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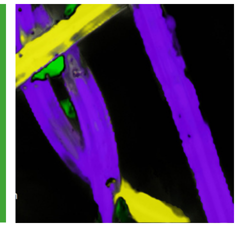
Raman Spectroscopy

Characterization of protective mask fibers by Raman microscopy



Application Note

Pharmaceutical RA81

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Abstract: Due to Covid-19, a wide choice of protective masks are now on the market. These types of masks are distinguished, in particular, by their different efficiencies linked to their filtration capacity. These differences in protective properties have therefore made it essential to characterize the composition of the masks. In this paper, we present why LabRAM Soleil™ confocal Raman multimode microscope is the perfect tool for studying the distribution and composition of mask fibers.

Keywords: Raman microscopy, fibers mask, compounds distribution, ViewSharp™, SmartSampling™.

Introduction

Due to the Covid-19 epidemic health crisis, wearing a mask is compulsory in public places in addition to barrier gestures and physical distancing measures. It is now part of our daily lives and helps to limit the spreading risk of the coronavirus. There are three main categories with different protective properties: Respiratory protective masks (FFP), surgical-type masks and textile masks. Given this wide choice of protective masks, the study of their chemical composition has proved essential. Indeed, the identification of the substances that make up the fibers of masks informs about what we expose everyday our skin and therefore our health. In addition, it is interesting to study whether their structure and their chemical composition have an impact on their respective filtration capacity. This application note presents how Raman micro-spectroscopy and more particularly Raman mapping have allowed the characterization and identification of the chemical compounds that constitute the masks.

Raman spectroscopy is a very powerful non-destructive and non-invasive characterization technique. It uses the inelastic scattering principle which is based on a phenomenon arising from a radiation-matter interaction and provides detailed information on molecular structures and chemical identification. The main advantage of this technique also comes from its capability to be applied to any sample, with no need for preparation.

In addition, the coupling of a Raman spectrometer to a confocal microscope makes it possible to perform surface or in-depth analyzes and achieve a spatial resolution of the order of μm^3. Confocal Raman microscopy is therefore particularly suitable for the investigation of a micrometric sample and is presented as a perfect tool for the study of mask fibers.

Instrument and methods

Our HORIBA LabRAM Soleil™ is a confocal Raman microscope offering the highest throughput in the market with no compromise on resolution. This is a consequence of the unique optical design of this microscope based on dielectric mirrors, with very low signal loss, coupled with high quality gratings, our main expertise. It is fully confocal, providing high image quality and spatial or depth resolution. These outstanding characteristics are required to obtain the best quality spectra needed for the discrimination, the analysis and the identification of the different types of fibers that make up a mask.

In order to differentiate the chemical composition of fibers, the integrated MVAPlus™ multivariate tool is the solution. Including multiple algorithms, MVAPlus™ is able to analyze up to 4,000,000 of spectra to find the major compounds of a sample and display their distribution. Additionally, ViewSharp™ from EasyNav™ package is also used to analyze 3D chemical visualization of the sample. Indeed, this tool based on the video image contrast, produces an image in which all surfaces are in focus simultaneously and creates a 3D topography image.

Furthermore, a considerable time savings have been achieved by SmartSampling™ image acquisition. Indeed, its functionality consists in selecting the acquisition points of a Raman map

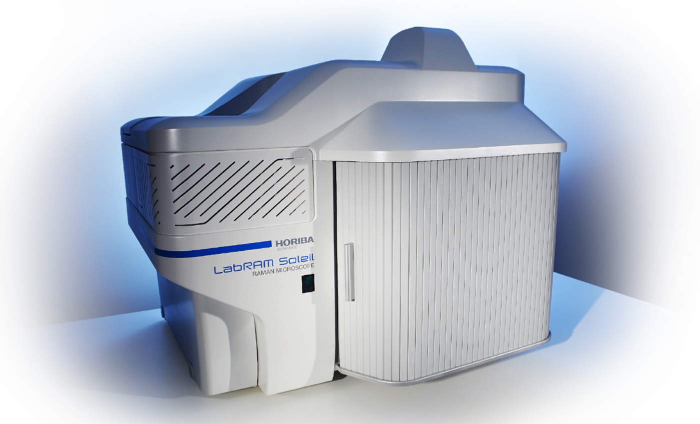

*Figure 1: HORIBA **LabRAM Soleil™** Raman microscope*

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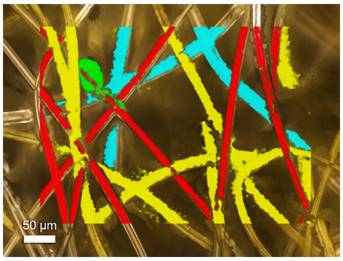

*Figure 2: Raman maps of "homemade" fabric mask. Raman spectral identifications based on KnowItAll® databases. Each color represents a specific compound. **Yellow:** Pigment yellow, **Cyan:** Polyester, **Red:** Cellulose, **Green:** Polypropylene + TiO_2*

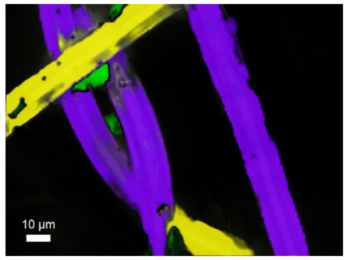

*Figure 3: Raman maps of mask with additive particles. Raman spectral identifications based on KnowItAll® databases. Each color represents a specific compound. **Yellow:** Cellulose +TiO_2, **Violet:** PET, **Green:** Additive particles.*

according to their relevance. Based on the video image of the sample, its algorithm will automatically split the video image in multiple area and define an optimum path to decrease the total mapping time. It intelligently combines precision and speed.

In order to compare the composition of the different masks available on the market, three samples were analyzed: surgical mask, a "homemade" fabric mask and a mask with additive particles.

Results

For each piece of mask, a Raman map was 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 calculated reference spectra. These spectra were then automatically identified thanks to the Raman KnowItAll® databases provided with LabSpec6™ software and powered by Wiley Science Solutions.

First the piece of "homemade" fabric mask and the mask with additive particles were analyzed and their Raman map are shown on Figure 2 and 3 respectively. It has been observed that the sample of “homemade” mask is composed of a mixture of fibers of Pigment yellow, Polyester, Cellulose and Polypropylene + TiO_2. As for the other mask (Figure 3) it is composed Cellulose +TiO_2, PET, and additive particles.

Table 1: Mapping time comparison between classical mappings and SmartSampling™ mappings

Type of mask	Classical pt-by-pt mapping time	SmartSampling™ mapping time
Homemade	20h	**4h (5x faster)**
Surgical	6h40	**20min (20x faster)**
With additives	51h	**31min (100x faster)**

Based on these analyzes, a complete and rapid characterization of fibers that make up a mask is carried out. Indeed, the use of the ultimate tool SmartSampling™ organizes and optimizes the measurement points to achieve the most precise and efficient Raman Imaging. SmartSampling™ is so a perfect tool to compromise between resolution and total mapping time, as reported in Table 1.

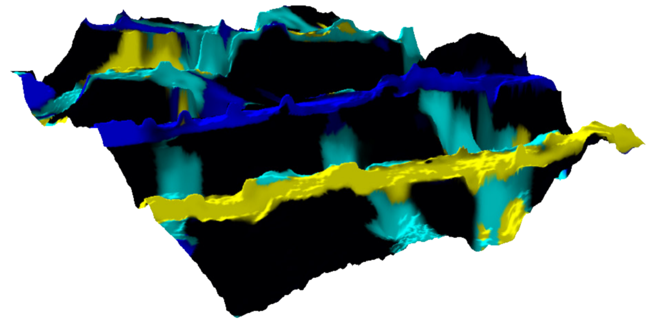

*Figure 4: Raman maps of classical surgical mask. Chemical compounds distribution overlayed on the topographical image resulting from ViewSharp™. Colors: **Yellow:** white fiber, **Cyan:** light blue fiber, **Blue:** Navy blue fiber.*

Then, 3D Raman mapping (Figure 4) was performed on the piece of surgical mask using the ViewSharp™ tool. The automatic focus adjustment based on the profile topography provided by this module is then correlated with the multivariate analysis in order to generate a highest quality 3D chemical visualization. ViewSharp's powerful ability to study rough samples such as a surgical mask fiber weave is illustrated in Figure 4.

According to the analyzes carried out, the protection / filtration capacity of the masks does not seem to lie in the multiplicity of polymers types used but in the quality of the polymer fibers selected.

Conclusion

To summarize, it has been shown how Raman confocal microscopy is an excellent technique for characterizing the fibers of different chemical natures that make up a protective mask. In this case, Raman spectroscopy offers a complete chemical identification based on spectra, correlated with high-quality information about spatial distribution.

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