GC-MS/MS method validation for determination of acrylamide in fried potato.

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Introdution

Acrylamide (CH_2=CH-CO-NH_2) is an important industrial chemical[1]. The neurotoxicity of acrylamide in humans is well established from occupational and accidental exposures, and experimental studies have shown reproductive, genotoxic and carcinogenic effects in animals. Acrylamide has been classified as probably carcinogenic to humans by the International Agency for Research on Cancer[1]. Acrylamide, which is formed during normal cooking practices, such as roasting, baking or frying, was found primarily in carbohydrate-rich foods prepared or cooked at high temperatures[2]. The major pathway involved in the acrylamide formation is the Maillard reaction between amino acids and carbonyl compounds, such as reducing sugars,at temperatures above 120 ºC [1,2].

The aim of this study was to validate a method for the determination of acrylamide in fried potato using solid phase extraction (SPE) sample preparation, and gas chromatography-mass spectrometry (GC-MS/MS).

Results and Discussion

Raw potato samples were used in the method validation. Sample preparation involved purification with SPE graphitized carbon black column, derivatization with bromide and addition of triethylamine for the completely conversion of 2,3-dibromopropionamide to 2-Bromopropenamide. The $^{13}C_3$-acrylamide were used as internal standard[3].

The separation were performed in a GC-MS/MS (Shimadzu) using a RTX-5MS column (30 m x 250 µm x 0.25 µm). The GC parameters were: injector: 200 °C (splitless), oven: 50 °C (1 min), followed by an increase of 5 °C min^{-1} to 90 °C, and after 25 °C min^{-1} to 260 °C. He at flow: 2.0 ml min^{-1}. Mass spectrometer conditions: electron ionization (70 eV); Low Collision energy: 10 eV; SRM: scan time: 0.15 sec.; Channel #1: transition 151>70; Channel #2: transition 154>73; Voltage detector: 0.8 kV. The validation of the analytical method was performed by the following parameters: linearity, matrix effect, precision, accuracy, repeatability and limits of detection and quantification according to IUPAC[4]. Acrylamide showed linearity in the concentration range of 0.5 to 80 µg kg^{-1}, with correlation coefficients (r) of 0.9999. No matrix effect was observed, enabling the use of standards in solvent for quantification. The accuracy was evaluated in three different levels with recoveries ranging from 97 to 112%. Repeatability was verified with six repetitions, performed in a short period, of one concentration level of the compounds with RSD ranged from 2.3 to 19.2%. The results were satisfactory for acrylamide, since the RSDs were below 40% [4]. The limit of detection were from 0.3 µg kg^{-1}, and the limit of were 5 µg kg^{-1}.

Conclusões

The results of the method validation indicate that the GC-MS/MS is suitable for the determination of the acrylamide in fried potato, which demonstrates the versatility of the method that has been widely used for analysis of residues in several matrices.

Agradecimentos

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