Journal of Food Engineering 214 (2017) 79–89

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Effect of maturation on the bulk optical properties of apple skin and cortex in the 500–1850 nm wavelength range

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ARTICLE INFO

Article history:
Received 1 February 2017
Received in revised form
19 April 2017
Accepted 15 June 2017
Available online 18 June 2017

Keywords:
Apple
Double integrating spheres
Unscattered transmittance
Absorption
Scattering
Anisotropy factor

ABSTRACT

To facilitate the design of new optical measurement systems for biological systems, the knowledge of light propagation properties is essential. Therefore, the bulk optical properties of skin and cortex of three apple cultivars were studied during maturation, in the 500 nm–1850 nm range. A clear absorption signature was observed with absorption peaks which can be related to present anthocyanins, chlorophyll, carotenoids and water. During maturation, the skin absorption at 550 nm increased in the bicolored cultivars '*Braeburn*' and '*Kanzi*', while the absorption at 680 nm decreased in the cortex. Both the bulk scattering coefficient and the anisotropy factor were larger for the skin compared to the cortex tissue. Also during maturation, the skin scattering increased in the two bicolored cultivars, while a general decrease was seen in the apple cortex. Physiological changes during maturation, like cell growth, the formation/degradation of pigments and the formation of a cuticle layer on the skin, may explain the observed evolutions. As a result, using the bulk optical properties, these physiological changes can be monitored and linked to the maturity stage in the orchard, supporting the selective harvest of apples.

1. Introduction

Optical measurement techniques like visible (vis) and near-infrared (NIR) spectroscopy (Nicolaï et al., 2014), hyperspectral imaging (Boldrini et al., 2012), spatially resolved spectroscopy (Van Beers et al., 2015) and time resolved spectroscopy (Torricelli et al., 2013) have shown to be valuable for the nondestructive quality measurement of different types of intact fruits. Photons entering a material can be absorbed by the molecular bonds, or scattered at mismatches in the refractive index within the turbid product. The absorption, therefore, refers to the chemical properties of the material, while the scattering of photons is related to the (micro) structure (Lu, 2004). Firmness, crispiness and mealiness are important fruit quality properties which are associated with the microstructure of the apple cortex. As the latter influences the light propagation through the tissue, these properties might be extracted from the signals measured by optical sensors. Yet, in non-destructive measurements, the photons will first have travelled through the pigmented skin layer before reaching the apple cortex (Saeys et al., 2008), therefore influencing the obtained signal. Consequently, the signals measured by optical systems contain information on absorption and scattering of both the apple skin and cortex. Thus, the correlation between quality parameters and the optical output is generally based on chemical and physical attributes of both layers, which complicates the construction and interpretation of calibration models. Therefore, the optical properties of both apple skin and cortex should be better understood, to allow light propagation models to be used as a tool for the development of new optical measurement techniques.

The propagation of light in turbid media, like biological materials, can be described using the bulk optical properties (BOP): the bulk absorption coefficient μ_a, the bulk scattering coefficient μ_s and the angular scattering pattern, represented by the normalized scattering phase function $p(\theta)$ (Aernouts et al., 2014). The phase function describes the normalized scattering probability as a function of the scattering angle θ. However, this function is often too complex to interpret and is, therefore, represented by the anisotropy factor g, which equals the mean cosine of the scattering

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http://dx.doi.org/10.1016/j.jfoodeng.2017.06.013
0260-8774/

angle (Aernouts et al., 2013). The wavelength dependent scattering direction of vis/NIR light in biological tissues can often be described with an anisotropy factor in-between isotropic Rayleigh scattering ($g = 0$) and complete forward scattering ($g = 1$) (Aernouts et al., 2015b,c). The reduced scattering coefficient μ_s' combines the bulk scattering coefficient and the anisotropy factor into a single parameter, which is sufficient to describe light scattering in the diffusion regime (Tuchin, 2007).

Several researchers have tried to estimate the bulk optical properties of apple tissues with non-destructive optical measurement techniques such as spatially resolved spectroscopy (Cen et al., 2013; Nguyen Do Trong et al., 2014; Qin et al., 2009; Qin and Lu, 2008) or time resolved spectroscopy (Cubeddu et al., 2001; Seifert et al., 2014), in combination with inverse light propagation models (Torricelli et al., 2013). However, it is difficult to estimate the properties of both skin and flesh simultaneously from such measurements. Moreover, most studies on apples focused on the estimation of μ_a and μ_s' (Cen et al., 2013; Lu et al., 2010; Rowe et al., 2014), while less attention has been given to the actual bulk scattering coefficient μ_s and the scattering anisotropy g. If *in vitro* characterization of a thin (mm-range) homogeneous sample slab is feasible, the total reflectance and transmittance can be measured with integrating spheres, generally accepted as the 'golden standard' method to estimate BOP for biological tissues (Aernouts et al., 2013; Bashkatov et al., 2005; López-Maestresalas et al., 2015; Rowe et al., 2014; Saeys et al., 2008; Zamora-Rojas et al., 2013). Moreover, this technique allows to measure BOP of separate tissue layers. In addition, if the BOP can be monitored during maturation, changes in the measured BOP could be related to physiological changes during this period. This would enable the design of non-destructive optical measurement techniques to monitor apple maturity, supporting the selective harvest and an accurate harvest time prediction.

Therefore, in this research, the evolution of the bulk optical properties (BOP) of apple skin and cortex were studied during maturation, as well as their relation with relevant apple quality and ripeness indicators. Three different apple cultivars, two bicolored (*'Braeburn'* and *'Kanzi'*) and one green cultivar (*'Greenstar'*), were tested. Reliable double integrating sphere (DIS) and unscattered transmittance (UT) measurements were used to obtain accurate estimates for the bulk absorption coefficient μ_a, the bulk scattering coefficient μ_s and the scattering anisotropy factor g.

2. Materials and methods

2.1. Apple samples

Apples of the cultivars *'Braeburn'*, *'Kanzi'* and *'Greenstar'* were harvested during the 2014 season in Belgium over a timespan ranging from 45 d before to 17 d after the start of the optimal harvest window. All apples were freshly picked at the 'Centre Fruitier Wallon' in Merdorp, Belgium (50°38′32.7″N 5°00′13.6″E) on the day of the optical measurements. Due to the experimental nature of this orchard, not all apples were removed from the tree in the determined harvest window, and apples were still available for picking until 17 d after the harvest window started. The Belgian harvest windows for the different cultivars were determined by the Flanders Centre for Postharvest Technology (VCBT) based on the evolution of firmness, starch index, soluble solids content (SSC), acidity, size, background color and Streif index after comparing them to historical records (Peirs et al., 2005). Due to differences in the harvest window between cultivars, not all apples were measured over the exact same time span. Weekly, 5 apples per cultivar were harvested in the mid-section of the tree. Accordingly, a total of 50 *'Braeburn'* apples, 35 *'Kanzi'* apples and 40 *'Greenstar'* apples were picked and the BOP of the skin and fruit cortex were measured.

Parallel to the optical characterization of apple skin and cortex, additional apples of the same cultivars and location were harvested for destructive analysis on the same days. The SSC (°Brix) of the apple juice was measured using a digital refractometer (PR-101α, Atago, Tokyo, Japan). As a measure of apple firmness, the maximum force to puncture the apple for 8 mm using an 11 mm diameter plunger at 8 mm/s was determined using a universal testing machine (LRX, LLOYD Instruments Ltd., Hampshire, UK) (Van Beers et al., 2015). The SSC and firmness were determined on a set of 20 apples per picking date, while for the starch conversion value a different set of 10 apples was used, to ensure this measurement was performed as close as possible to the moment of harvest (Peirs et al., 2005). The starch conversion value was determined using a starch conversion chart (scale from 1 to 10, Ctifl, Paris, France). After performing a Lugol's test, which stains the present starch particles using a KI/I_2 solution on equatorially cut apples, the resulting staining pattern was visually compared to the chart. Based on the discoloration over the entire surface, a score between 1, a high discoloration, and 10, a low discoloration, was defined (Peirs et al., 2002). All these parameters were measured by the Flanders Centre for Postharvest Technology (VCBT) according to the ISO/IEC 17025 standard.

2.2. Sample preparation

Both apple skin and cortex samples were optically characterized on the sun exposed side (blush side), resulting in two measurements per apple. As this side is exposed, it is most interesting for future field measurements. To only obtain the apple skin, first a slice of 6 mm in thickness was cut from the apple using a meat slicer (Junior sup 19 mod 30–595A, CAD, Italy). After this, the excess fruit cortex was scraped off using a scalpel. This remaining skin layer essentially consisted of the cutin layer plus the epicuticular wax, the epidermis and probably also some layers of hypodermis cells. Next, these skin samples were cut into disks of 30 mm in diameter and placed in a glass cuvette. The custom-made glass cuvette consisted of two parallel 1.1 mm thick glass plates (Borofloat33, Schott, Germany) separated by a spacer of 0.55 mm in thickness. This spacer had a round hole with a diameter of 30 mm. By putting the sample in the provided space, the sample was positioned between the two 1.1 mm glass plates. As the apple skin samples were typically thinner than 0.55 mm, demineralized water was added to remove any unwanted air bubbles and to reduce the refractive index mismatches at the boundaries between the glass plates and the sample. The apple cortex samples were obtained by slicing the apple at the same place where the skin sample was removed. The same meat slicer was used to slice the apple cortex to a thickness of about 0.50 mm. These slices were again loaded in a similar custom made glass cuvette after which demineralized water was added. For both the apple skin and cortex samples, the thickness was measured in triplicate using a digital caliper before loading the samples into the glass cuvette. Also, if obvious defects in one of the samples (e.g. cracks, air bubbles,…) occurred, this sample was not measured and a new sample was prepared.

Once the samples were prepared, the glass cuvette was placed into the sample holder between the double integrating spheres to measure the total reflectance and transmittance. All samples were measured directly after preparation, by which drying and browning effects due to enzymatic reactions were minimized. Repositioning of the sample in a different measurement path allowed to measure the unscattered transmittance (Aernouts et al., 2013).