

Method optimization and validation for the analysis of tropane alkaloids in cereals and legumes samples using functionalized mesostructured silica as strong cation-exchange solid-phase extraction sorbent

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Gold statement

- New method for the determination of tropane alkaloids in cereals and legumes has been applied.
- Functionalized silicas with sulfonic acid groups were used as sorbents in solid-phase extraction.
- Analysis was performed by high-performance liquid chromatography-tandem mass spectrometry.

Introduction

Tropane alkaloids (TAs) are secondary metabolites produced by a different variety of plants such as *Brassicaceae* or *Solanaceae*, between others [1]. Atropine and scopolamine are the two most representative compounds of this group. The presence of TAs in food due to contamination of crops by these toxic plants can have serious health consequences. For this reason, EFSA has recommended the analysis of TAs in different matrices such as flours or cereal-based products [2]. In this sense, sample preparation is a very important step to avoid interference in HPLC-MS/MS analysis, so interest in the use of new sorbents as functionalized mesostructured silicas is growing.

Body

In this communication, we report a new method based on the use of functionalized mesostructured silica (SBA-15 type) during the sample preparation stage. Firstly, SBA-15 was synthesized and later functionalized with sulfonic acid groups (SBA-15-SO₃⁻) groups. The resulting material was characterized by different techniques and evaluated as strong cation-exchange solid phase extraction (SCX-SPE) sorbent for the determination of atropine and scopolamine and in cereals, pseudocereals and legumes samples (buckwheat, sorghum, corn, corn blue, quinoa, millet, red and green lentil, chickpea, pea, teff and amaranth) prior to HPLC-MS/MS analysis.

The optimized method consisted of a solid-liquid extraction with an aqueous-acid medium (pH1, HCl), followed by a purification of the extract by SCX-SPE using 150 mg of SBA-15-SO₃⁻. This method was validated following the SANTE guidelines, showing recovery rates between 93-96% and quantification limits lower than 5 µg Kg⁻¹, amount recommended by the European Commission. Also, the prepared material was compared with a commercial sorbent, under the same conditions, obtaining worse recovery rates than the material synthesized in this work. Finally, the method was applied to the different samples, finding atropine concentrations in nine samples and scopolamine in one sample.

Conclusion

The new material has proven to be suitable for the extraction of TAs by SCX-SPE. Therefore, it is useful for the control of atropine and scopolamine in different types of cereals and legumes samples susceptible to being contaminated by these compounds.

References

- [1] P.P. Mulder, *EFSA Supporting Publications* **13** (2016) 1-200.
- [2] EFSA, *EFSA Journal* **11** (2013) 1-113.