# A detailed study of spectral electron spin resonance parameters to determine the firing temperature of archaeological and contemporary ceramics

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## Abstract

Electron spin resonance (ESR) spectra can be describe by the spectroscopic parameters: g-factor, peak-to-peak linewidth and signal intensity. Signal intensity corresponds to the value of the double integral and/or to the peak-to-peak amplitude of the spectrum. Changes in any of these spectroscopic parameters can be used in archaeometry as a reference to determine the firing temperature of ceramic objects. Due to the complexity of the spectrum is not possible to determine the resonance Hamiltonian, and therefore this association is purely empirical. Thus, the most studies in literature estimate the firing temperature of archaeological ceramics using the changes of the denominate “experimental g-factor”, which define the g-factor value of the peak of the central line of Fe(III). However, some others studies use others spectroscopic ESR parameters to estimate the firing temperature. Thus, this work made a detailed analysis of spectral ESR parameters changes of six different between archaeological and contemporary ceramic samples to determine the better parameters to estimate firing temperatures. All archaeological and contemporary ceramic sherds samples studied were collected in archaeological sites of the Marajó island at Pará state, Brazil. This place is found at the eastern part of the Brazilian Amazonia. For analysis, the external surface of samples were cleaned with tungsten carbide drill bits, to preventing contamination, since the fragments were dirty. Then, to ensuring the chemical homogeneity, we extract over five fragments of each ceramic sample to obtain representatives powder samples of ~800 mg of each sample. For ESR analysis, each of these powder samples was divided in 11 aliquots with similar masses (~70 mg). Later, to estimate the firing temperatures of the different ceramic samples using the ESR technique, the aliquots were heating to ten different temperatures between 400 and 850$^{o}$ C. Finally, ESR measurements were carried out in a JEOL FA-200 X-Band ESR spectrometer at room temperature. ESR measurements showed typical signals of Fe(III) species in different environments, and as expected, the spectra showed clear changes with reheating temperature increasing. A detailed analysis of each ESR spectrum showed that the spectral parameters that have been changed more clearly to determine the firing temperature were the g-factor and the peak-to-peak linewidth of the central resonance line of Fe(III). Changes in these parameters may be used due to a oxidation of species of Fe(II) to Fe(III). Thus, for the studied samples, the better spectra ESR parameters to determine the firing temperatures were the g-factor and the peak-to-peak linewidth of the central resonance line, and not the “experimental g-factor”.