

MEDICAL UNIVERSITY OF LUBLIN

FACULTY OF PHARMACY



Justyna Kawka

**Application of selected separation techniques  
in the analysis of organic and inorganic ions  
in environmental samples, body fluids, and tissue homogenates**

**SUMMARY**

Doctoral dissertation carried out in the Department of Analytical Chemistry  
of the Department of Chemistry of Medical University of Lublin

Supervisor : prof. dr hab. Jolanta Flieger  
Auxiliary supervisor: dr Małgorzata Tatarczak-Michalewska

**Lublin 2021**

The development of new methods for the determination of small molecules in biological fluids such as serum, urine, saliva or cerebrospinal fluid is becoming more and more difficult as the sensitivity and robustness of the analytical method need to be improved. To ensure easy availability, new methods should use apparatus that is standard equipment in most laboratories. An additional goal should be to ensure conditions that meet the criteria of the so-called "Green chemistry", consisting in limiting the consumption of volatile, toxic organic solvents. Researchers should also pay attention to waste products or products that are difficult to dispose of in order to find new uses for them.

The aim of this study was to develop new methods for the determination of inorganic anions in biological and environmental samples with the innovative use of the IAM column and to indicate the possibility of using waste materials in extraction techniques.

In the presented work, the following research directions can be distinguished:

- I. Testing of environmental samples and body fluids on the IAM.PC.DD2 Regis HPLC Agilent Technologies column for inorganic ion content:
  - A. determination of  $I^-$  ions in mineral waters and  $I^-$  and  $Br^-$  ions in medicinal salts,
  - B. determination of  $NO_3^-$  and  $NO_2^-$  ions in human saliva samples,
  - C. determination of  $SCN^-$  ions in samples of tobacco smokers' saliva, e-cigarettes, and non-smokers.
- II. Testing of plant tissue homogenate on a Zorbax Eclipse XDB-C18 Agilent Technologies column
  - Testing the sorption properties of selected plastics (polyvinyl chloride, polypropylene, polyethylene) for the selective isolation of chlorophyll from spinach leaf extract.
- III. Comparison of sorption properties of natural and synthetic zeolites on the example of sorption of selected metals from acidic aqueous solutions using the AAS technique
  - Sorption of chromium, manganese, selenium, nickel, cobalt and iron ions from acidic solutions under batch and dynamic conditions on natural and synthetic zeolites.

#### IV. Examination of the body fluid with the ICP-OES technique

- Assessment of the elemental composition of the aqueous humor of the anterior chamber of the eye in patients with cataracts after microwave mineralization.

Developed methods for the determination of iodide anions in water samples and environmental, nitrates (III), nitrates (V), and thiocyanates in biological fluids, on the IAM column, are effective, are characterized by a short analysis time, favorable detection limits and follow the trend of the so-called "Green chemistry", thanks to the use of aqueous salt solutions as eluents.

The study showed that polyvinyl chloride (PVC), thanks to the formation of stable molecular complexes with chlorophyll, can be used to extract green dye from biomass. In practice, the extraction efficiency was at the level of 98% in terms of chlorophyll A. There is also the possibility of multiple uses of the polymer thanks to the developed regeneration conditions.

It was confirmed that natural and synthetic zeolites have different sorption capacity for Cr, Mn, Se, Ni, Co, and Fe ions from acidic aqueous solutions. A procedure was proposed for the elimination of selenium in the form of an oxoanion from aqueous samples using a synthetic Na-X zeolite loaded with iron ions. Due to the use of cheap sorption materials, the method can be used on a larger scale to remove Se from aqueous samples.

The presented results of the analysis of the elemental composition of the aqueous humor of the anterior chamber of the eye in cataract patients confirm the accumulation of toxic elements, i.e. thallium, tellurium, cesium, lead, aluminum, cadmium, bismuth. The statistical analysis of the obtained results confirmed the existence of 5 independent groups of elements, occurring at various levels of concentration. The resulting division has clinical significance and sheds new light on the causes of this disease. It can also be a benchmark for future research.