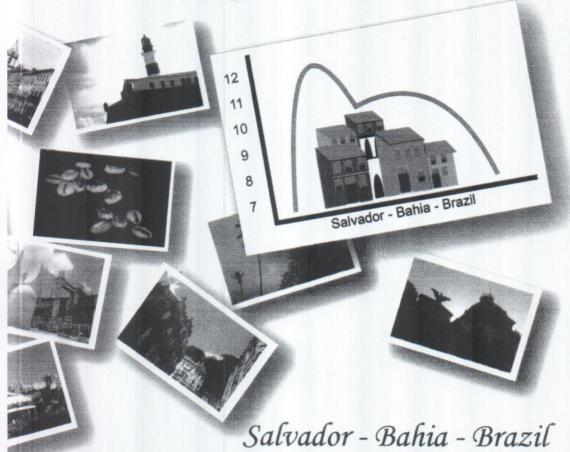# Book of Abstracts

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Analysis de Ca in corn cereals by Laser Induced Breakdown Spectroscopy

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Laser Induced Breakdown Spectroscopy (LIBS) is a kind of emission spectroscopy which utilizes a focused high energy laser pulse to create a plasma in a solid, liquid, or gaseous media. In analysis of solid samples, part of the energy in the plasma is used to ablate the material. After the ablation process, the plasma rapidly expands, sending a shock wave into the surrounding media. In the core of the plasma, effective temperatures can easily exceed 10,000 K. During this stage, material in the core of the plasma is vaporized, atomized and ionized, and the plasma is typically highly ionized. As the plasma cools, continuum emission from the plasma fades, typically much faster than emission lines from neutral and singly-ionized atomic lines. Thus, by adjusting an optimal temporal detector gating it is possible to collect elemental emission.

Since excited species return to their fundamental states emitting characteristic radiation, the qualitative analysis of emission spectrum provides the "fingerprint" of sample with regard to its elemental composition. The use of LIBS for element identifications is advantageous in that it allows direct and fast analysis, besides being almost non-destructive, since the ablated sample mass for laser pulse is in the order of micrograms. Despite attractive LIBS features, quantitative analysis should be evaluated for different samples and analites, especially with regard to calibration and matrix effects.

In this proposal a LIBS method for Ca determination in cereals was studied. Only corn cereals were considered to avoid different matrix influences effects. Six commercial cereals presenting Ca in a range of 41mg kg^{-1} to 3200 mg kg^{-1} were considered to perform the calibration curve. The amounts of Ca given for manufactures were confirmed by ICP-OES analysis of decomposed samples. Before analysis the samples were homogenized by cryogenic grinding. For LIBS analysis the samples were submitted to a pressure of 12 ton to make pellets. The pellets were analyzed in a system arranged by spectrometer LIBS2500plus, a LIBS- SC sample chamber (both from Ocean Optics) and a Big Sky laser which provides a maximum laser pulse of 50mJ. Ten spectra were acquired for each calibration sample corresponding to two accumulated pulses. A cleaning shot was always used before spectra acquisition. Emissions intensities at 393.2 nm of each spectrum previously corrected for offset were used. The analytical curve showed R-value of 0.997 and the Levene´s test applied to check the residuals showed the population variations are not significantly different at the 0.05 level, meaning the curve really presents a fit linear. The precision of measurements were in order of 6%.

The validation was performed using a corn bran certificate reference material (NIST-8433). The concentration value obtained was (418 ± 55) mg kg^{-1}, which presented good correlation with certified value $((420 \pm 38)$ mg kg$^{-1})$. The method presented good performance dispensing the step of sample preparation and it was applied to Ca concentration prediction in other commercial samples.

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