Multivariate and univariate Chemiometrics develop single analytical procedure for determination of metals in different tissues of blue crab

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Palavras Chave: *single analytical procedure, effect of matrix, Desirability.*

Abstract

Treatment of the data array multivariate showed that there was difference between the concentration of analytes in the different tissues and factorial design shows the existence of difference matrix in the procedure.

Introdução

Development of a single analytical procedure for the determination of Cu, Fe, Mn and Zn in different tissues of the animal needs to consider the following factors: the difference of the concentration of analytes in different tissues, equivalence in sample preparation of different tissues, and thus the residual sample have similarity in physical and chemical characteristics, because if there is a significant difference may introduce systematic errors in the analytical technique for the determination[1].

Resultados e Discussão

The treatment of the data array for PCA and HCA showed that there was difference between the concentration of analytes in the different tissues, graphs as shown in the figure 1.
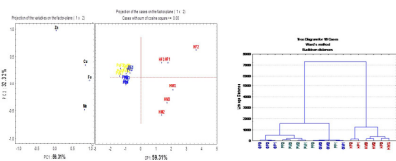

Fig.1 Graphics of PCA and Dendogram of HCA.

Factorial design shows the existence of difference matrix in the procedure of prepare. Afterwards, the answers, concentration of the metals, were transformed into overall desirability individually for each tissue and the date processing reports there is difference between the tissues with respect to the significant factors to each tissue, Figure 2.
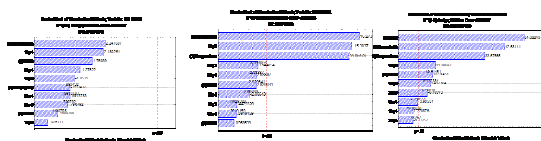

Fig.2 Pareto graphic of the (a) claws, (b) paws of locomotion and (c) hepatopancreas tissues planning in relation to OD.

Therefore, overall desirability function, from the individual desirability, was performed to determine a single condition to all samples, which the ideal conditions were the followings for total digestion assisted by radiation microwave: temperature 160 °C; time of 21 minutes; 3.0 mL of nitric acid and 3.0 mL of hydrogen peroxide to approximately 0.2000 g of lyophilized sample. For the technique of determination, the matrix did not influence on the determination of analytes in different tissues, with a view that there was no significant difference at 95% confidence level between the sensitivity of the following methods of calibration: external, residual acidity and addition of analyte in different tissues. The accuracy of the procedure was confirmed by analysis of certified reference material (CRM) oyster tissue (NIST 1566B). A statistical evaluation using expanded uncertainty[2] showed that there is no significant difference between the value obtained with the use of the proposed procedure and the certified value, at 95% confidence level. The analytical characteristics of the proposed procedure for Cu, Mn, Fe and Zn were: limits of quantification of 10.5; 1.6; 8.9 and 19.3, in µg g-1, respectively. The precision estimated by the analysis of samples (% RSD, n = 7) had values below 5 % in all three tissues. The proposed procedure was applied in samples blue crab of Cachoeira river estuary, Ilhéus, Bahia, Brazil.

Conclusões

Multivariate optimization using the following methods; HCA and PCA, full factorial planning and desirability function and test significance made it possible to obtain a single condition (instrumental and chemical) that enabled the simultaneous decomposition of the three tissues at a microwave due the different compositions of these matrices.

Agradecimentos

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