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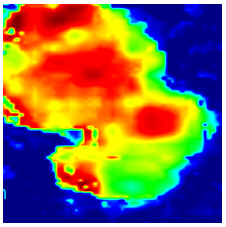
Raman Spectroscopy

Characterisation of Encapsulated Flavours using Raman Spectroscopy



Application Note

Food Beverage RA52

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Abstract

Raman spectroscopy has been used to analyse the process of micro-encapsulation of flavours. This contribution shows the example of distribution of limonene and quantification of its content within the micro-particles.

Key words

flavour analysis, micro-encapsulation, Raman spectroscopy, Raman imaging, quantitative analysis

Introduction

Micro-encapsulation of flavours has become of great importance in the flavouring and food industries. It involves the mixing of liquid flavours with a carrier to obtain a dry flavour powder, easier to handle than the liquid form. This technique provides protection against degradative reactions, prevents the loss of flavour, and also enables the controlled release of flavours during food processing and storage. Up to now, gas chromatographic (GC) methods have been extensively applied for the determination of individual components of essential oils. Raman spectroscopy has become an increasingly important tool in characterisation of encapsulated flavours. In this study we investigated the suitability of Raman spectroscopy for the distribution of limonene – the main component in citrus oils - in spray-dried particles, as well as its quantification.

Distribution of the flavour within micro-particles

Spray drying - a method of producing a dry powder from a liquid or slurry by rapidly drying with a hot gas - is the predominant encapsulation process of flavours. Efficient spray drying results in homogeneous distribution of the flavour within the carrier, which is critical to ensure an optimal controlled release of the flavour. A 30 µm spray-dried particle consisting of maltodextrin (carrier) and limonene (flavour) was mapped and analyzed by Raman microspectroscopy to examine the distribution of the flavour on the microscopic scale.

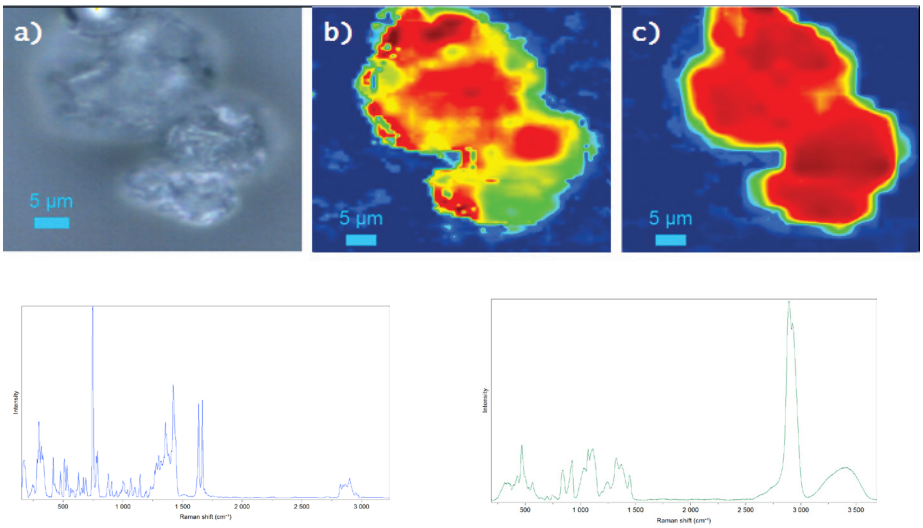


Figure 1: Microscopic image of spray-dried sample (a), distribution of limonene (b) and maltodextrin (c) according to DCLS model, spectra of limonene (d) and maltodextrin (e).

Monitoring the spectral features of limonene and maltodextrin within the mapping dataset enables the construction of chemical images showing the distribution of each compound. The chemical image of maltodextrin shows homogeneous coverage over the particle: this is logical since maltodextrin is the carrier. More informative is the pretty uniform distribution of limonene, suggesting an efficient spray-drying process.

Quantitative determination of the encapsulated limonene

Quantifying the amount of flavour in the dried powder is important to monitor the stability of flavours in micro-particles over time. Traditional GC-FID (Gas chromatography - flame ionization detector) methods are the standard procedures to determine these contents, but they are time-consuming compared to Raman spectroscopy. After a simple extraction of the limonene in cyclohexane, a calibration curve based on Raman spectra of these solutions was built for the concentration range 2.0–16.7 %, by correlating the area of a limonene peak with the concentration of the standard samples.

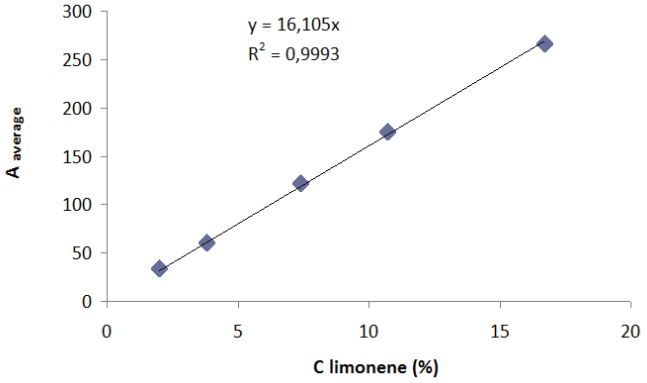
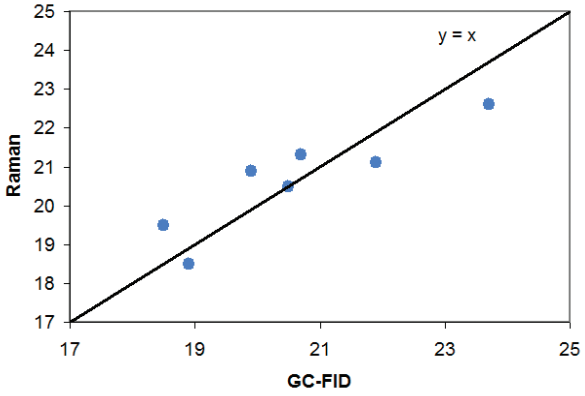


Figure. 2: Calibration curve for limonene in cyclohexane ($A_{average}$ is the area under peak at 1678 cm^{-1}).

A seven sample test set analyzed with the GC-FID reference method and the Raman calibration gave similar results with the two techniques (Fig. 3). The limonene concentration in the spray-dried particles that were examined was found to be in the range 18.5–23.0 % ($w_{limonene}$/w_{powder}). The relative difference in content of limonene obtained with both methods was within 5.0 %.

The concentrations given by the Raman in the table are outside the calibration range as they were re-calculated to take into account the dilution factor introduced during the preparation of the samples. The main advantage of RS method over the existing GC-FID method is its simplicity, immediacy and speed, and that it is non-destructive.



	Raman	GC-FID
Sample 1	22,6	23,7
Sample 2	20,9	19,9
Sample 3	21,1	21,9
Sample 4	18,5	18,9
Sample 5	19,5	18,5
Sample 6	20,5	20,5
Sample 7	21,3	20,7
Average	**20,6**	**20,6**

Figure. 3: Limonene concentration determinations of a 7 sample test set using both GC-FID and Raman techniques.

Summary

Raman spectroscopy has proven to be a powerful, rapid and reliable technique that can be used to quantify limonene and show its distribution in spray-dried samples. Based on these findings, the application of Raman spectroscopy for monitoring the stability of flavours in microparticles can be envisaged.

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