Sensors and Actuators B 137 (2009) 715–721

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

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

Sequential injection ATR-FTIR spectroscopy for taste analysis in tomato

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ARTICLE INFO

Article history:
Received 17 October 2008
Received in revised form 19 January 2009
Accepted 28 January 2009
Available online 10 February 2009

Keywords:
Sequential injection analysis ATR-FTIR
Tomato
Central composite design
Multivariate data analysis
Taste

ABSTRACT

The potential of attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) for food analysis has been demonstrated in many publications. Most applications, however, deal with the major disadvantage of extensive sample manipulation and cleaning of the ATR sampling compartment, which greatly reduces the sampling rate. To address this problem, in this paper, the potential of sequential injection analysis (SIA) in combination with ATR-FTIR has been studied for sugar and organic acid analysis of Belgian tomato samples. SIA allows automation of the sampling and measurement protocol. The SIA-ATR-FTIR system is optimized for measurement performance accuracy using a central composite design. The four important operational parameters studied are spectral resolution, number of co-added scans, flow rate, and sample temperature. A clear optimum is found for the number of scans (256 co-added scans) and resolution (16 cm^{-1}) while sample temperature and flow rate are found not to significantly influence the measurement performance on the most important taste components in fruit. Hence, a stopped flow analysis – to save sample – at room temperature with 256 co-added scans at a resolution of 16 cm^{-1} is selected as optimal for tomato juice measurements. This setup results in a sample throughput of 24 samples/h. Different tomato cultivars were discriminated based on their acid and sugar content, which are the most important chemical components determining tomato taste. Prediction models for D-glucose, D-fructose, citric acid and L-glutamic acid concentrations in tomato samples were successful for the measured tomato cultivars with RMSEP-values of 1.81 g L^{-1}, 1.63 g L^{-1}, 0.41 g L^{-1} and 0.35 g L^{-1} respectively. However, the prediction model for L-malic acid, which is present in very small concentrations in tomato fruit, was not successful.

1. Introduction

Traditionally, sensory and instrumental techniques are used to determine the taste of food products. Although sensory panel analysis is by far the most realistic technique to obtain information on human taste and aroma perception, it has some serious drawbacks, being the correctness of training, standardization of measurements, reproducibility, high cost and taste saturation of the panelist [1]. Instrumental techniques such as high pressure liquid chromatography (HPLC), gas chromatography (GC), soluble solids content (SSC), titratable acidity (TA) and pH give information on the chemical composition of the sample and could be used to describe the taste of a food product. These traditional techniques, however, often require a laborious and time-consuming sample preparation and skilled people to operate the equipment [2].

In food research there is a need for objective high throughput taste profiling to complement sensory panels. Fourier transformed infrared spectroscopy (FTIR) has proven to be a good alternative for traditional chromatographic techniques in the analysis of individual chemical components in food. Using FTIR with advanced optics and a high signal-to-noise ratio, detection of individual components as well as small compositional differences between complex samples is possible [3–5]. Attenuated total reflectance (ATR) as a sampling technique for FTIR measurements offers interesting possibilities for the analysis of liquid and solid foods. Examples of the use of this technique in food analysis are the determination of sucrose in beetroot [6], organic acids and sugars in tomato [2] and apple juices [7,8], caffeine in soft drinks [9] and alcohol in distilled liquors and wines [10]. In the last decade there has been a shift towards automation of FTIR-measurements by implementing peristaltic pumps, or flow injection (FIA) and sequential injection analysis (SIA) systems which results in an increased throughput. Compared to FIA, SIA is considered as more robust, versatile and better adapted for multiparametric and stopped-flow techniques [11]. Many sample

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doi:10.1016/j.snb.2009.01.056

Table 1
Composition of four mixtures used for the optimization of the SIA-ATR-FTIR system. Concentrations are given in g L^{-1}.

	D-Glucose	D-Fructose	Sucrose	Citric acid	L-Malic acid	Tartaric acid	Quinic acid
a.							
Mixture 1	20	20	5	10	30	10	15
Mixture 2	40	40	20	5	15	15	10
Mixture 3	10	10	30	30	10	5	5
Mixture 4	30	30	10	20	5	2	2
b.							
Mixture 1	20	20	5	10	20	5	5
Mixture 2	40	40	20	5	15	10	10
Mixture 3	10	10	30	30	10	2	7
Mixture 4	30	30	10	20	5	7	2

and instrumental parameters determine the measurement performance and accuracy of this technology. It is therefore important to optimize the operational parameters for the quantification of the main taste components. Different flow systems have been developed and reported successful for the discrimination of foodstuff, e.g. red wines, edible oils and beer [12–14] or the rapid quality control of spirit drinks and beer [15]. Special interest has been shown for the determination of individual taste components such as sucrose and glucose [16,17] and sugars in soft drinks [18].

The objective of this study is to develop and optimize an SIA-ATR-FTIR measurement technique for high throughput analysis in large scale experiments such as the taste screening of new Belgian tomato cultivars. Hereto firstly, a SIA-ATR-FTIR system will be developed and optimized for quantification of the main taste components in fruit samples, which include D-glucose, D-fructose, sucrose, citric acid, L-malic acid, tartaric acid, and quinic acid. Secondly, using the set of optimal operational parameters, the ability of SIA-ATR-FTIR as an optical tongue to classify tomato cultivars according to their taste profile will be studied. Finally, the measurement performance to quantify the concentrations of the main taste components, specifically for tomato fruit, will be evaluated and validated with the results of an enzyme based reference technique. These components include D-glucose and D-fructose as the most important sugars and citric acid, L-glutamic acid and L-malic acid as the most important organic acids [19,20]. With respect to tomato fruit, vibrational spectroscopy has been used in the past for the measurement of lycopene and carotene content [21] or for metabolic fingerprinting of salt-stressed tomatoes [22].

2. Experimental

2.1. Development and optimization of the SIA-ATR-FTIR system

2.1.1. Mixtures

Four mixtures, composed of the main taste components in fruit samples [23], including three sugars (D-glucose, D-fructose and sucrose) and four acids (citric acid, L-malic acid, quinic acid and tartaric acid) were prepared in triplicate for analysis with the SIA-ATR-FTIR system. The composition of the mixtures is shown in Table 1a and covers the concentration range of the different components in the fruit samples. Stock solutions of the mixtures were prepared and stored at −80 °C until measurement. As such calibration in the optimization study is based on 12 measurements.

In an extra experiment to complete the optimization of the SIA-ATR-FTIR system four (slightly) different mixtures of the same sugars and acids were prepared in triplicate. The composition of the four mixtures is shown in Table 1b. Stock solutions of the mixtures were prepared and also stored at −80 °C until measurement.

2.1.2. ATR-FTIR

The ATR-FTIR measurements were performed on a Bruker Tensor 27 spectrometer (Bruker, Karlsruhe, Germany) equipped with a mid-infrared source and a mercury-telluride detector (MCT). The sampling station was equipped with a flow-through horizontal attenuated total reflectance (ATR) accessory with multiple reflections (9 reflections) (PIKE Technologies, Madison, USA). A closed AMTIR (amorphous material transmitting infrared light) with a channel for sample containment (0.5 mL) was used. Background spectra were created between every measurement using distilled water. The number of co-added scans and the resolution were optimized in the flow system. Single beam spectra in the range of 1800–800 cm^{-1} were obtained and corrected against the background to present the spectra in absorbance units. Opus software version 5.5 (Bruker, Karlsruhe, Germany) was used to operate the FTIR spectrometer and collect all the data. Measurements were performed on the mixtures of pure components and on the centrifuged and filtered tomato juices. During ATR-FTIR measurement the samples were placed in a temperature controlled water bath (Julabo TW8, VWR, Belgium).

2.1.3. Flow system

The flow injection system was constructed with a milliGAT® pump, combined with a Microlynx-4 micro-electric controller (Global FIA Inc., Fox Island, USA). A large advantage of the pump is the redundancy of refill cycles or syringe changes unlike traditional syringe pumps. The stepper motor of the pump is fully computer controllable. The milliGAT® pump was connected through Teflon tubing (1/16"OD 0.030"ID, Alltech Associates Inc., Deerfield, USA) to the ATR cell. A valve (Cheminert, C22Z-2180D, Valco Instruments Co. Inc., Houston, USA) was introduced in the system to switch between sample and cleaning reservoir. The valve position, flow rate and direction were controlled by a homemade program written in Labview 8.0 (National Instruments Co., Austin, USA). Initially, distilled water is pumped through the cell for about 60 seconds at a flow rate of 6 mL min^{-1}. Subsequently the sample is pumped through the crystal for 30 seconds at the flow rate of interest. After scanning the sample (30 seconds), distilled water is pumped through the system during 90 seconds to clean the crystal and to obtain the background spectra. The use of this sequence results in a throughput of 24 samples/h.

2.1.4. Optimization design

Four parameters were selected for optimization of the SIA-ATR-FTIR system: spectral resolution, number of co-added scans, flow rate and sample temperature. The first three instrumental parameters determine the measurement accuracy, the sample volume and the measurement time, while the fourth one might influence the repeatability of the measurements as influenced by environmental factors. A central composite design (CCD) was used for the optimization of the four factors. This type of design has a hyper spherical symmetry and requires five levels for each factor [24]. An overview of the levels per factor is given in Table 2. The number of co-added scans was expressed as a power of two to obtain equidistant levels for this factor. Compared to a full factorial design,