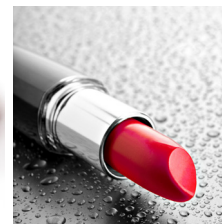


Soap compound investigation by Raman mapping



Application
Note

Cosmetics
RA69

Thibault Brulé, Application Engineer, HORIBA Scientific, Palaiseau, France

Abstract

Soap compound investigation is an important point in quality control in the soap industry. In this application note, we demonstrate how Raman micro-spectroscopy can be a powerful technique to investigate the composition of soap bars by studying different types of hard soaps.

Introduction

In the soap industry, many chemical reactions are involved for the preparation of the final product. A tight control of the reactions is a major part of the quality control mission in these industries in order to ensure the non-invasivity of the final product on human skin. Many soaps also contain additives as fragrances or dyes that should improve the commercialized result. However, these additives could be involved in chemical reactions and their presence in the final bar could be questioned. In this application note we show how Raman micro-spectroscopy, and more specifically Raman mapping, can help to investigate the presence of additives and/or traces of reagents in soap bars.

Experimental conditions

Raman Spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure, phase, polymorphism, crystallinity and molecular interactions. It is based upon the interaction of light with the chemical bonds within a material. The system used is an XploRA™ PLUS. It incorporates unique and powerful functions in a reliable, high performance system, ideally suited to the research and analytical labs. It is fully confocal, providing high image quality, spatial and depth resolution.

As a complex product, soaps may contain various organic compounds that may scatter fluorescence when interacting with a laser source. As we would like to obtain the proper Raman signal, it is necessary to reduce as much as possible the fluorescence emission. Depending on the fluorescence level scattered by the different soaps, different lasers (638, 785 nm) have been used to acquire the Raman fingerprints of the compounds. In order to find additives

and/or reactive traces, and to obtain the chemical distributions of the different compounds, soaps have been mapped with the laser using a 50xLWD objective. The acquisition time was one second per pixel of 2x2 μm-sizes.

Multiple soap bars were studied: Aleppo soap, olive oil soap, grape seed oil soap, Marseille's soap, and a low-cost soap. The three first soaps are handmade, and the two others are industrial products.

In terms of composition, soaps are traditionally an association of fatty acids / oils, glycerol, and many additives like perfumes, dyes, emulsifiers, etc. All of these additives aim to enhance the final product.



Figure 1: Pieces of soap studied by Raman spectroscopy. (From left to right) Aleppo soap, olive oil soap, grape seed oil soap, Marseille's soap, low-cost soap.

Results

For each soap, a Raman map were acquired. In order to highlight the different compounds, a Multivariate Curve Resolution (MCR) analysis was applied on the different maps. For each map, the colors are based on the decomposition on the associated reference spectra. These obtained reference spectra were then compared with the Raman KnowItAll®

databases provided with LabSpec6 software and powered by BIO-RAD.

We first analyzed the handmade soaps. The Raman maps of the different soaps are shown on Figure 2.

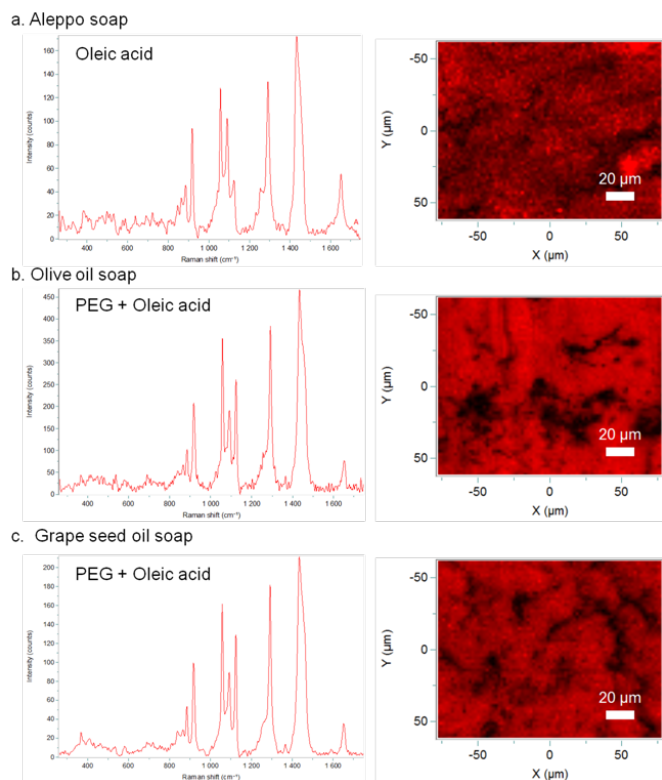


Figure 2: Raman reference spectra and Raman maps of different handmade soaps. Raman spectral identifications based on KnowItAll® databases. (a) Aleppo soap. (b) Olive oil soap. (c) Grape seed oil soap.

Based on the Raman analyses, it is clear that the main compound of all soaps is oleic acid. Poly-ethylene glycol (PEG) contribution is also obtained in the more manufactured handmade soaps. PEG plays the role of emulsifier in the soap composition. This higher complexity in the formulation, compared to the Aleppo soap was expected. Indeed, Aleppo soap, is a very simple soap made from olive oil and lye. Instead of the addition of PEG in the olive oil and grape seed oil based soaps, the distributions of the elements are relatively homogeneous and no other compound were found for these soaps. This homogeneity is the direct result of the handmade manufacturing during which all soap elements are highly mixed.

In order to understand the lack of differences between the two complex handmade soaps, it is important to notice that these soaps were manufactured by the same company.

That explains the homogeneity between the two soaps formulations/composition.

The second type of analyzed soaps were industrial soaps. The Raman maps of the different soaps are shown on Figure 3.

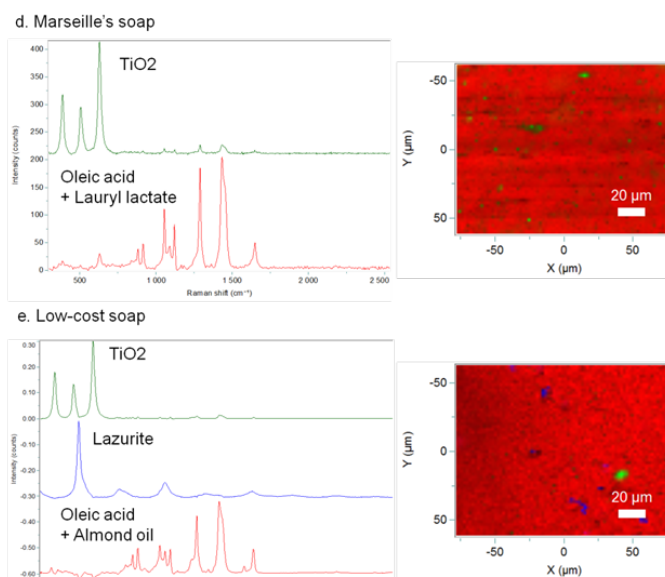


Figure 3: Raman reference spectra and Raman maps of different industrial soaps. Raman spectral identifications based on KnowItAll® databases. (d) Marseille's soap. (e) Low-cost soap.

Based on the Raman chemical analyses, it is clear that these soaps are not from handmade manufacturing. Indeed, the soap compositions are less homogeneous and some single compounds are found. Thus, in both Marseille's and low-cost soaps, TiO₂ was found. This compound is mainly used in the soap industry for its ability to lighten the color of the soap. In the case of the low-cost soap, pigment molecules are also observed (lazurite). All of these compounds poorly mixed confirm the industrial preparation of these soaps. Raman is a good way to evaluate the quality of preparation of soaps and to investigate the presence/absence of additives.

Another benefit is the ability of Raman spectroscopy to differentiate between the different oils used during the soap preparation.

Conclusion

To summarize, we have shown how Raman microspectroscopy can be a powerful technique to characterize soap homogeneity and to investigate the presence/absence of additives or traces of reagents in the final product.

