

Green analytical chemistry and its perspectives in Bulgaria

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The concept of green chemistry emerged in the 1990s with the aim of minimizing the environmental impact of chemical activities. The basic principles to which a chemical procedure should correspond in order to be recognized as environmentally friendly (green) are reviewed. These include reduction of reagent and solvent usage, minimization of solid, liquid and gaseous materials, produced by the processes involved, reduction of energy and water consumption. Special emphasis is put on the features of green analytical chemistry, the challenges to the introduction of green principles to analytical laboratories, the assessment of the environmental impact and the waste management. The present state and the perspectives of green analytical chemistry in Bulgaria are discussed with an emphasis on the contributions of Bulgarian researchers in this field.

Key words: green analytical chemistry, basic principles, perspectives in Bulgaria, review.

This article is dedicated to the memory of Professor Dr. Nikolay Jordanov on the occasion of his 90th anniversary.

SUSTAINABLE DEVELOPMENT AND GREEN CHEMISTRY

In 1987 the World Commission on Environment and Development defined sustainable development as a form of development that meets the needs of the present generation without compromising the ability of future generations to meet their own needs [1]. One of the major goals of sustainability is to maintain an optimal balance between increases in manufacturing output, and a clean and safe environment. In the 1990s the concept of green chemistry emerged with the aim of minimizing the environmental impact of chemical activities. In their book “Green chemistry” published in 1998 [2], P. Anastas and R. Warner defined the principles to which a chemical procedure should correspond in order to be recognized as environmentally friendly. These include reduction of reagent and solvent usage, minimization of solid, liquid and gaseous materials, produced by the processes involved, reduction of energy and water consumption. Environmentally friendly (green) chemistry has received widespread interest in the past two decades due to its ability to harness chemical innovation to meet economic and environmental goals simultaneously [3–7].

Green chemistry embodies two main

components. First, it addresses the problem of efficient utilisation of raw materials and concomitant elimination of waste. Second, it deals with the health, safety and environmental issues associated with the manufacture, use and disposal or re-use of chemicals. The activities in the area of green chemistry should meet two goals [1]:

- teaching of basic aspects of environmental science at all levels of education, resulting in the production of specialists, capable of handling and solving existing and potential environmental problems;
- creating a fresh approach to typical chemical activities, leading to the environmentally more friendly use of facilities.

GREEN ANALYTICAL CHEMISTRY

An important part of the green chemistry philosophy is the need to develop and adopt green analytical techniques and procedures. Analytical chemistry takes a special place in the green chemistry concept. It is aimed to detect and quantitatively determine various substances by means of methods which often use harmful reagents. As a result, the analysis itself may become a source of pollution. Analytical chemistry is considered to be a small-scale activity, but this is not always true in the case of controlling and

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monitoring laboratories with a large number of runs performed. The use of instrumental methods instead of wet chemistry; the miniaturization and automation are the new trends of analytical chemistry, making this branch of chemistry more sustainable [8]. The determination of a broad spectrum of analytes at low concentrations (ppb, even ppt) in samples of complex matrix composition has been facilitated by the introduction of a new generation of highly sensitive analytical devices and by the development of new sample preparation procedures. The main features of green analytical chemistry are [9]:

- elimination or significant reduction of reagents, especially toxic substances and organic solvents from the analytical procedures;
- reduced emissions of vapors and gases, as well as liquid and solid wastes, generated in the analytical laboratories;
- reduced labor, energy and water consumption of the analytical procedures;
- reduced time between sampling and obtaining of the desired information about the sample.
- The challenges to the introduction of sustainable development (green) principles to analytical laboratories are related to:
 - preferable use of direct methods of analysis;
 - simplification, intensification and acceleration of the sample preparation procedures;
 - miniaturization, integration and automation of the analytical systems;
 - assessment of the environmental impact of the analytical procedures.

The challenges are magnified when trace analysis and particularly microtrace analysis is concerned.

Methods of analysis

The development of direct instrumental methods is a general trend in analytical chemistry resulting in time saving and waste reduction. Optimization of instrumental methods is often related to a decrease in sample volume needed for analysis. In some cases, there is a choice of direct techniques of analysis which may be defined as green processes, especially when the method is automated and uses a minimal amount of sample.

Presently, spectroscopic methods dominate the area of green analytical chemistry [10]. A special issue of *Spectroscopy letters* appeared in 2009 as an attempt to put green spectroscopy in the first line of the objectives of the spectroscopy community. The

issue was dedicated to green analytical techniques in the spectrometric analysis of environmental and biological samples [11], simple alternatives for sample pretreatment and analyte determination [12], alternative solid sample pretreatment methods in green analytical atomic spectrometry [13], a.o.

The most common method of sample introduction in atomic spectrometry is via a solution despite the fact that the large volume of solvent compared to the analyte may present problems to the atom cell: decrease in temperature, increase in the time of atomization, *etc.* The use of a thermospray sample introduction system is a way to remove or considerably reduce the solvent in a solution prior to its delivery to the atom cell. This can minimize potential interferences and improve the accuracy and precision of the analysis, as well as reduce the amount of waste [14,15].

Solid sampling spectroscopy strongly contributes to the development of green analytical chemistry methodologies. Direct solid sampling methods of atomic spectrometry are particularly useful for the analysis of materials that need sophisticated and time-consuming decomposition. Only several milligrams of the sample are consumed; contamination and loss hazards are brought to the minimum, no reagents are needed and there are no wastes. A number of direct solid sampling methods of atomic spectrometry are developed and optimized for the trace and microtrace analysis in various matrices like industrial materials, plants, plastics, foods, *etc.* [16–23]. Slurry sampling is another highly efficient green approach of atomic spectrometry, applicable to the trace element analysis in solid samples by flame AAS, electrothermal AAS, total reflection X-ray fluorescence spectrometry (TXRF) and other spectroanalytical techniques [24–31].

There are several benefits of using field analysis to reduce environmental impact. Along with better efficiency and financial profile, field techniques allow for analysis and data collection to be conducted on-site. As an example the field portable X-ray fluorescence (XRF) facilities for analysis of environmental samples may be mentioned [32, 33]. Energy dispersive XRF has found application as a field analysis method for the determination of trace and microtrace element contents in a large variety of samples like objects of arts, industrial materials, as well as for *in-vivo* measurements [34–38].

Sample preparation methods

Direct methods of analysis are although preferable, but not always available, particularly in the case of microtrace analysis in samples of complex matrix composition. In these cases sample preparation procedures are unavoidable. Sample preparation has three main functions [39]: (i) digestion/dissolution of the sample; (ii) preconcentration of the analytes; and (iii) separation of the analytes from the matrix components.

The choice of a separation/preconcentration procedure depends on the particular analytical task – method of analysis, type of analytes and matrix, required level of sensitivity, accuracy and precision of the analysis, loss/contamination hazards. Some of the most popular separation/preconcentration techniques are liquid-liquid extraction, precipitation/co-precipitation and solid phase extraction. These techniques are continuously improved and new techniques are introduced by the demand for faster, more cost-effective and environmentally friendlier analytical methods. For example, a simple and efficient sample preparation procedure was proposed for the flame or electrothermal AAS determination of trace elements in nails and hair by sample solubilization with aqueous tetraalkylammonium hydroxide [40].

Both existing methods and new procedures were improved by miniaturization and integration to provide higher sample throughput and/or unattended operation using minicolumns, coiled or knotted reactors, most often in flow injection performance [41–57]. A modern way of increasing the selectivity of the separation/preconcentration process is the solid-phase extraction using molecularly imprinted or ion imprinted polymers [58–61]. The hyphenation (integration) of separation and detection, as well as the incorporation of microwave or ultrasonic treatment considerably contributes to increase the efficiency and environmental safety of the methods, *e.g.* [62–67].

ASSESSMENT OF THE ENVIRONMENTAL IMPACT AND WASTE MANAGEMENT

Nowadays, in the development of new analytical procedures, the amount and toxicity of the wastes are as important as any other analytical feature. Miniaturization and automation are explored as approaches for waste minimization. As stated in the priority order for establishing of cleaner analytical

methods, if the use of toxic reagents cannot be avoided, the involved amounts should be minimized. This approach also has the advantage of reducing operational costs, including those spent on waste treatment and disposal [24]. Promoting clean methodologies in environmental remediation is the best way to address future challenges [68].

The use of organic solvents in separation and preconcentration methods is the main source of organic waste. The search for alternative solvents is an important step in making an analysis “greener” and environmentally friendlier. Moreover, alternative solvents like supercritical fluids and ionic liquids are even more attractive due to the possibility of varying their properties like solubility, polarity or volatility. Using supercritical fluids instead of organic solvents is becoming popular for most liquid-liquid extractions, especially when supercritical water or CO₂ are used as solvents [8,69]. The application of ionic liquids is intensifying in many areas of analytical chemistry, particularly in chromatography [70]. The non-volatility and good solvating properties, together with a large range of spectral transparency make them suitable solvents for spectroscopic measurements as well [8].

During the last two decades cloud point extraction has become a versatile and simple alternative to liquid-liquid extraction. In this method the organic solvents are replaced with non-ionic surfactants [71,72]. Some recent examples for the use of cloud point extraction for the preconcentration of metal ions prior to their spectrometric determination are given in [73–79].

Green analytical chemistry in Bulgaria

The analytical laboratories in Bulgaria are presently in a process of renovating their methodologies and adapting them to the European norms. This renovation is based on the green chemistry concept, recognized as one of the main tools for reducing environmental pollution. In Bulgaria, however, this process is at the initial stage. Nevertheless, some of the methods for trace element analysis by atomic spectrometry (XRF, AAS, ICP-OES, ICP-MS), published by Bulgarian scientists, are essentially green analytical methods, *e.g.*, [44, 80–93].

Recently, two projects in the area of green analytical chemistry were funded by the National Science Fund of Bulgaria. According to the first one, the GAMA – Green Analytical Methods Academic Centre was created at the University of

Plovdiv "Paisii Hilendarski". The aim of this research centre was to unify the efforts of prominent experts and young scientists of five universities and to focus their varied expertise towards development and application of modern analytical methodologies of trace element analysis in environmental samples like soils, plants and animal tissues. This project accentuates on the possibilities of inductively coupled plasma mass spectrometry and electrothermal atomic absorption spectrometry, combined with suitable sample preparation methods by observing the principles of green chemistry. A major activity of the GAMA Centre is the teaching of university students, PhD students and specialists on the issues of green analytical chemistry.

The second project deals with the development and practical application of total reflection X-ray fluorescence spectrometry (TXRF) at the Institute of General and Inorganic Chemistry of the Bulgarian Academy of Sciences. As an exemplary green analytical method TXRF uses sample amounts at the milligram level; involves minimum sample pretreatment for the analysis of solutions, suspensions or powders; the analysis is rapid and automated, with low energy, water and gas consumption [33, 94-96]. Within the project, TXRF will be applied to the trace element analysis of bottled Bulgarian mineral, spring and table waters, to the determination of the elemental composition of archaeological glasses and the determination of major and trace components in ashes of industrial biomass. Educational activities in the field of green analytical chemistry are also foreseen.

The dissemination of the theoretical and practical results of the mentioned projects at an educational, scientific and industrial level will contribute to achieving the goals of green chemistry in Bulgaria by:

- educating specialists, capable of handling and solving environmental problems in the (micro)trace analysis of different materials;
- creating a new approach to typical chemical activities leading to environmentally friendly use of facilities in science and technology.

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ЗЕЛЕНА АНАЛИТИЧНА ХИМИЯ И НЕЙНИТЕ ПЕРСПЕКТИВИ В БЪЛГАРИЯ

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(Резюме)

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Терминът „зелена химия“ е въведен през 90-те години на миналия век с цел да се минимизира влиянието на химичните дейности върху околната среда. В обзора са разгледани основните принципи, на които трябва да отговаря една химична процедура, за да бъде призната за зелена (щадяща околната среда). Тези принципи включват използване на по-малки количества реагенти и разтворители, отделяне на по-малки количества твърди, течни и газообразни продукти в резултат на използваните процеси, по-малко потребление на вода и енергия. Специално внимание е обърнато на особеностите на зелената аналитична химия, въвеждането на зелени принципи в аналитичната практика, оценката на влиянието върху околната среда и оползотворяването/обезопасяването на отпадните продукти. Разгледано е състоянието и перспективите за развитието на зелената аналитична химия в България, като е посочен приносът на българските аналитици.