Defence

TNO | Knowledge for business

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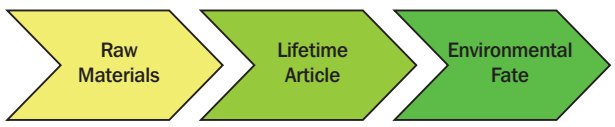
From Cradle-to-Grave

Chemical Analysis During Lifecycle

Chemical analysis is one of the most important parts within an energetic material lifecycle. It starts with the qualification and quantification of the raw materials and/or the intermediate materials used for the manufacturing of the final product. When the product is finished there is quality control and finally after functioning or demilitarisation components will end-up in the environment.



The chemical analysis of the raw material is important to establish information on the purity of the material, the size and shape of the particles and sometimes on the composition (for example the nitration degree of nitrocellulose).
During the lifecycle the interest lies in the changes of the different components of the article due to degradation, migration or reactions between components, which can result in malfunctioning.

At the end of the lifecycle, after functioning of the article, components will enter the environmental chain. The analyses performed here are in general focused on the presence of environmental unfriendly and/or toxic component left in the soil, sediment or water.
For all these steps it is important to choose the right analysis technique for the right questions. In this poster some of the analysis techniques used by TNO are presented.

Techniques

For the metal-salts analysis X-ray diffraction (XRD) and electron microscopy (SEM) with x-ray micro-analyse (RMA) are the techniques to use. With SEM the particle structure can be identified and with RMA the elemental information. For information about the components present, XRD-analysis is used; however this technique is limited to crystalline components.

The organic components can be analysed with liquid chromatography (LC) or gas chromatography (GC) optional coupled with a mass spectrometer (MS). The materials have to be dissolved to be able to analyse it with chromatographic techniques.

For a material such as polymer bonded explosives (PBX), which is not dissoluble, it is possible to try to use attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR). This is a form of IR where solid materials are pressed onto a crystal where the IR-bundle is send through. The beam enters the sample for a few micrometers and the reflection is send to the detector. It is a surface technique and not able to deal with inhomogeneous samples.

Another important parameter is the determination of the water content. Water is often the cause of degradation. Karl-Fisher is for this purpose the right technique.

Examples
Raw materials

Nitrocellulose is a material containing multiple parameters which can influence the processability and the performance of the end-product, including chain length and nitration degree. Size-exclusion chromatography (liquid chromatography based on the separation on hydrodynamic volume) is used to obtain an indication of the chain length/molecular weight and its distribution (Figure 1).

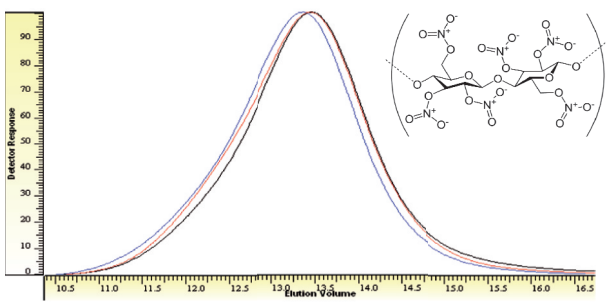


Figure 1: Nitrocellulose sample with different hydrodynamic volume and different distributions.

The retention volume is calibrated with polystyrene standards and for this reason the determined value is corresponding to molecular weight of polystyrene.

Ageing

For ageing studies of pyrotechnic components XRD can provide information if the oxidation of metal compositions did take place. From a diffractogram a library is fitted and from here the components are obtained. To visualize the ageing, SEM is the technique. Cracking or broken crystals can be detected in materials (figure 2).

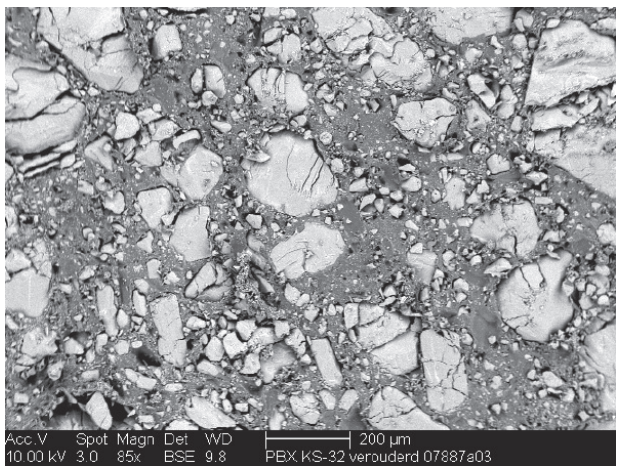


Figure 2: SEM-picture of cracked crystals.

Environmental fate

After extraction from the environmental media the analysis (Figure 3) with LC(-MS) can be performed. Not only can this provide qualitative information about the organic energetic components, additives and degradation products, but also quantitative information.

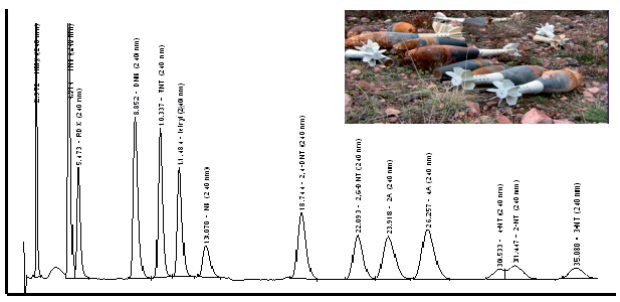


Figure 3: LC-separation of energetic materials in a soil sample.

Identification of unknown peaks can be solved by the MS or in combination with infrared spectroscopy.

Conclusion

For every step in the lifecycle of materials, there are techniques to conform or identify the material composition. Especially the combination of techniques can help to understand different processes, from synthesis till degradation. The importance is to keep in mind the limitations of every analysis and every technique.

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