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Effect of powder compact density on the LIBS analysis of Ni impurities in alumina powders

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Abstract

The quantification of elemental impurities in aluminum oxide powder is important due to its large demand for various industrial applications. One of the techniques to perform such analysis is Laser-Induced Breakdown Spectroscopy (LIBS) and to that end, powders are usually pressed into pellets. However, the density of the powder-compact is often not reported and its effect on the analytical figure-of-merit is unknown. In this work, we study the influence of aluminum oxide powder-compacts density of these pellets on the calibration curve of nickel contaminant. Our results show that the sensitivity increases with the grain size for a given density and that its relative standard deviation decreases with the density of the powder compacts without any grain size effect. These results imply that both the powder-compact density and the ultimate particle size of the powders must be specified in the protocol for building the calibration curve.

Key words: LIBS; powder; matrix effect

1. Introduction

Laser-Induced Breakdown Spectroscopy has established itself as a versatile technique for elemental analysis [1-3]. The technique is inexpensive compared to ICP-AES, XRF, ICP-MS and other mass-spectrometry based techniques [4]. It can detect all the elements in the periodic table, has simultaneous multi-elemental analysis capability, requires little or no sample preparation and imparts minimal damage to the samples. LIBS can be used to analyze solid, liquid or gaseous samples for qualitative and quantitative purposes [5]. The aforementioned traits along with its potential for real-time, *in-situ* application [6] have attracted LIBS to many fields such as metallurgy [7, 8], ceramic processing [9, 10], environment [11-14], geology [15-17], biology [18, 19] and forensics [20-23].

Aluminum oxide (alumina, Al_2O_3) is a material of interest for many industries. It is extracted from bauxite by the Bayer process and then used for several applications. Besides its consumption as a precursor for aluminum production by the Hall-Heroult process, it is used in a broad range of technologies from fillers in beauty products [24] to catalysts [25], from optical ceramics [26] to electronic insulators [27] and from refractory materials [28] to synthetic gems [29]. The required purity for the alumina as well as the type of impurities depends on its application. While research has been done to analyze bauxite directly [30] in the form of pressed powder pellets or fused glass beads, the analysis of alumina is important as well. The analysis of elemental impurities in powdered alumina can be performed by atomic spectroscopic techniques such as LIBS. Powdery samples are analyzed by LIBS, either in the form of loose powders on a substrate [31], loose powders on micro-structured targets [32, 33] or more commonly pressed pellets [34] but the way the sample is prepared for the analysis can influence the analytical figure-of-merit.

This work focuses on the impact of matrix density and morphology in the quantification of nickel in aluminum oxide powder. Alumina powders with three different morphologies were spiked with nickel chloride at concentrations ranging from 500 to 3000 ppm by weight, and compacted to various densities without binding agent to study the effect of compaction and particle size on the sensitivity and its relative standard deviation of calibration curve for nickel.

2. Experiment

The different varieties of α-alumina powders used in this study are labelled S1, S2 and S3. Sample S1 (99.99% purity, Inframat® Advanced Material™) is composed of 190 ± 64 nm-sized particulates agglomerated into particles with average hydrodynamic diameter of 180 nm. Sample S2 (99.99% purity, Inframat® Advanced Material™) has a