COUPLED SEM-EDS-RAMAN: A COMPLEMENTARY APPROACH FOR CHARACTERIZATION - APPLICATION TO GEOMATERIALS



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Introduction

The combination of elemental and crystallographic analysis is frequently required to identify and describe natural and synthetic materials. Physical and Chemical characterization requires a set of analytical spectroscopic and/or imaging techniques to access a thorough knowledge of the sample, to better understand the structural, chemical, and textural factors that control their properties, their behavior, and their reactivity... As an example, a mineral description often requires chemical information (Major, minor, and trace elements), structural information (crystal structure, crystallinity...), and morphological information (crystal shape and size).

Among these techniques, Scanning electron microscopy (SEM) and Raman spectroscopy occupy a major place among the characterization techniques and have often been combined in many studies. SEM is a microscopy technique that provides various information on morphology, topography, and chemistry at a nanometric scale, supplemented by electron beam-based techniques providing chemical or crystallographic information, like Energy-dispersive X-ray Spectrometry (EDS), Wavelength-dispersive X-ray Spectrometry (WDS), Electron Backscattered diffraction (EBSD) or cathodoluminescence (CL). Micro-Raman spectroscopy is also widely used in the organic or inorganic materials characterization at a micrometric / sub-micrometric scale. The spectrum is unique for a given compound. It can provide details on the chemical and structural properties (functional groups, lattice defects, crystallinity, etc.). Raman offers various information, which includes, among others, the identification of polymorphs, crystallographic orientation, evaluation of temperature, and stress effects on the structure. Moreover, micro-Raman spectroscopy provides complementary chemical and structural information to that obtained from SEM-EDS. Then, both are often used in parallel.

Switching from one to the other is in some cases considered a problem that cannot be solved using common technical solutions (precise location, identification of particles...). Coupling SEM-EDS and confocal Raman imaging on the same analytical system is an efficient method for the advanced physicochemical characterization of materials and the determination of structural properties of materials at the micro/submicrometric scale.

The technical aspects of coupling Raman and SEM in a unique system are first described, as well as the practical aspects and limitations inherent in this type of coupling. The interest of the coupling will be illustrated by various examples of application to geomaterials and mineralogy, including cultural heritage materials, and naturally occurring asbestos, for which this coupling makes it possible to solve problems for characterization.

[1] Wille G. et al 2014 Micron 67 50–64

[2] Wille G. et al 2014 in: Spectroscopic Properties of Inorganic and Organometallic Compounds: Volume 45, 79–116 (ISBN 978-1-84973-919-1 - DOI 10.1039/9781782621485) (Ed. The Royal Society of Chemistry)

[3] Wille G. et al 2018 in: Springer series in surface science 66 Confocal Raman microscopy 2nd edition, 559-580. (ISBN 978-3-319-75378-2 - DOI 10.1007/978-3-319-75380-5) (ed. Springer)

[4] Wille G. et al 2019 J Hazard Mater 374 447–458