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Original scientific paper

A Low-Cost Plasma Spectroscopy Instrument Design

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Abstract: While any instrumental elemental analysis is often thought of as an expensive and complicated technique requiring large investments, we present a design of a high-temperature plasma spectrometer, a Spark Discharge Optical Emission Spectroscopy (SD-OES) instrument, built with inexpensive, easy-to-find components, aimed at applications in education, industry and research. Opportunities that aim to make SD-OES accessible to a broad community as a high-quality alternative to the more costly instruments are highlighted. We describe and demonstrate the new, low-cost, prototype instrument for elemental analysis of solid and liquid materials. Application of the instrument to elemental analysis of various samples is described. The presented instrument design shows that high-precision SD-OES instrumentation can be accomplished using very modest and widely approachable components making the design described in this article affordable for various low-income educational institutions or small industrial entities. The high availability of free characteristic emission spectral lines databases, built for other similar analytical methods such as Laser Induced Breakdown Spectroscopy (LIBS), that can be used by this instrument, makes this design even more available for the general community.

Keywords: low-cost instrument; plasma spectroscopy; spark discharge optical emission spectrometry (SD-OES)

1 INTRODUCTION

Spark discharge Optical emission spectrometry (SD-OES) is an analytical technique based on Atomic Emission Spectroscopy (AES) [1]. This technique has become one of the standard methods, especially for element analysis of metallurgical materials [2-3].

SD-OES technique applies energy in the form of a spark generated between electrodes or between an electrode and a conductive sample. The atoms in the sample, vaporized by this event, are brought to a high energy state within a so-called "discharge plasma". In the plasma, these excited elements, atoms and ions, generate a unique emission spectrum specific to each element present. Therefore, each element generates several characteristic emission spectral lines. Furthermore, individual atoms can assume different excitation states depending on the complexity of the atom and the energy input [4]. Generated electromagnetic radiation is then captured by a spectrometer with a spectral range of about 200 - 800 nm.

SD-OES technique is similar to Laser-induced breakdown spectroscopy (LIBS); a technique that gained popularity in recent years due to the development of laser technology. The difference between the techniques is in the energy source that generates "discharge plasma". SD-OES uses an electric arc between the electrodes instead of a laser pulse paired with focusing lenses used in LIBS. However, LIBS is an expensive technique that requires high-cost pulsed lasers, and many portable LIBS instruments on the market are usually equipped with low-peak-power excitation lasers combined with low-sensitivity and low-resolution spectrometers [5], resulting in unsatisfactory performance in possible practical applications of the technique. On the other hand, SD-OES does not have this problem. The pulse power generating potential of the high voltage circuit utilized in SD-OES far exceeds that of the portable laser. Also, components of the high voltage pulse generating circuit are much cheaper than the pulsed lasers used in LIBS. With an increased discharge power, the SD-OES systems generate stronger emission spectral lines, making it feasible to use less sensitive spectrometers in this technique.

According to published works [6-12], SD-OES shows a similar or slightly better performance than LIBS for laboratory metal analysis. For laboratory analysis, SD-OES outperforms LIBS regarding sensitivity, reproducibility, and trueness [13].

Therefore, the SD-OES technique can be used to detect all metals, semimetals, and a number of non-metals, for instance, sulphur, carbon, phosphorous, oxygen, or nitrogen.

SD-OES is a minimally intrusive and effective technique that has been implemented in various fields of science and industry. In recent decades, many methods using this technique have been developed to determine plasma parameters such as electron density, vibrational and rotational temperatures, and electron temperature.

Although these techniques have a reputation for being expensive and hard to acquire for low-income educational or smaller-sized industrial organisations, our design shows that this can be accomplished using very modest and widely approachable instrumentation.

As opposed to the costly state-of-the-art SD-OES instruments that can cost tens of thousands, this article presents the design of a low-cost and precise SD-OES instrument that can be built for less than $1000. Another advantage of this design lies in the fact that above mentioned characteristic atomic emission spectral lines are the same no matter which technique of generating them is used. The expansion of LIBS has led to the development of a significant number of emission spectra databases in recent years, many of which are available online and for free [14] and therefore can be used by the potential owners of the instrument design described here. In this sense, the design allows for the use of high-cost instrument infrastructure on low-cost ones. The following sections present a detailed design of the low-cost SD-OES.

2 DESIGN CONSIDERATIONS

The SD-OES instrument consists of a high voltage pulse generating circuit, discharge electrodes, and a means to collect and analyse created discharge plasma light emission. To make it simple, the following design is

divided into the basic components, as shown in Fig. 1, which will be explained in detail in the following text.

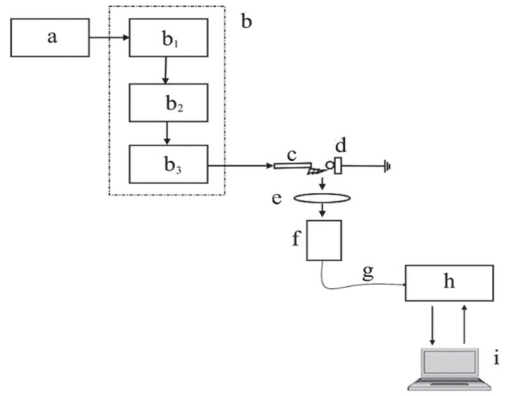

Figure 1 Basic components of the low-cost SD-OES instrument with: a) direct current low voltage source, b) high voltage pulse generating circuit (consisting of: b1) oscillating circuit, b2) high voltage step-up transformer, b3) pulse discharge circuit), c) high voltage discharge electrode, d) high voltage counter-electrode, e) light gathering lens, f) light-gathering optics, g) optical fiber, h) spectrometer, i) data acquisition and control

2.1 Direct Current Low Voltage Source

Various kinds of direct current (DC) voltage sources, ranging from common 3 volt (V) batteries to more expensive laboratory-grade DC power supplies, can be used in this setup. In this design, we can use 0 - 15 V, 0 - 3 amperes (A), variable Velleman LABPS1503 DC power supply or 0 - 30 V, 5 A, WEP-305D, or similar power supply.

2.2 High Voltage Pulse Generating Circuit

To create "discharge plasma" and evaporate part of the sample analysed, high voltage pulsed discharge is needed. In this stage, the DC supplied by the above-mentioned power supply is converted to high-frequency alternating current using the simple oscillating circuit. The high-frequency step-up transformer (HFTR) by Sanhe, China, is then used to increase the voltage above 5000 V. Created high AC voltage is further increased and rectified in the pulse discharge circuit to obtain high energy DC pulses created by fast discharge of the discharge condensers that are part of this circuit. The peak energy of the created high voltage pulse, responsible for creating the mentioned "discharge plasma", thus depends on the used discharge capacitor voltage (V) and capacitance (C) and the total impedance (Z) of the discharge circuit.

Schematic of the high voltage pulse generating circuit is shown in Fig. 2.

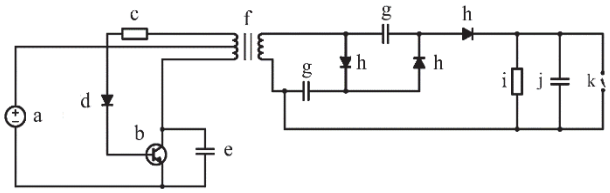

Figure 2 High voltage pulse generating circuit schematic, where; a) DC power supply, b) NPN transistor, c) 30 Ohm (Ω) resistor, d) 1N4007 diode, e) 50 nF capacitor, f) high-frequency transformer, g) 100 pF high voltage capacitor, h) high voltage diode i) 500 MΩ high voltage resistor, j) 5 nF high voltage capacitor, k) pulse discharge contacts

2.3 High Voltage Discharge Electrode and Counter

In the SD-OES analysis method, the choice of electrodes influences the sample spectra, because the characteristic atomic emission spectral lines belonging to the electrode material will be part of it. Therefore, the most convenient choice would be the material that has low emission intensity lines, or the characteristic atomic emission spectral lines, that lie far from the lines of interest on the spectrum of the sample analysed, as is the case with graphite electrodes. However, for simple qualitative analysis, easy-to-produce stainless-steel electrodes can be used but the lines belonging to electrode material must be taken into account or omitted. Likewise, analysis can be done in the air atmosphere, but lines belonging to oxygen and nitrogen from the air environment will be present. However, if higher precision results are needed, the electrodes could be placed in a simple plastic enclosure connected to an argon gas source, as shown in Fig. 3, to avoid emission from the air environment. However, to keep the design simple argon gas application is mentioned just as an option in this work. The counter electrode is made having the hollow part on the discharge receiving end, in which the sample to be analysed can be placed. When spark discharge happens between the electrodes, part of the sample, which is therefore placed in the discharge region, will vaporize into the "discharge plasma".

Fig. 3 shows the discharge electrode and counter electrode enclosure setup.

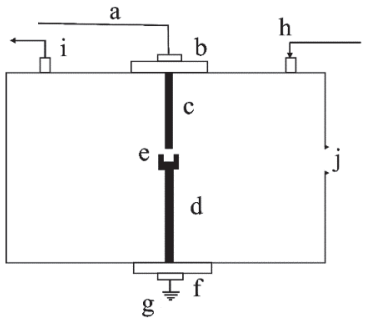

Figure 3 SD-EOS electrode setup in plastic enclosure with; a) connection to high voltage pulse generating circuit, b) discharge electrode el. connection, c) high voltage discharge electrode, d) high voltage counter electrode, e) counter electrode sample holder, f) counter electrode el. connection, g) ground connection, h) argon gas inlet valve, i) argon gas outlet valve, j) light gathering window

2.4 Light-Gathering Optics

Light emission that leaves the electrode enclosure through the light-gathering window (Fig. 3) is collected by light-gathering optics. The plano-convex lens with 25 mm diameter and 3 cm focal length is used in our setup. Light is focused by the lens to the SMA905 fiber optic coupler with an SMA 905-AD2 cosine corrected adapter and sent to the spectrometer light inlet through the FT0 30-Y multimode optical fiber.

2.5 Spectrometer, Data Acquisition and Control

ASEQ Instruments LR1-B, UV-VIS spectrometer with 300 - 800 nm spectral range is used for spectra collection.

A free Spectragryph v.1.2.16.1 [15] optical spectroscopy software is used as data acquisition and control software. Since the software has a characteristic atomic emission line identification function, it is also used for the acquired data processing.

3 DESIGN EXPANSES

A list of the parts used in the design of the low-cost SD-EOS instrument and their respective price is shown in Tab. 1.

Table 1 Lists the parts used in the design of the low-cost SD-EOS instrument and their price

No.	Part name	Pos. in text	Description	Cost ($)
1	DC Power Supply	Fig. 1a	0 - 15 V, 0 - 5 A	55
2	High voltage pulse generating circuit	Fig. 2	NPN transistor, IN 4007 and HV diodes, 30 Ω and 500 MΩ resistors, 100 pF, 5 nF, 50 nF capacitors, HFTR	20
3	Electrode setup	Fig. 3	Graphite or stainless-steel electrodes DIN EN 10088-3 1.4571 (AISI 316Ti), plastic box, gas valves, steel screws, and bolts	15
4	Lens	Fig. 1e	Plano-Convex lens, diameter 25 mm, 30 mm focal length	12
5	Optical fiber coupling	Fig. 1f	SMA905 coupling with SMA905-AD2 cosine corrected adapter	12,5
6	Optical fiber	Fig. 1g	1 meter, FT0 30-Y multimode	(included in spectrometer price)
7	Spectrometer	Fig. 1h	ASEQ-Instruments, LR1-B, UV-VIS, 300 - 800 nm	850
8	Data acquisition and control software	Fig. 1i	Spectragryph v.1.2.16.1 optical spectroscopy software	Free for educational purposes
Total cost ($)				964,50

Fig. 4 shows the designed low-cost SD-OES system constructed from the components listed in Tab. 1.

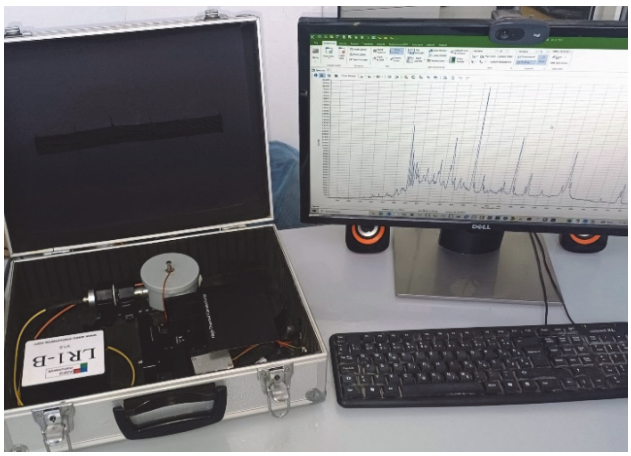

Figure 4 Photograph of a designed SD-OES system

4 DESIGN APPLICATION ON QUALITATIVE ANALYSIS OF MATERIAL SAMPLES

4.1 Analysis Method

A sample of a fine lime dust material (building lime, mainly Ca(OH)2, sourced by GIRKKalund.d. lime plant, Croatia) and a sample of granite rock, originating from Swiss Alps, are placed in the sample holder on the counter electrode as described earlier. "Discharge plasma" is created by the above-described high voltage pulse generator operating on 20000 V and 10 Hz frequency in air atmosphere over 28 mm^2 surface area of the sample between DIN EN 10088-3 1.4571 (AISI 316Ti) electrodes. Emission spectra are obtained using the mentioned ASEQ Instruments LR1 UV-VIS spectrometer. Spectra are averaged over 1000 ms intervals and peak values of interest are identified by Spectragryph v.1.2.16.1 optical spectroscopy software and cross-referenced with the National Institute of Standards and Technology (NIST) online database [16].

ASEQ Instruments LR1-B, UV-VIS spectrometer with 300 - 800 nm spectral range is used for spectra collection.

A free Spectragryph v.1.2.16.1 optical spectroscopy software is used as data acquisition and control software. Since the software has a characteristic atomic emission line identification function, it is also used for the acquired data processing.

4.2 Results and Discussion

The obtained lime sample spectrum is shown in Fig. 5, and the granite sample spectrum is shown in Fig. 6. As can be seen in Fig. 5 the high-intensity emission doublet on 393,4 and 396,8 nm belonging to ionized calcium species (Ca II) dominates the spectrum, as is expected for high calcium-containing material. Also, the spectral lines belonging to neutral oxygen (O I) on 777,6 nm and hydrogen atoms (H I) 654,6 nm are present along with neutral atomic and ionic spectral lines belonging to sodium (Na II), potassium (K II), atomic (Si I) and ionic silica (Si II) as is common for this material [17]. While the spectral lines belonging to atomic calcium (Ca I) and ionic Ca II, and also Na II, K II, Si I, and Si II originate from the sample material, the spectral lines belonging to O I and H I could originate from sample material, that is from the carbonate group and the hydroxyl group present in the lime sample respectively, but also from the air environment.

Likewise, the spectrum of the granite sample shown in Fig. 6 reveals that the granite rock is composed of Si, Ca, K, Fe, Mg, Al, Na, and Li which is in agreement with published works [18].

As is the case with the lime sample spectrum, the spectral lines belonging to the species originating from the air environment are also present.

As can be seen, the peak intensity of a key species and the overall quality of obtained spectra are of high quality and can be used for educational purposes as well as for quantitative analysis of the present species of interest. As can be seen in Fig. 5 and Fig. 6, there are several spectrally overlapping emission lines of neutral and ionized species present on both spectra. Therefore, a deeper investigation of trace element content would require a higher spatial

resolution spectrometer and there lies an opportunity to further improve the instrument.

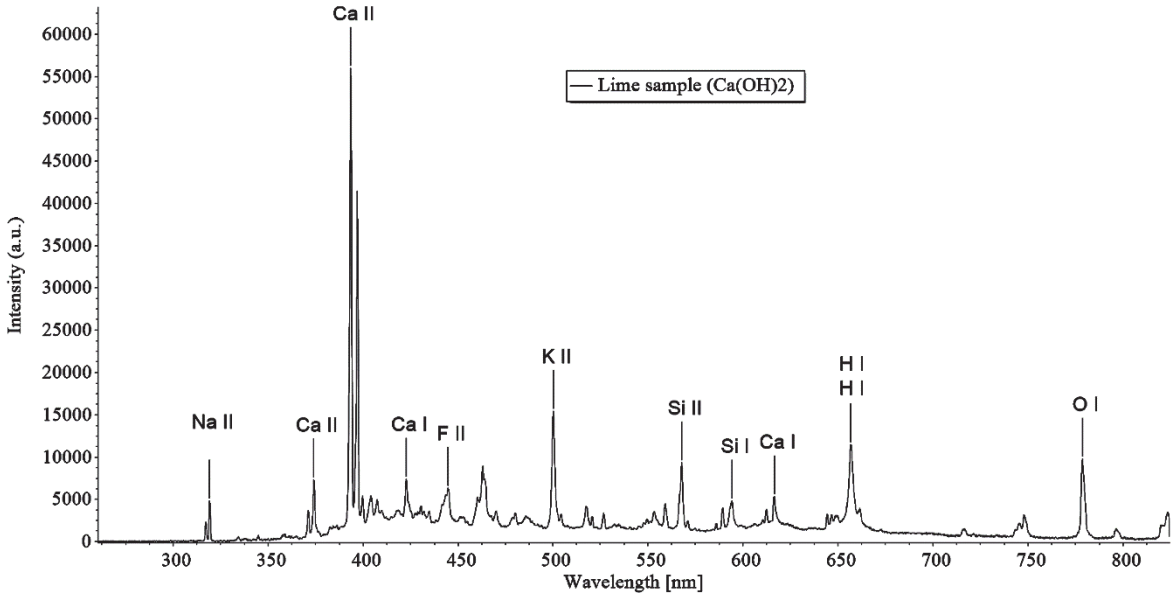

Figure 5 SD-EOS spectrum of the Lime sample

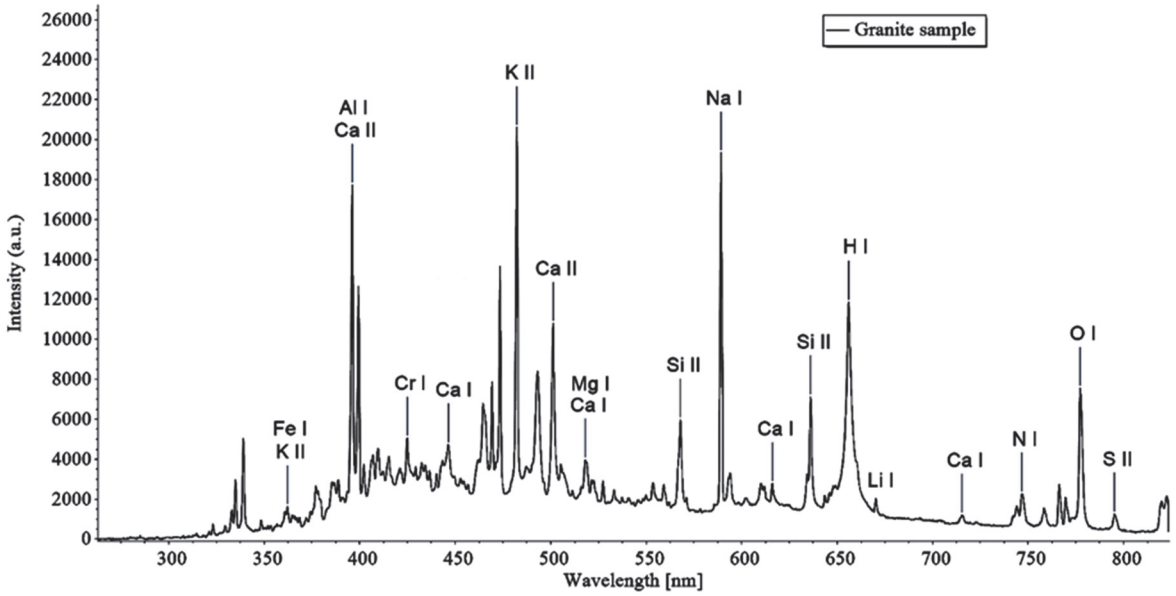

Figure 6 SD-EOS spectrum of the Granite sample

5 CONCLUSION

The designed low-cost SD-OES instrument obtains characteristic emission spectra of high enough quality to be used for educational or qualitative analytics purposes in industrial laboratories. Designed SD-OES is also suitable for scientific research where high spatial resolution of emission lines is not a primary requirement. Opportunity for the improvement of the instrument setup involves the installation of a higher resolution spectrometer capable of resolving spectrally overlapping emission lines of neutral and ionized species. The described method can be utilized to investigate chemical changes in most polymer materials where a change in the obtained spectra intensity of the carbon dimmer band could suggest a chemical degradation or corrosion of the polymer; e.g. Morosavljević et al. study [19] utilized SD-OES to investigate the corrosive effects of ozone on poly(methyl-methacrylate) where the change in the C2 Swan bands emissions was observed.

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