Development of a MSPD-GC-MS analytical method for PAH extraction and analysis in cashew nuts (*Anacardium occidentale L.)*.

Jaiane S. Suzarte[1] (IC), Michel R R Souza[1] (PQ), Marcelo R Alexandre[1]* (PQ).

[1]Departament of Chemistry, Federal of Sergipe University, São Cristóvão - SE, Brazil.

Key Words: Cashew nuts, PAH, MSPD, GC-MS.

Introduction

Cashew Nut is a high nutritional value food consumed worldwide. To be marketed, it is processed by drying, roasting and breaking out the nut. In this process, the cashew nut may be contaminated by compounds derived from pyrolytic sources, such as the polycyclic aromatic hydrocarbons (PAH). The PAH have been considered as priority pollutant by the Environment Protection Agency of the United Stated[1] due they mutagenic, teratogenic and carcinogenic characteristics[2]. The aim of this work is to develop a Matrix Solid-Phase Dispersion[3,4] associated with gas chromatography coupled to mass spectrometry (MSPD-GC-MS) analytical method for determination of 16 priorities PAH in roasted cashew nut (*Anacardium occidentale L.*) marketed in the city of Aracaju-SE, Brazil.

Results and Discussion

The analytical method was developed starting by establishment the chromatographic conditions to analyze 16 PAH in a GCMS Shimadzu, model QP-2010 plus, with a 5MS ZB capillary column was used (30 m, 0.25 mm ID, 0.25 µm of film thickness, Phenomenex, USA). The initial oven temperature was 40°C (2.0 min); heating rate of 25°C min $^{-1}$ up to 100°C (0 min); 5°C min^{-1} up to 230°C (0 min); 2°C min^{-1} up to 270°C (5 min); 3°C min^{-1} up to 320°C (0 min). Injection was done in the splitless mode (1 min) at 40°C. The temperature of the interface was 320ºC and the carrier gas helium (99.995%) was set at 0.69 mL min^{-1} (15.8 kPa). The mass spectrometer was operated in SIM mode (selected ion monitoring) with ionization Electron Impact (EI) at 70 eV. The injection volume of the extracts was 1 uL. The total analysis time was 72.07 min.

The cashew nuts were obtained in the Aracaju city market, Sergipe. They were taken to the laboratory, crushed and stored in a clean glass vial. The MSPD was developed by using the external standard method: 0.50 g of cashew nut [spiked with 25 uL of HPA mixed solution of 16 (10 mg ml^{-1}) and 25 uL surrogate p-terphenyl D14 (10 mg mL^{-1})] was dispersed in Florisil® (1.5 g). C_{18} (0.5 g) was used as auxiliary column. The elution solvent was acetonitrile, 10 mL.

The initial results have showed recovery ranging from 32.41 up to 98.55% with RSD less than 20% for most of the studied PAH (n = 3, table 1). According to the literature, for complex samples such as cashew nuts, recoveries between 70-120% are acceptable with precision until ±20%[5].

Table 1. Relative Standard Deviation (RSD %) and Recovery (REC %) of the studied PAH

PAH	REC(%)	RSD(%)
naphtalene	32.41	84.27
acenaphtalene	55.32	29.54
acenaphthene	57.33	28.33
fluorene	70.87	7.79
phenanthrene	88,33	6.61
anthracene	86.47	5.91
fluoranthene	87.26	12.48
pyrene	90.26	11.75
p-Terfenil D14	91.26	6.57
benzo[a]anthracene	93.62	15.68
chrysene	97.69	17.66
benzo[b]fluoranthene	98.55	14.51
benzo[k]fluoranthene	98.25	11.42
benzo[a]pyrene	98.44	18.86
indeno[1,2,3-cd]pyrene	94.53	20.51
dibenz[a,h]anthracene	95.15	19.91
benzo[ghi]perylene	91.66	18.07

Conclusions

The association of MSPD and GC-MS techniques gave a quick, easy and efficient extraction and analysis method for determination of 16 priorities PAH in roasted cashew nuts. The next step is to use the internal standard procedure for the analytical method validation, as well as adjustments on the chromatographic conditions.

Acknowledgements

CAPES, CNPq and FAPITEC-SE for research supporting.

[1] US Environmental Protection Agency (EPA), Method 610, **1982**.
[2] Lau, E.V.; Gan, S.; Ng, H. K.; Poh, P.E., *Env. Pollut.*, **2014**, *184*, 640.
[3] Barker, S.A., *J. Chromatogr. A.* **2000**, *885*, 115.
[4] Souza, M.R.R.; Moreira, C.O.; Lima, T.C.; Aquino, A.; Dorea, H.S., *Microchem. J.* **2013**, *110*, 395.
[5] Ribani M.; Bottoli C.B.G.; Collins C.H.; Jardim I.C.S.F.; Melo L.F.C. *Quim. Nov*. **2004**, *27*, 771.