Food Chemistry 208 (2016) 103–110

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

In-line monitoring of the coffee roasting process with near infrared spectroscopy: Measurement of sucrose and colour

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ARTICLE INFO

Article history:
Received 25 November 2015
Received in revised form 7 March 2016
Accepted 29 March 2016
Available online 30 March 2016

Keywords:
Coffee roasting
Near infrared spectroscopy
Sucrose
Colour
In-line monitoring
Chemometrics

ABSTRACT

In this work, a real-time and in-situ analytical tool based on near infrared spectroscopy is proposed to predict two of the most relevant coffee parameters during the roasting process, sucrose and colour. The methodology was developed taking in consideration different coffee varieties (Arabica and Robusta), coffee origins (Brazil, East-Timor, India and Uganda) and roasting process procedures (slow and fast). All near infrared spectroscopy-based calibrations were developed resorting to partial least squares regression. The results proved the suitability of this methodology as demonstrated by range-error-ratio and coefficient of determination higher than 10 and 0.85 respectively, for all modelled parameters. The relationship between sucrose and colour development during the roasting process is further discussed, in light of designing in real-time coffee products with similar visual appearance and distinct organoleptic profile.

1. Introduction

Coffee is not just a functional beverage but also a gourmet product widely appreciated. Therefore, the development of a differentiated coffee brand within the coffee market is of utmost importance to coffee roasters. This challenge requires a deep knowledge of the different characteristics of raw coffee varieties/origins and roasting conditions. Furthermore, keeping the quality of a developed blend presents an additional difficulty, as coffee is a natural product and it presents inherent crop-to-crop variability. A step toward the improvement of the coffee product control may be the in-line chemical monitoring of the coffee roasting process. Typically the roasting process is fast (less than 20 min) and it is performed at high temperatures (around 200 °C) (Eggers & Pietsch, 2001; Murkovic & Derler, 2006). In this work, two of the most important parameters used in coffee characterisation are analysed: low weight carbohydrates and colour.

Low molecular weight carbohydrates are among the main constituents of green coffee beans. Within this class of compounds, sucrose is the most abundant species and may account up to ca. 9% and 7% w/w dry weight basis (dwb) for Arabica and Robusta varieties, respectively (Arya & Rao, 2007). Fructose and glucose are also present, although in much lower levels (<1%) especially in coffee beans wet-processed after harvest (Knopp, Bytof, & Selmar, 2006). A decrease in all low molecular weight carbohydrates is known to occur during the roasting process. Sucrose may reach ca. 0.01% w/w dwb for a dark roasted coffee (Perrone, Donangelo, & Farah, 2008). The initial high concentration of sucrose and its subsequent pronounced decrease during roasting is, therefore, particularly impacting over coffee final quality. The main chemical reactions associated with sucrose are fragmentation, Maillard reactions and caramelisation (Arya & Rao, 2007). This compound is an aroma precursor and the major source of aliphatic acids (Ginz, Balzer, Bradbury, & Maier, 2000; Toci, Farah, & Trugo, 2006). Sucrose is one of the carbohydrates presenting higher sweetness impact. Therefore it is likely to contribute to the beverage sweetness if present at concentration levels above human perception threshold (Wrolstad, 2012). The determination of carbohydrates can be performed following the Somogyi-Nelson or Munson-Walker methodologies, and also through chromatographic techniques, due to their higher selectivity (Perrone et al., 2008). Still, none of these approaches is adequate for in-line measurements. In this context, the development of an analytical tool able to monitor sucrose concentration during the coffee roasting process is of high interest especially for coffee roasters that aim

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http://dx.doi.org/10.1016/j.foodchem.2016.03.114
0308-8146/

at controlling this parameter. To the best of our knowledge, application of NIRS to the in-line carbohydrates quantification during coffee roasting has not been exploited yet and, furthermore, no alternative methods were found to this aim. When comparing NIRS with chromatographic methods for sucrose determination, the following advantages and disadvantages may be pointed. NIRS selectivity, achieved through the use of mathematical multivariate methods and NIRS limits-of-detection, typically between 0.05% and 0.1% w/w, are worse than the ones obtained in chromatography. However, NIRS allows for in-situ and real time determinations at low cost, without sample pre-treatment or additional reagents consumption. Furthermore NIRS is less laborious and easier to manipulate, features that are more desired for routine analysis, thereby justifying this approach.

Coffee colour is also an important parameter in coffee processing as it is empirically used to define the end-of-roast. Colour still defines a classification criterion for coffee connoisseurs, ranging from light roast or "cinnamon roast" to very dark roast aka "French roast". The measurement of roasted coffee colour can be performed with colour disks (Ferraz et al., 2010) or resorting to the CIE L^*, a^* and b^* parameters. A major attention has been given to the L^* parameter (luminosity) as it has been successfully correlated with the degree of coffee roasting (Wei & Tanokura, 2015). Furthermore, L^* has been used as a normalising factor for different roasting time-temperature programs (Altaki, Santos, & Galceran, 2011) or as a direct index of the total thermal effect (Sacchetti, Di Mattia, Pittia, & Mastrocola, 2009). The colour prediction of roasted coffee beans using near infrared spectroscopy (NIRS) was investigated by Pizarro, Esteban-Diez, Gonzalez-Saiz, and Forina (2007), exploiting a chemometric strategy based on off-line spectra measurements; Bertone, Venturello, Giraudo, Pellegrino, and Geobaldo (2016) developed a unique chemometric model for colour and variety ratio prediction also based on off-line NIRS measurements.

Making in-line measurements through the roasting process is the most reliable and effective methodology for the real-time estimation of the process end-point ensuring a product with the desired properties. However, very few analytical approaches were described in this context (Goodman, Pascual, & Yeretzian, 2011; Hernandez, Heyd, & Trystram, 2008). NIRS has appropriate characteristics for an in-line implementation in this type of processes. It does not require sample pre-treatment and provides measurements typically within seconds. In spite of several applications of NIRS in the coffee analysis context (Barbin, Felicio, Sun, Nixdorf, & Hirooka, 2014), the use of this technique as an in-line analytical tool for monitoring the roasting process is, to the best of our knowledge, described for the first time in monitoring titratable acidity (Santos, Lopo, Rangel, & Lopes, 2016).

The aim of this work is the development of a NIRS-based analytical tool to monitor in-line sucrose concentration and colour, directly inside the coffee roaster. Different coffee origins and varieties were roasted according to different experimental conditions. The process was sampled periodically and sucrose and colour determined resourcing on chromatography and CIE $L^*a^*b^*$, respectively. Chemometric models for the estimation of sucrose and CIE $L^*a^*b^*$ based on NIR spectra were developed. The correlation between sucrose concentration and colour development through the coffee roasting process is further discussed in light of potential exploitation for coffee products design.

2. Materials and methods

2.1. Roasting process and sampling

Roasting trials were performed using two Arabica samples, from East-Timor and Brazil, and two Robusta samples from Uganda and India. A coffee roaster (Hottop Coffee roaster KN-8828B-2K), which allows direct visualisation of the roasting drum through a frontal observation glass window, was used. Two different roasting programs (slow and fast roasting programs) were defined for all coffee samples (Santos et al., 2016). During the slow roasting program, the coffee beans were continuously heated for 25 min up to 183 °C. During the fast roasting program, the coffee beans were continuously heated for 18 min up to 220 °C. Coffee beans were poured into the coffee roaster when the roasting drum temperature reached 75 °C. Batches of 250 g of green coffee beans were used for each roasting trial. Coffee sampling during the roasting procedure was performed respectively after 5, 8, 12, 14, 16 and 18 min for fast roasting assays; and after 5, 10, 13, 16, 19, 22 and 25 min for slow roasting assays. Each sampling implied to stop the roasting procedure at the end of each referred roasting period and collecting the whole 250 g of coffee batch.

2.2. Low weight carbohydrates determination by HPLC-RI

2.2.1. Reagents and solutions

D(+)-sucrose (⩾99%-HPLC) and D(-)fructose (⩾99%-HPLC) were from Fluka (St. Louis, MO, USA). Acetonitrile (HPLC grade) and ethanol (analytical grade) were from Merck (Darmstadt, Germany). Water was purified with a Milli-Q System (Millipore, Bedford, MA, USA).

Low weight carbohydrates stock solutions (20 mg mL^{-1}) were prepared in a mixture of water:acetonitrile (50:50, v/v) after rigorous weighing of the solid and stored at 4 °C until use. Diluted standard solutions were prepared daily by rigorous dilution of the stock solutions with the same solvent. Solvents for HPLC were degassed under vacuum for at least 15 min before use.

2.2.2. Extraction procedure

The experimental procedure followed for the low molecular sugars extraction was adapted from Knopp et al. (2006). One hundred milligrams of ground coffee samples were rigorously weighed into a centrifuge tube and mixed with 5 mL of ethanol (80% v/v). The suspension was stirred for 30 s and then treated in an ultrasonic bath (FungiLab SA, Barcelona, Spain) for 15 min at 70 °C to carry out the extraction step. Subsequently, the mixture was centrifuged at 5000*g* at 4 °C for 10 min for separation of the liquid extract from the solid matter. Thereafter, 2.5 mL of the supernatant were measured and left under a nitrogen stream to a volume of 500 μL in order to reduce the ethanol fraction. The final volume of the liquid extracts was then rigorously adjusted to 1 mL with acetonitrile and left to stand for 20 min. Under these conditions, a final extract solution more similar with the mobile phase was attained, avoiding possible precipitate formation during the chromatographic analysis.

The final solution was centrifuged at 5000*g* for 10 min at 4 °C and filtered through a 0.22 μm PTFE filter prior to injection.

All extractions were carried out in duplicate and/or following a concordance criteria of RSD < 7% for carbohydrates concentration higher than 0.2 g/100 g and of RSD < 15% for carbohydrates concentration lower than 0.2 g/100 g.

2.2.3. Low weight carbohydrates determination

The chromatographic analyses were performed using an HPLC (Jasco, Japan), equipped with a quaternary pump (PU-1580), a refractive index detector (RI 132 Gilson, Middleton, WI, USA) and an injection valve (Rheodyne 7125, Cotati, CA, USA) fitted with a 20 μL stainless steel sample loop. The HPLC system was controlled via the Borwin PDA Controller Software (JMBS Developments, Le Fontanil, France). Carbohydrates separation was performed on an amino column Tracer excel 120 APS (5 μm particle size, 250 mm length × 4.6 mm i.d.) from Teknokroma (Barcelona, Spain). The