Development of direct-current, atmospheric-pressure, glow discharges generated in contact with flowing electrolyte solutions for elemental analysis by optical emission spectrometry

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Direct-current, atmospheric-pressure, glow discharge (dc-APGD) generated in contact with flowing sample solutions is a new, very promising excitation source for analytical optical emission spectrometry, due to its low maintenance requirements and its analytical performance. Since analyzed solutions act as liquid cathodes, this discharge system is useful for the direct determination of elements dissolved in solutions without having to produce their aerosols by pneumatic nebulization.

The review presents different systems and designs of dc-APGD generated with the liquid cathode applied to spectrochemical analysis in the past 20 years. We discuss the effect of experimental conditions on analytical response and performance of the discharge. We include analytical figures of merit obtained with different discharge systems and their application to the analysis of environmental and biological samples containing various trace elements.
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1. Introduction

The need for compact portable instruments that can be operated in the field for on-line analyses is one of the most important motivations for developing and miniaturizing analytical measurement systems and radiation sources used in atomic and mass spectrometry [1–7]. Indeed, small sizes and weights of various microplasmas and microdischarges constructed and developed in past two decades provide real potential for the portability of analytical instrumentation and the possibility of real-time, on-site measurements [4]. Lower operating costs associated with a lower consumption of consumables (e.g., working gases, electric current, and cooling water) are additional advantages of these miniaturized plasmas and discharges [4,7].

In 1993, Cserfalvi et al. [8,9] introduced a new, exotic concept into optical emission spectrometry (OES) analysis [i.e. the direct contact of atmospheric pressure glow discharge (APGD), with the sample solution through the usage of the electrolyte itself as the cathode in the dc discharge]. This resultant low-power, direct-current APGD (dc-APGD), generated between metallic anodes and cathodes that are electrolyte solutions overflowing inlet tubes or capillaries (electrolyte cathode discharge, ELCAD) or sprayed at the exit of capillaries toward counter electrodes (liquid-sampling APGD, LS-APGD), is nowadays considered one of the most promising and alternative excitation sources to

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traditional plasmas [i.e. inductively-coupled plasma (ICP) and microwave-induced plasma (MIP)] used in OES for the determination of different metals in water samples and other environmentally relevant liquid matrices [10–14].

Fig. 1 shows a characteristic glowing appearance and a zonal structure of such APGD systems with regions distinguished from a surface of an electrolyte solution to a metallic anode. Fig. 1 includes the cathode spot (CS) [i.e. the cathode surface covered by the discharge, the cathode dark space (CDS), the negative glow (NG), the Faraday space (FS), the positive column (PC), and the anode glow (AG)] [12].

Atomic and molecular excitation phenomena taking place in ELCAD and LS-APGD systems are primarily related to this zonal structure despite the small sizes and compact geometries of these discharges.

The uniqueness of both these dc-APGD approaches, which distinguishes them from ICP and MIP, is a convenient radiation characteristic of atoms emanating from this type of miniaturized excitation source. Relatively simple atomic emission spectra and a very low ionic emission, resulting in rare spectral overlaps, are distinctive characteristics of ELCAD and LS-APGD and make both excitation sources convenient for direct, online determinations of traces of metallic contaminations in different sample solutions [10,13–18]. The power density focused between electrolyte solutions and counter electrodes typically reaches 5–85 kW/cm^3 [19,20]. This is also one order of magnitude or more greater than the power density reported for commercial ICP sources, and directly relates to very low volumes of both discharges. Also, in most ELCAD applications, quantitative measurements with reasonably good detection power (from tens to single μg/L) and short-term precision (from few to several %) when using OES for the detection are possible under conditions of very low consumption of discharge gases or completely in the air with no gas supply [21]. The total electric power required to sustain such discharges is also very low (i.e. 22–82 W), and mostly dissipated in CDS that controls the evaporation rate at the gas-solution interface [17,18,20–23]. Analyzed sample solutions serve as liquid cathodes and therefore allow direct sampling and routine analyses in process control or environmental monitoring studies [17,18,20–23]. When coupled to mass spectrometry (MS), LS-APGD can be used as a low power, low sample-consumption ionization source, quite desirable due to the small amounts of oxide and hydroxide ions of elements produced [24,25].

In terms of simplicity of spectrochemical devices, discharge systems of dc-APGD generated in contact with the liquid electrode can easily be sustained and controlled with little effort and low operating costs [14,17,18,21,26–29]. The progress made in constructions of discharge cells made in recent years [14,18,20,22,23,30–36] and fundamental studies of spectroscopic properties of these microdischarges [17,21,29,36,37] surely indicate that it is possible to enhance the concentration of atoms in the discharge and

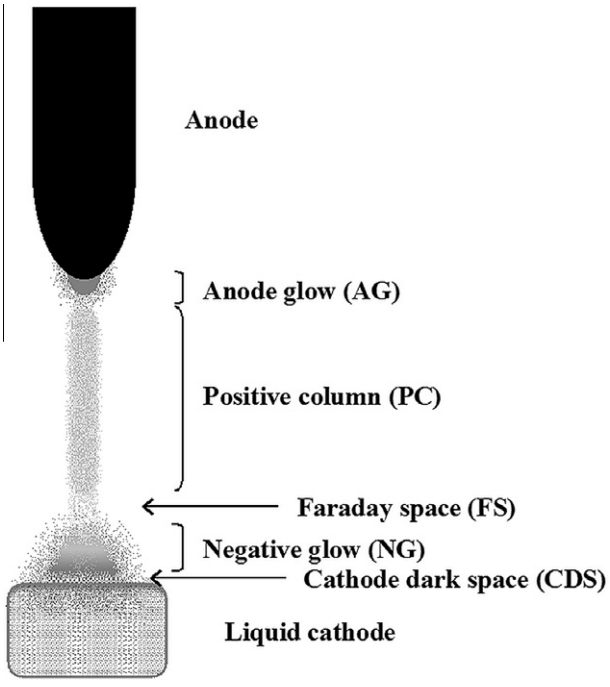

Figure 1. Discharge zones in direct current atmospheric pressure glow discharge (dc-APGD) generated in contact with a flowing liquid cathode (adapted from [9,30,36]).

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