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Session 7 · Environment, Security, and Safety

Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS) in accelerating DWPF melter operations. The capability of direct analysis of slurry will significantly increase analytical throughput and will reduce waste generation in radiological analytical facilities, providing analyses suitable for waste acceptance and production records. Laser Induced Breakdown Spectroscopy (LIBS) will serve as the basis for the system for direct analysis of DWPF Sludge Receipt and Adjustment Tank (SRAT) product (a slurry consisting of only sludge or waste). The analytical results will be used to support the determination of the appropriate amount of frit to be combined with the sludge in the melter. The main issues to be resolved with LIBS analysis of liquid samples are poor detection sensitivity and precision. Because water can guench the laser plasma and suppresses the LIBS signal, poor sensitivity may result. Large standard deviations for LIBS liquid data are due to the laser induced shock wave caused by turbulence on the liquid surface. Slurry samples contain a large amount of water and large particle sizes. The effects of water content and particle sizes on LIBS measurement will need to be studied. To evaluate the figure of merit of direct slurry measurement with LIBS, two DWPF simulant slurry samples with different acid levels from SRNL were used in the study. The test results with these slurry samples showed that the splattering and surface cavitations result in large signal fluctuation. To improve LIBS' reproducibility and detection limits for slurry measurements, various experimental parameters which can affect LIBS' analytical figure of merit were studied. The study shows that by using the appropriate slurry sample handling systems, optimum experimental parameters and some data processing techniques, reasonable accuracy and precision (below 5%) based on 2.5% error confidence for the major elements can be achieved. For minor elements, we found the accuracy and precision are poorer (about 10-15%), Current work demonstrates that LIBS has the potential to be developed for on-line analysis and control of DOE wastes slurry processing. However, further work on improving signal sensitivity and data reproducibility is needed. The details of the sample handling systems and their effect on the LIBS signal will be described in this paper. The effect of the slurry sample rheology on the LIBS signal will also be presented.

Analysis of trace elements in liquids using LIBS combined with Laser-Induced Fluorescence

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Session 7 · Environment, Security, and Safety

Analysis of liquids by existing analytical techniques is well established, and does not call for the laborious preparatory steps often needed for solids. However, the reliable on-line analysis of liquids required to meet demands for improved control of industrial processes that are often difficult to achieve by conventional techniques. LIBS has been proposed for monitoring various elements in liquids, during industrial processing, as an alternative to sampling for subsequent laboratory analysis. In this work, the combination of the Laser-Induced Breakdown Spectroscopy (LIBS) and Laser-Induced Fluorescence (LIF) techniques was investigated to improve the limit of detection (LoD) of trace elements (iron, lead and gold) in liquid in order to meet industrial and environmental requirements. The plasma was produced by a 266 nm frequency-quadrupled Q-switched Nd:YAG laser and then re-excited by a nanosecond Optical Parametric Oscillator (OPO) laser. To improve the reproducibility of the measurements, we built and used a cell that enables the laser to sample a fresh surface while preventing problems associated with the laser liquid interaction such as splashing. aerosols, bubbles etc. The influence of the main experimental parameters on the LIF signal, namely the ablation fluence, the excitation energy, and the inter-pulse delay, was studied. We will also discuss about the importance of the selected excitation-fluorescence scheme. Using the LIBS-LIFS technique, we demonstrated LoDs for Fe and Pb of a few tens of ppb after accumulating over 100 laser shots while its value was about a few hundreds of ppb for Au. These values represent an improvement of about two orders of magnitude with respect to LIBS.

Utilization of Laser-Induced Breakdown Spectroscopy for monitoring of trafficking and deposition of selected heavy metals and nutrition elements in plant compartments

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Laser-induced breakdown spectroscopy (LIBS) belongs to the most developing spectroscopic techniques for spectrochemical analysis1. LIBS, similarly to laser-assisted inductively coupled plasma mass/optical spectrometry (LA-ICP-MS/OES), allows fast and accurate study of different materials both for analysis in the laboratory and industrial environments1. The LIBS technique utilizes the high power densities obtained by focusing the radiation usually from a pulsed, fixed frequency laser to generate in the focal region a luminous micro-plasma from an analyte (solid, liquid and gaseous samples). The micro-plasma emission is subsequently analyzed by spectrometer1. The main difference between LA-ICP and LIBS resides in



