Abstract

Laser-induced breakdown spectroscopy (LIBS) is considered as a relatively young technique among other well-established analytical methods. It uses a high-energy pulsed laser beam for the generation of a plasma as the spectroscopic emission source for the determination of the elemental composition of a sample. Owing to its advantages, LIBS has already covered many research domains. However, due to the complex nature of a breakdown plasma, there are several challenges on its way to be used for routine analysis. Further research for analyzing the different kinds of materials is highly important for the optimization and the development of the technique. The main goal of this thesis is to understand the feasibility of calibration-free (CF) LIBS and artificial neural network (*ANN*) for the qualitative and quantitative study of materials in multi-disciplinary fields and offer a basis for the further growth of the LIBS method.

In the introductory section, an overview of the issues related to the topic and the details related to the experimental conditions have been reported (Chapter-4). Three different LIBS set-ups have been used to perform the experiments pertinent to this thesis. The main findings and the results obtained from the experiments have been divided into three chapters (Chapter 5-7). Chapter-5 of this thesis reports an extensive study of the influence of different experimental parameters (laser energy, gate delay, slit-width, lens to sample distance, gas pressure, and the composition of the surrounding gas atmosphere) on the LIBS signal and laser-ablation craters. This study has been carried out for four pure metals, *Al, Cu, Si,* and *Sn*. This initial study was conducted to determine the optimum experimental parameters and signal collection conditions. The next chapter (Chapter-6) dives into the study of fusion-related pure materials (*W, Mo, Ta,* and $W - Zr$ alloy) and coatings (*Be*-containing mixed coatings, $W - Ta + D/Mo$, and $W - Zr - D$), with special attention on fuel retention and depth profile analysis. Some of the results have been compared with other techniques (*e.g.,* SIMS, TOF-ERDA, GDOES, etc.). The analysis of these samples included the selection of the most suitable spectral lines and the determination of the plasma parameters (electron temperature and density) of generated plasma under the different experimental conditions (gate delay, gate width, pressure of the surrounding atmosphere). Boltzmann and Saha-Boltzmann plot methods have been explored for the determination of the electron temperature, while the Stark broadening of the spectral lines has been used for the estimation the electron density. In the subsequent part of the thesis (Chapter-7), multi-disciplinary samples (archaeological bones, *Fe/Co*-bilayer ribbon, pharmaceutical samples, and gold alloys) have been investigated using CF-LIBS and chemometric methods (*PCA* and *ANN*) for the qualitative and quantitative compositional analysis, depending on different requirements. With the combination of LIBS and chemometric methods, LIBS becomes more powerful to deal with complex datasets.

Through these applications, this thesis demonstrates the versatility and capability of the LIBS method for the compositional analysis of materials in diverse multi-disciplinary fields. This thesis is laying grounds for future work related to the widening of horizons of the LIBS technique.