School supplies raw material evaluation using microwave sample preparation

José Augusto Garcia[1] (PG), Edenir R. Pereira Filho[2*] (PQ)

[1]*Centro de Tecnologia da Informação Renato Archer, Campinas – SP, CEP 13069-901.*
[2]*Universidade Federal de São Carlos, Departamento de Química, São Carlos – SP, CEP 13565-905*

Keywords: *school supplies, factorial design, toxic elements, lead, microwave oven extraction.*

Introduction

School supplies raw material presents a great variety of samples matrices (paper, wood, ink, rubber, caulim, polymers/masterbatch, among others). In addition, this type of material is considered toys in several countries and metals content are very restrictive. Elements like Ba, Cd, Cr, Hg and Pb can cause several healthy problems, mainly in children[1-3]. In this case the aim of this study is to evaluate a procedure to extract Ba, Cd, Cr, Hg and Pb in solid samples, using microwave oven with closed vessels. The development of this study was based on directive 2009/48/CE[4], which specifies the levels of elements in the materials used by children. Chemometric strategies were used to evaluate the results obtained.

Results and Discussion

A total of eight type of samples (masterbatch, polymer, pigment, caulim, paper, acrylic ink, fabric and moulding mass) were used to evaluate the extraction process using MARSXpress (CEM) tubes. This system (low pressure) permits the preparation of up to 40 samples simultaneously and presents high analytical frequency. Masterbatch is a polymer with high pigment content. Two full factorial designs were performed and in the first one a 2^3 was done and principal component analysis (PCA) was used to select only critical samples (simultaneously high remaining solid content and metals concentration) for the proposed process, reducing the number of samples tested. The variables tested were H_2O_2 content, analytes addition and the possibility to perform a sequential extraction. The analytes were determined using ICP OES (inductively coupled plasma optical emission spectrometry). Masterbatch, polymer and caulim were simultaneously related to high solid content and high metals concentration. These samples were selected for the next factorial design (2^3) and the following variables investigated: addition of H_2O_2, addition of analytes and HNO_3 volume. The responses observed for the second factorial design were recoveries of analytes (Ba, Cd, Cr, Hg, and Pb) and desirability function[5] was used to combine all responses. Caulim and polymer presented the same trend and required similar extraction conditions (2 mL H_2O_2 and 5 mL HNO_3). On the other hand, masterbath required only high volume of HNO_3 (10 mL). These samples were also analyzed using a high pressure system (XP1500, CEM). This system permits the preparation of 12 samples with the same chemical composition due to the use of monitoring vessel. The obtained results for Pb were compared with those obtained with the low pressure system (see Table 1, n = 3).

Table 1: Pb content (mg kg^{-1}) in the selected samples.

Sample	MARSXpress	XP1500
Caulim	21.2 ± 1.7	25.6 ± 3.2
Masterbatch	32.7 ± 4.5	25.5 ± 1.2
Polymer	1.1 ± 0.8	6.6 ± 0.5

The maximum Pb content permitted[4] in this type of samples is 13.5 mg kg^{-1} and both tested systems (low and high pressure) were able to identify concentrations higher than this limit. For samples where the Pb concentration is between 10 µg L^{-1} (Limit of quantification obtained by ICP OES) and 13.5 mg kg^{-1} a high pressure system (XP1500) must be used in order to confirm the result.

Conclusions

It is not possible to prepare some samples with different physicochemical characteristics (masterbatch and polymer, for example) in the same batch and the proposed system can be used in order to perform a sample screening.

Acknowledgments

The authors are grateful to Faber-Castell, Fapesp, CNPq, Capes and the Chemistry Graduation Program of UFSCar for the opportunity to develop a professional master course.

[1]Hillyer, M. M.; Finch, L. E.; Cerel, A. S.; Dattelbaum, J. D., Leopold, M. C.; *Chemosphere* **2014**, *108*, 205.
[2]Guney, M.; Zagury, G. J.; *J. Hazard. Mater.* **2014**, *271*, 321.
[3]Da-Col, J. A.; Sanchez, R. O.; Terra, J.; Bueno, M. I. M. S; *Quim. Nova* **2013**, *6*, 874.
[4]http://ec.europa.eu/enterprise/sectors/toys/documents/directives/index_en.htm, acessada em agosto de 2014.
[5]Candioti, L. V.; de Zan, M. M.; Cámara, M. S.; Goicoechea, H. C.; *Talanta* **2014**, *124*, 123