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Development of LC-Q-TOF-MS and LC-MS/MS method for the determination of grayanotoxins in foodstuffs and its application to food science study

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sensitive and specific high-performance liquid chromatography-Quadrupole-time of flight mass spectrometry (LC- $\mathbf{1}_{Q}$ -TOF-MS) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) method was developed for the determination of grayanotoxin I (GTX I) and grayanotoxin III (GTX III) in foodstuffs. Grayanotoxins (GTXs) were extracted from foodstuffs via solid-phase extraction using HLB solid-phase extraction cartridges. LC-Q-TOF-MS was used to determine the fragmentation patterns of GTXs and to characterize their fragmentation pathways. LC-MS/MS method was developed and validated for quantitative determination of GTX I and GTX III in foodstuffs. Separation of LC-Q-TOF-MS was made using a Waters ACQUITY BEH C18 column (100×2.1 mm, 1.7μ m) and separation of LC-MS/MS was made using a Capcell pack MG II C18 column (2.0 mm \times 100 mm, 3 μ m). The mobile phase consisted of distilled water (v/v, A) and methanol (v/v, B) containing 0.1% formic acid. Electrospray ionization mass spectrometry was operated in the positive ion mode. The calibration curves of LC-MS/MS obtained were linear over the concentration range of 10-100 ng/mL (GTX I), 20-400 ng/mL (GTX III) with a lower limit of quantification of 7.5 ng/mL (GTX I) and 15 ng/mL (GTX III), respectively. The relative standard deviation of intra-day and inter-day precision was below 10.35% and accuracy ranged from 83.7 to 112.0%. The analytes were stable in the stability studies. An investigation of 51 foodstuffs from the online and offline market reveals the presence of GTXs. A quantitative estimation indicated total toxin concentrations of 1.84-101 ng/mL (GTX I) and 1.53-37.4 ng/mL (GTX III) in foodstuffs. The potential of this approach is especially demonstrated by the fact that at least two of these toxins have not been previously described in the literature. The validated method was successfully applied to the quantification study of GTXs in foodstuffs for the first time.

Biography

Taeik Hwang is working as Scientific Officer at Food and Drug Safety evaluation in Ministry of Food and Drug Safety. He completed his Masters from Soonchunhyang University in Korea.

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