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Application News

FTIR-spectroscopy

Destroying free Analysis of Asbestos in old materials -Floor plates in the focus of FTIR and EDX

No. SCA-110-114

User Benefits

- Fast screening of samples
- Analysis space minimal
- Quick handling using standard libraries

▪ Introduction

Asbestos a never ending story about mineral fibres which are banned because of generating cancer in human body.
When is was found as flame retardant it was the best filler in diverse construction materials. Very often used from 1950ies to 1970ies. One example for indoor was the usage of it in linoleum. Inside of linoleum it was embedded into polymer and not a free fibre. As time goes by most of owners from buildings with floor plates doesn´t remember or are new owners and do not simply know about the content of such linoleum plates. As the reconstruction companies know about such plates they have to check the quality of such material before wasting it. The handling of asbestos fibres is under national controls and strongly regulated[1]. Since 2003 (directive 2003/18 / EC), there has also been a ban on the use of asbestos or materials containing asbestos at European level. And since 2005 it is rule for all EU members. But before rules take effect the material must be identified.
Highly recommend is to use the FTIR for the identification of such substances, or the identification of fibres with stereo microscopy or composition via elemental distribution analysis.
With this application will be shown what is possible without any chemical treatment. The both techniques FTIR-ATR and elemental analysis, with the EDX (Energy Dispersive X-ray), are the working tools for this. The Asbestos fibres here in this application note were embedded or fixed fibre. No free asbestos fibres, fibres in a matrix.

What is Asbestos by nature? It is a group of silicates building crystals in fibre form. In combination with other elements they form different minerals like chrysotile, crocidolite, amosite and tremolite for example.
The identification of them can be done with help of the stereo microscope or with infrared spectroscopy.[2]

For this application vinyl asbestos floor plates were in focus found in a building from the 1960ies.
Presence of asbestos makes the re-construction of the floor expensive and time consuming.

Figure 1: On top the picture of typical floor plates from today, below the view to the edge of an old floor plate with Asbestos fibres.

A quick analysis with the Shimadzu FTIR in combination with ATR or EDX system can be done with the fibres or parts directly, destroying free in manner of no chemical treatment. The usual procedure is chemical treatment, burn it down to ash and after the residual particles measured as KBr-pellet- the classical infrared preparation technique. [3]

- **FTIR-ATR**

To get a quick overview about materials the FTIR method is the right choice. Within seconds you can identify the material. As it is the talk about solids which will be tested without any chemical treatment, just a piece as it is. The accessory for the analysis of the solid is the single reflection ATR technique with diamond window. For a good ATR measurement, the polymer must be soft so that it can be pressed homogenously over the complete measurement area against the diamond window.

- **EDX Method**

The EDX sample compartment is designed to cover huge samples. With a camera and collimator system an area of the sample can be selected. With a strong energy Xray light source analysis can be done on the surface and partly inside of the sample. By the hardness of the energy X-ray fluorescence occurs and can be detected. The analysis is sensitive for ppm and percentage concentrations of elements, all depending on the matrix of the sample and the characteristics of an element under the Xray beam..

- **Analysis with FTIR**

In this case of FTIR-ATR analysis the match to the asbestos was found just by random. It is not to forget that the ATR technic is a surface analysis with a penetration of approx. 2µm into the sample surface. This analysis showed that the top measurement reached not the under layer with asbestos. Even the PVC was overlayed by other top layer based on acrylate, might be a gloss effect for the floor plate appearance. Only the bottom analysis at a position cleaned from glue gave access to asbestos fibres. Analysis was done following the determination table [1].

The manual for product safety shows a chart for determination from asbestos fibres. For any trial it is to point out, that the selection table is for the pure mineral and an orientation for decision about which asbestos is present. In this case the material including asbestos fixed the fibres in polymer PVC and filler $CaCO_3$ (as whitener and amount of TiO_2) material.

Asbestos was found in a section from the bottom of the linoleum plate, a position which was glue free.

Direct fibre analysis was not successful because of too many other fibres in fibre mix. The infrared spectrum was representing non asbestos material, it was cellulose like materials. Such materials were used for the sound proofing of floor plates.

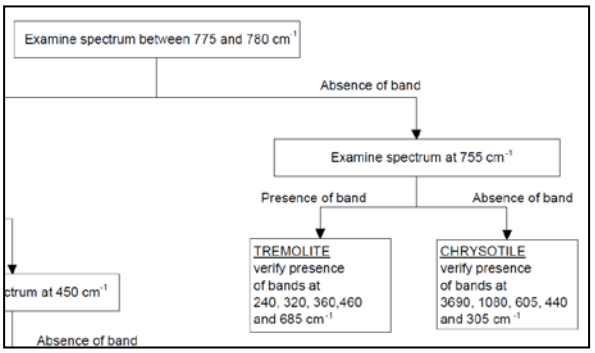


Figure 2: cut out of [1]. The way of exclusion procedure to find chrysotile.

- **Analysis with EDX**

The EDX measurement was successfully detecting the asbestos composition of elements as well as the major polymer the PVC because of its high chlorine content. Even the relation between the organic and inorganic materials was found in realistic distribution because in the past it was usual to spare expensive polymer against fillers. Refer to the content of organics in table 1.

The analysis was done with EDX fundamental parameter set.

- **Combination of EDX and IR**

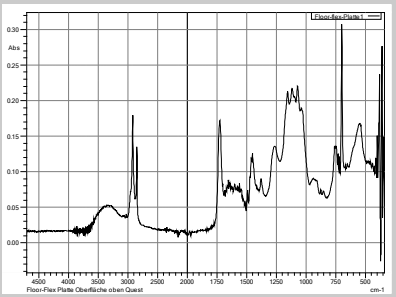
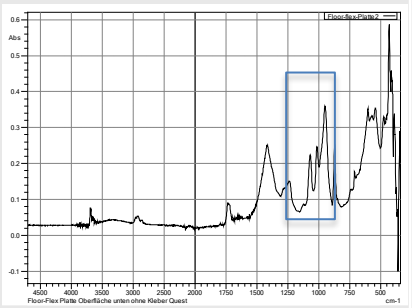
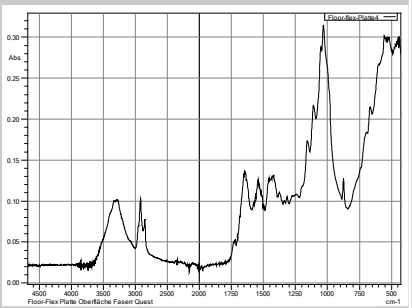
In this case the combination of both technics gave a good match for the asbestos. The challenge here is the destroying free analysis.

Natural the sample must open the access to fibres. The penetration to the surface of the FTIR was not possible to detect the asbestos fibres which were arranged under additional surface layer. Only the view inside of the material made it possible.

Important was that no chemical treatment was necessary. Natural it is also chemical treatment possible. [3]

Once isolated the fibres can be easily identified via determination table (fig. 2) or with the support of an asbestos library [4].

- **Analysis table with EDX and FTIR results for Floor-Flex**

Position at Floor-Flex plate	EDX-Result	IR-spectrum	Remark
Top	Analyte / Result Cr 0.0822 Pb 0.0450 Cl 5.3411 Ca 4.5031 Ti 0.9853 Si 0.8378 Zr 0.0005 CH2 87.5365		EDX: filler Ca, Si based, high organic and chlorine indicates PVC FTIR: 1. Acrylate, 2. Most nearest PVC2 as MIX of PVC and terephthalate
Inside	Analyte / Result Ca 13.1812 Cl 10.5372 Mg 4.5516 Si 3.2155 Ti 2.7803 Fe 1.0677 Cr 0.1919 Pb 0.1080 K 0.0912 Mo 0.0023 CH2 64.1718	FTIR: without glue 	EDX: shows Chlorine and organics which is PVC, Mg and Si, Fe are belonging to asbestos and Ca as well as Ti are typical for filler presence in polymers. FTIR: 1. $CaCO_3$ 2. Chrysotile by three positions [1], 3691, 1070 and 605cm^{-1} after subtraction of $CaCO_3$ filler 3. Hits in libraries for chlorine, and Al/Mg silicate salt 4. At all the group around 1000 -1100cm^{-1} is high match to chrysotile mineral.
Under or Bottom	Analyte / Result Cr 0.1547 Pb 0.0843 Ca 10.1105 Cl 8.0622 Si 2.4710 Ti 2.0131 Fe 0.8104 P 0.5112 Zr 0.0008 CH2 75.6788	FTIR: diverse fibers from the sample edge 	EDX: The under measurement resulted in similar element distribution as it was seen on the top of sample. FTIR: The fibre selection (Fig. 1) could not give a clear match. The spectrum is a mix of diverse materials. The control signals for chrysotile were not found. The subtraction analysis showed: $CaCO_3$ small content, cellulose fibre and match to protein substructure.

- **Result**

The EDX analysis showed directly the composition of an Asbestos and PVC.

With FTIR it was not that simple because the technique of the FTIR-ATR allows a penetration of the analysis beam into the surface of approx. 2µm at 2000cm-1. As the asbestos fibres were fixed by polymer the thickness of the polymer made it partly impossible to see the fibre. Only at thinner amount of polymer and filler (CaCO3) it was possible to detect directly asbestos. The Asbestos in this case was the Chrysotile.
The FTIR measurement were done with the standard DLA-TGS detector. In case of the asbestos it is also a trial worse to use the FTIR equipped with CSI detector for better access to FIR range. The asbestos have some important signals for identification in the FIR region.
In this case the material was handled as fast as possible, with all protection clothes to avoid any contamination.

For the correctness of the analysis the sample was as well analysed from a certified institute. And they found the Asbestos using higher class equipment from the microscopy (phase contrast, electrone) or Xray diffraction.

- **The Package EDX and FTIR**

❑ *Main Unit*
IRTracer-100: Fourier Transform Infrared Spectrophotometer compact, speedy and stable, with DLA-TGS Detector and KBr-window

❑ *Accessory*
QATR-10 with diamond window and fixed anvil pressure
Slicer

❑ *Software and Libraries*
LabSolutionsIR and integrated application
Spectral Database Mineral and Clays, Wiley
EDXIR Library tool

- **Conclusion**

For a fast screening the standard equipment EDX in combination with FTIR can be a good solution. Results can be available with in 15 minutes for both analysis equipment and their analysis time frames together.

For the purpose of library it is possible to combine both results in the EDX-IR library for such dedicated material like asbestos in complex appearances- floor-flex plates, or asbestos cement. [5]

- **Literature**

[1] EU Directive 2009/148 / EC on the protection of workers from the risks related to asbestos at work (formerly: Directive 83/477 / EEC)

[2] "Detection and identification of various asbestos fibres in consumer products", Health Canada, Product safety reference manual, Book 5- Laboratory Policies and Procedures, Part B. Test Method Section, method C-26, 2003

[3] "Solid Analysis – Analysis of mineral fibres in water, soil and waste material using FTIR spectroscopy", Zeno Marco Sedin et al., ARC, Shimadzu News 2/2007

[4] Wiley, Mineral and Clays, Spectral Database Index - IR - Sadtler Minerals & Clays - Wiley (knowitall.com)

[5] Analytics of thermally treated asbestos cement, M. Mattenklott, Gefahrstoffe – Reinhaltung der Luft, 64(2004) Nr. 11/12 – Nov./Dez., 480-484

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