conditions based on the same temperature employed by reference methodologies (ISO 6673 and BRAMw).

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