

ラマン分光法を用いた道路建設資材の分析

道路建設に使用される固体材料を携帯型ラマン分光計で分析しました。分析対象は、 CaCO_3 、 TiO_2 、DEGALAN®などの従来型の顔料および樹脂です。測定されたスペクトルはそれぞれ大きく異なっていま

した。化学構造間の主な違いを評価するために、スペクトルのピークをそれらを生成した官能基に割り当てました。

INTRODUCTION

Different materials such as paints, (white) pigments, and resins are commonly used in the construction of roads. Together with glass beads (used for night-visible road marking) and various other materials, they help us to get safely from A to B.

In this study, different roadmaking materials

were analyzed using the Mira M-1 handheld Raman analyzer. The gathered spectra were compared with each other to see the main differences in the functional groups. The analysis proved that Mira M-1 is suited for the differentiation of such materials.

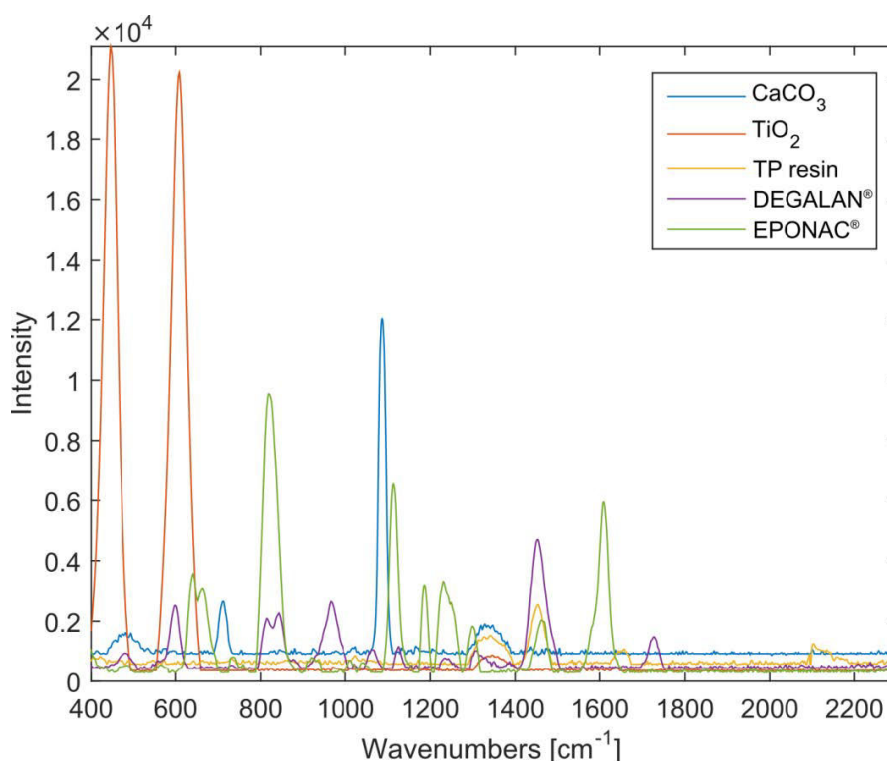


Figure 1. Overlay of spectra of different roadmaking materials

EXPERIMENTAL

All spectra were measured using the Mira M-1 Raman spectrometer in auto-acquisition mode, i.e., integration times were determined automatically. A laser wavelength of 785 nm and the Orbital-Raster-Scan (ORS) technique were applied. The measurements were done in small sample vials with the vial holder adapter.

The following samples were investigated:

1. Chalk (CaCO_3)

2. Titanium dioxide (TiO_2)

3. EPONAC® resin

4. TP resin

5. Pigment yellow

6. Pigment blue

7. Pigment red

8. DEGALAN® resin

RESULTS AND DISCUSSION

The measurement of calcium carbonate furnished a clear spectrum with two main peaks at 712 cm^{-1} (symmetric O–C–O bending

vibration) and 1087 cm^{-1} (symmetric stretching vibration).

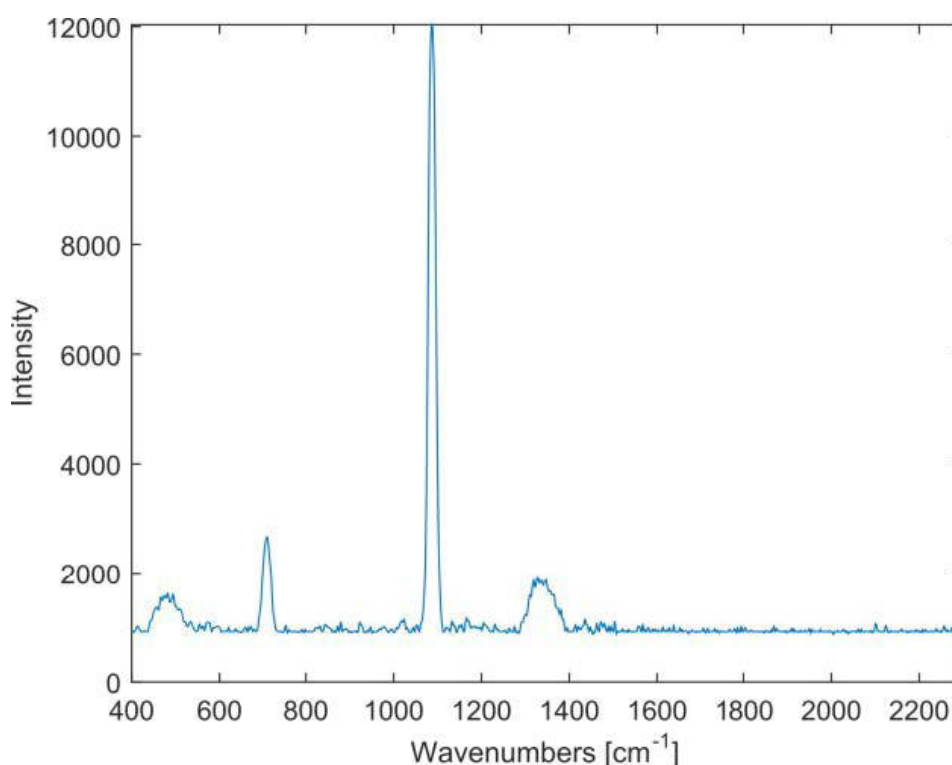


Figure 2. Spectrum of calcium carbonate.

Looking at the titanium dioxide spectrum, one can see two main peaks which give information on the present crystal modification (rutile or

anatase). Two peaks represent rutile, while three peaks are typical for anatase because of its crystal symmetry.

Both peaks are symmetric stretching vibrations and belong to O–Ti–O (446 cm^{-1}) and Ti–O (609 cm^{-1}). In the case of anatase, the peak at 446 cm^{-1}

¹ is split in two. This makes it easy to differentiate between rutile and anatase.

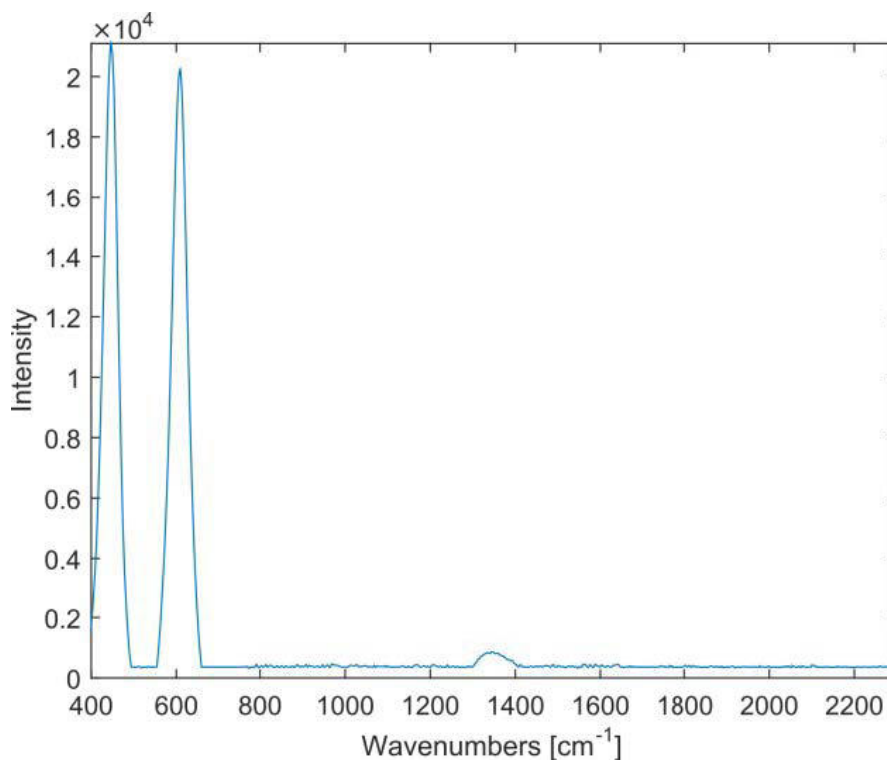


Figure 3. Spectrum of TiO₂ (rutile).

A glance at the spectra of typical resins such as EPONAC® or DEGALAN® reveals nicely separated

peaks which can be assigned to their corresponding functional groups (see below).

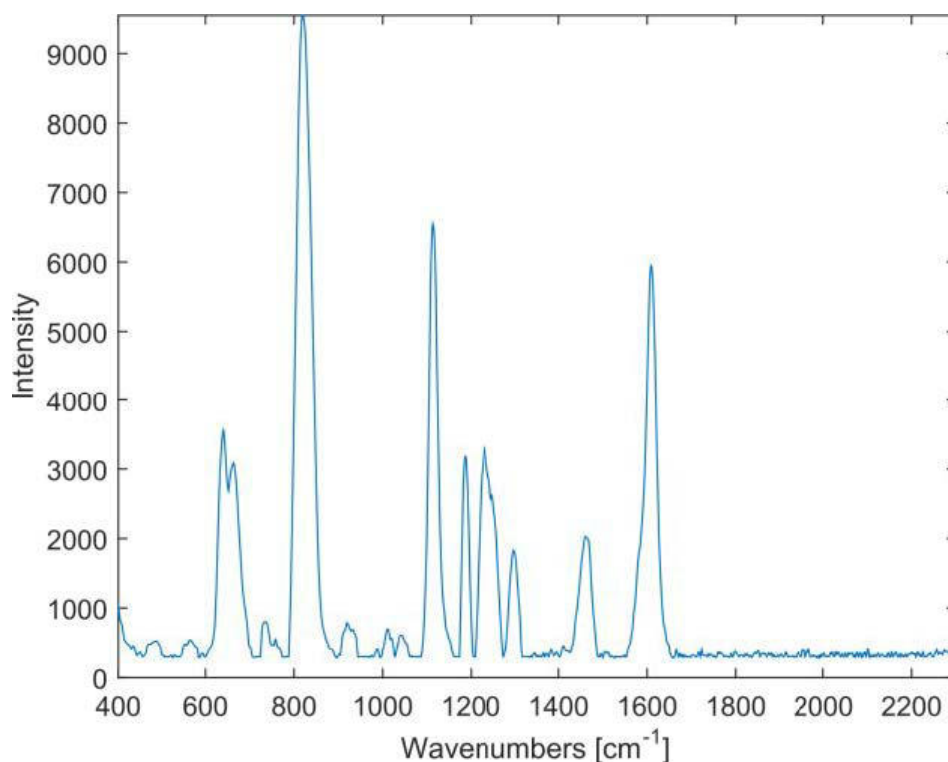


Figure 4. Spectrum of the EPONAC® resin.

Table 1. Peaks observed in the EPONAC® spectrum.

Peak [cm ⁻¹]	Description
640	Cyclic vibration (para-substituted benzene)
819	C–H bending vibration (para-substituted benzene)
1000	Various C–C stretching vibrations
1113	C–OH stretching vibration
1189	C–(CH ₃) ₂ stretching vibration
1231	C–O
1248	C–H (benzene)
1298	–CH ₂ – twisting vibration
1461	–CH ₂ – bending vibration
1609	C=C

The peaks are very characteristic for para-substituted benzene and confirm that EPONAC® is a copolymer of bisphenol A (BPA) and another component.

When comparing the EPONAC® spectrum with the DEGALAN® spectrum (see **Figure 5**), it is

obvious that the peak of the benzene at 1600 cm^{-1} is missing. The peak around 1700 cm^{-1} , together with the peaks slightly below and above 1200 cm^{-1} , is characteristic for carbonyl groups. In addition to that, the C–C peaks are more distinct for EPONAC® than for DEGALAN®.

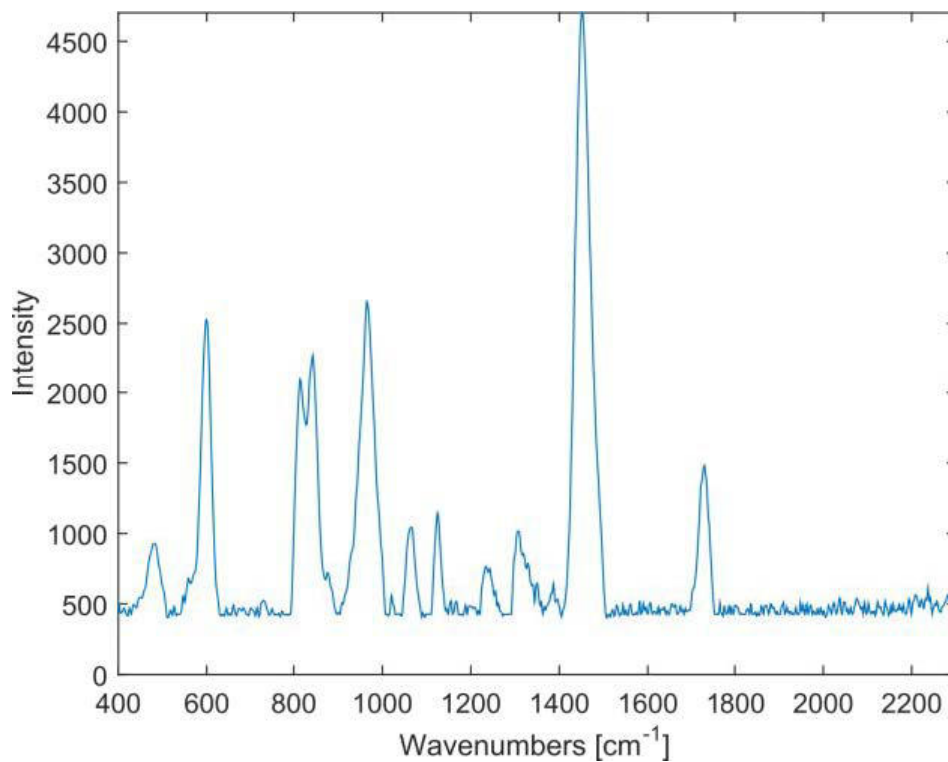


Figure 5. Spectrum of the DEGALAN® resin

Table 2. Peaks observed in the DEGALAN® spectrum.

Peak [cm ⁻¹]	Description
599	–COO bending vibration
814	Propionate bending vibration
843	C–CH ₃ stretching vibration
965	C–C stretching vibration
1065	C–COO stretching vibration
1125	C–O
1234	C–O
1308	–CH ₂ – twisting vibration
1452	–CH ₂ – bending vibration
1728	C=O carbonyl group, ester

Using these differences, it is easy to discriminate between the resins and also between resins and

pigments (see **Figure 6**).

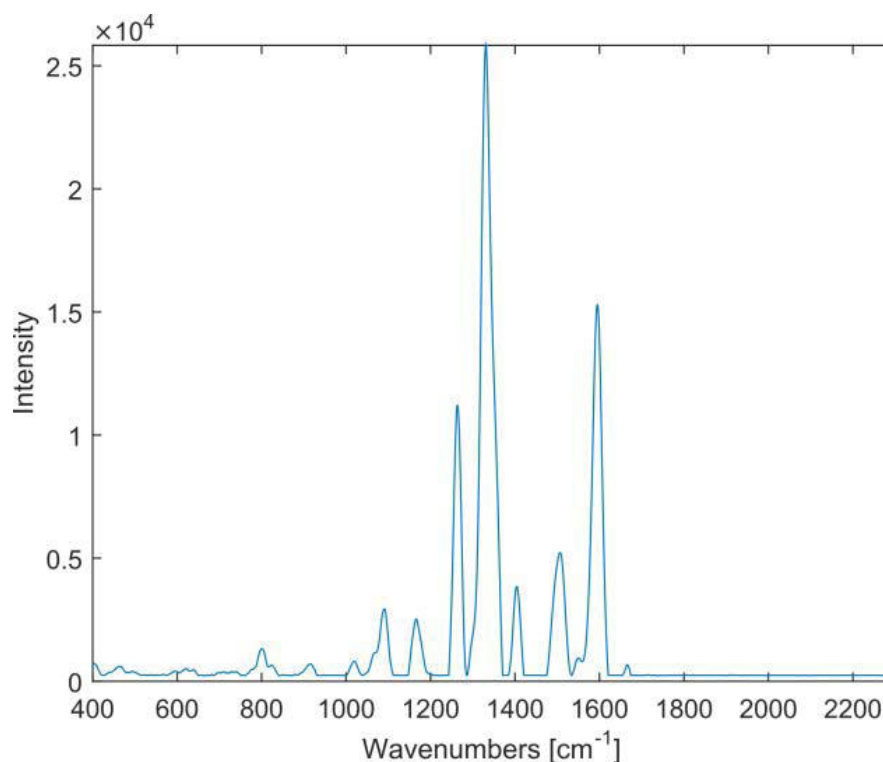


Figure 6. Spectrum of the yellow pigment «Hansa®-Brilliantgelb»

CONCLUSIONS

Because of the great differences in their spectra, handheld Raman spectroscopy is ideally suited for the analysis of materials used in road construction. The investigation of the spectra

showed that there are significant differences in the functional groups of the materials, thus allowing the identification with handheld Raman systems such as Mira M-1.

CONTACT

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CONFIGURATION



MIRA P Advanced

Metrohm Instant Raman Analyzer (MIRA) Pは、迅速な非破壊的計測および薬品有効成分や賦形剤などの様々な物質の検査に使用できる、高性能な携帯型ラマン分光計です。サイズはコンパクトですが、MIRA Pは非常に堅固で、弊社独自の軌道ラスタースキャン技術 (Orbital Raster Scan Technologie, ORS) を備えた作業効率の高い分光技術構造を有しています。MIRA PはFDA規則 21 CFR Part 11の要件を満たしています。

Advanced Packageには、物質を直接、またはオリジナル容器で分析することが可能なアタッチメントレンズ (レーザークラス3b)、およびガラスバイアル中のサンプル分析のためのバイアルホルダーアタッチメント (レーザークラス1) が含まれています。