INTERNATIONAL CONFERENCE OF LITHUANIAN SOCIETY OF CHEMISTRY

Theodor v. Grotthuss

Dedicated to 210th anniversary of
publication of the first theory of electrolysis
proposed by

THEODOR GROTTHUSS
(1785 - 1822)

LITHUANIAN ACADEMY OF SCIENCE
Gedimino Str. 3, Vilnius, Lithuania
April 28-29, 2016

BOOK OF ABSTRACTS

CHEMISTRY & CHEMICAL TECHNOLOGY
CONFERENCE 2016 VILNIUS

HOW STATISTICAL METHODS GUIDE THE SELECTION OF THE FTIR METHOD

A. Brangule[12], K. A. Gross[1]
1 Dept. of Human Physiology and Biochemistry, Riga Stradiņš University, Dzirciema 16, LV-1007, Riga, Latvia
2 Biomaterials Research Laboratory, Riga Technical University, P.Valdena 3, LV-1048, Riga, Latvia
E-mail: agnese.brangule@rsu.lv

Fourier transform infrared (FTIR) spectroscopy is an important tool in the analysis of calcium phosphate. The most commonly used methods are Attenuated total reflectance spectroscopy (ATR) and Transmission FTIR spectroscopy with KBr pellet (FTIR KBr) less frequent - Diffuse reflectance FTIR spectroscopy (DRIFT) and Photoacoustic spectroscopy (PAS). FTIR methods are used for both qualitative analysis and quantitative analysis, for example, the crystallinity index (CI) and estimation of the relative CO_3^{2-}, OH^- and HPO_4^- content.

The main task of this study was to identify advantages and disadvantages of each method in the research of carbonate containing calcium phosphate (CP) powders with different degree of crystallinity.

All four FTIR methods are nondestructive and useful to analyze powders and raw solids, but the best analysis method is not determined by the equipment on the bench of the laboratory, but by the most information provided by the appropriate method.

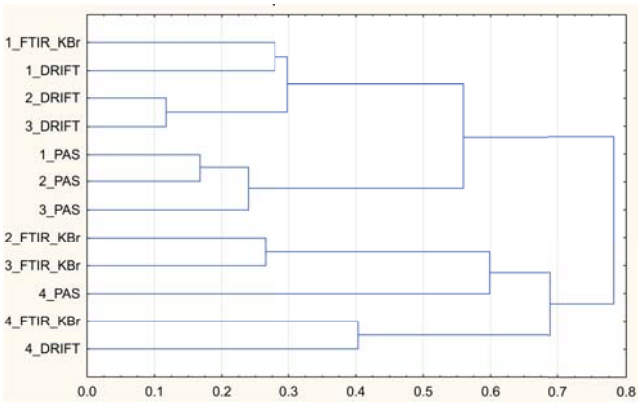

Figure 1. Chebyshev tree diagram

The statistical analyses of the spectra recorded from the CP powders involved 3 steps:

1. Preparation of carbonate containing CP powders with different level of crystallinity (crystallinity evaluated based on the XRD analysis and Rietveld method).
2. Parameterization of spectra: normalization and baseline correction; deconvolution of spectra in υ2, υ3 CO_3^{2-}, υ4 PO_4^{3-}, υL OH^- band region to enhance the resolution and selecting a spectral parts rather than using whole spectrum for analysis.
3. Statistical analysis, such as Principal component analysis (PCA), Factor analysis (FA), Pearson product-moment correlation coefficient (PPMCC) and Cluster analysis (CA). Cluster analysis uses Euclidean and Hierarchical methods.

Using statistical methods, we found that the parameters of all four methods are most consistent when we analyzed amorphous or microcrystalline samples. The biggest differences showed crystalline powder. This led to the conclusion that the analysis of such samples is essential both in the choice of methods and in information on the degree of crystallinity. By analyzing both the entire IR spectrum and specific parts of spectra, the greatest differences showed ATR method. This is explained by the low sensitivity of this method in CO_3^{2-} OH^- and H_2O.

References

1. I. Rehman, W. J. Bonfield, Mater. Sci., **8** (1997) 1-4.
2. J. Li, D. Brynn-Hibbert, S. Fuller, and G.Vaughn, Chemometr. Intell. Lab. Syst., **82** (2006) 50-58.