Screening for total ergot alkaloids in rye flour by planar solid phase extraction coupled to fluorescence detection and mass spectrometry

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Ergot alkaloids are commonly produced by the ergot fungus Claviceps purpurea and are responsible for poisonings and toxicological effects in mammals. The parasitic fungus is mainly growing on cereals, particularly on rye, and the infestation of grain with Secale cornutum, the permanent form of Claviceps purpurea, is therefore a serious problem. Nowadays, about 50 ergot alkaloids from Secale cornutum are known, commonly derivatives of lysergic acid. The total alkaloid content of Secale cornutum varies considerably, depending on the origin between 0.01 and 0.5%, when a content of 0.2% is assumed for Central Europe. Despite the known toxicity and the infestation of rye grain and rye flours with Secale cornutum, there are no maximum limits established for ergot alkaloids in grain and grain-based food in Europe [1]. Nevertheless, the European Union strongly recommends the monitoring of ergot alkaloids in food and feed and plans to regulate the total ergot alkaloid content of relevant food categories.

Since for monitoring the quantity of individual ergot alkaloids is not relevant, and only the sum of ergot alkaloids is monitored, the detection of ergot alkaloids as the sum is a meaningful and efficient new approach and offers the easy assessment of the exposure to ergot alkaloids.

Therefore, a fast screening method for the determination of the total ergot alkaloids in rye by planar solid phase extraction (pSPE) was developed. pSPE was recently introduced by Oellig and Schwack as a clean-up method for pesticide residue analysis in fruits and vegetables and tea samples, and offers the separation of target substances from matrix compounds and focus the target analytes in a single zone [2-4]. After a single methanol development on high-performance thin-layer chromatography (HPTLC) amino plates, ergot alkaloids are detected as the sum, according to the pSPE concept. For quantitation, the native fluorescence was enhanced with n-hexane/paraffin and scanned at UV 254/>400 nm. Method performance parameters were highly satisfactory with limits of detection and quantitation of 0.07 and 0.24 mg/kg rye, respectively, expressed as ergocristine, and near-100% recoveries for Secale cornutum spiked rye flours at spiking levels around the currently applied quality criterion limit for rye. The fast pSPE–FLD is an efficient alternative to the time-consuming HPLC determination of individual alkaloids and calculating the sum of them. HPTLC–MS offers the identification and determination of the ergot alkaloid composition in a single mass spectrum. Using the mass spectrum “fingerprint”, the differentiation of Secale cornutum from different origins will be easy possible.

Literatur: