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Ensuring food safety through the study of potential extractables and leachables in roasted coffee packaging by ultra-sensitive Headspace GC/MS

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1. Introduction

- The main function of food product packaging is to protect food against any contaminant. However, in some circumstances, this packaging can release possible substances that pose a risk to human health. These are extractables and leachables (E&L) compounds that migrate from materials into the product, especially under factors such as temperature, time, and solvents.
- Notably, Brazilian legislation lacks a consolidated assessment to monitor these compounds in the food industry.
- The Headspace Shimadzu HS-20 NX trap model allows the concentration of compounds, up to 10 times, in a trap material. This means that even very small traces of these contaminants can be detected and quantified accurately.
- When coupled with GCMS-QP2020 NX (**Fig.1**), the HS-20 NX becomes a powerful tool for identifying and analyzing potential toxic contaminants, significantly contributing to food safety and compliance with quality regulations.

Fig. 1 GCMS-QP2020 NX + HS-20 NX.

2. Methods

The HS-20 NX allows sampling in either loop or trap mode. In the case of trap mode, an electronic cooling system is integrated, enabling compound concentration and high-sensitivity analysis (**Fig. 2**). The gas in the headspace is directed to a tube with trap material, which is cooled to concentrate the components present in the sample. Subsequently, through the thermal desorption process, heating of the trap, the concentrated compounds are released and introduced into the GC. This allows for the analysis of samples that are even in small quantities, such as roasted coffee powder.

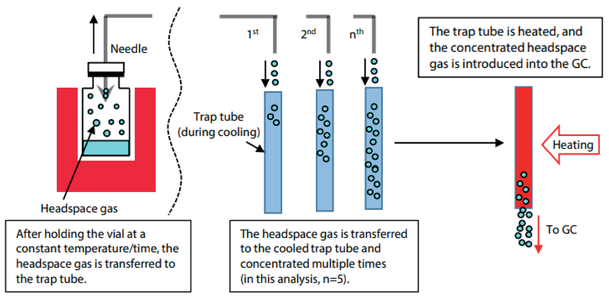

Fig. 2 Sample Concentration in Trap Mode.

2.1 Analytical Method

The analyses were performed in both loop mode and trap mode using 5 times the sample concentration, with the total chromatographic runtime set at 62 minutes. Separation of substances was achieved using a 0.32 mm × 30m × 0.25µm Stabilwax column. Out of this total runtime, 30 minutes were used for sample preparation on the HS-20 NX, while the remaining 32 minutes were dedicated to acquisition on the GC/MS.

2.2 Sample Preparation

To prepare the samples, several brands of roasted and ground coffee were chosen, packaged in different packaging (**Fig. 3**). The samples were divided and weighed into 20 mL vials, considering different components, such as adhesive and non-adhesive parts, aluminum parts and box. Each element, such as the capsule's internal filter, metal body and top seal, along with the corresponding roasted coffee powders, was analyzed.

Fig. 3 Illustration of sample preparation.

3. Results and Discussion

- During the analyses, only in Trap mode was it possible to detect E&L compounds due to increased sensitivity. Additionally, successful analysis of the characteristic coffee aroma was achieved.
- In the chromatogram (**Fig.4**), it's possible to see a magnified view of the difference between the modes in the same sample.

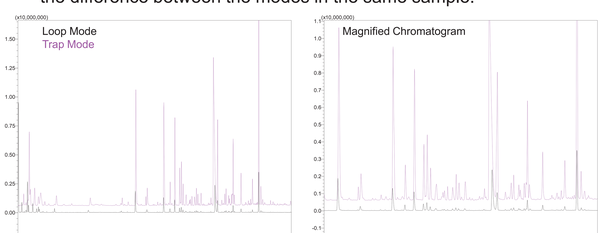

Fig. 4 Loop vs Trap comparison of the chromatogram of the coffee powder sample.

Using the m/z filter, specific fragments were detected confirming possible E&L compounds, such as Acetone, Ethanol, Ethyl Acetate, Toluene and Cumene. Additionally, the sample was compared with a matrix exposed to some of the solvents to confirm the results (**Fig. 5**). This fact was corroborated subtracting the chromatogram of the packaging from the coffee powder sample and the presence of some compounds exclusive to the packaging **(Fig.6).**

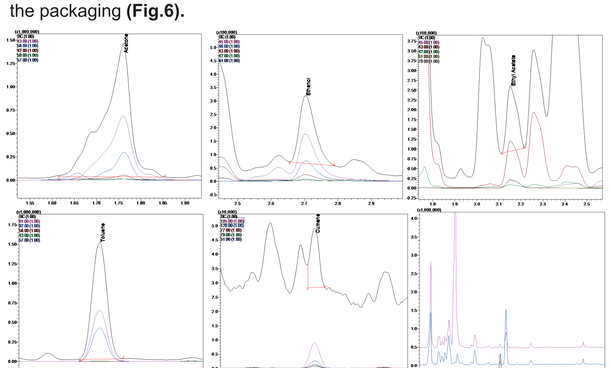

Fig. 5 TIC chromatogram and m/z of possible E&L compounds.

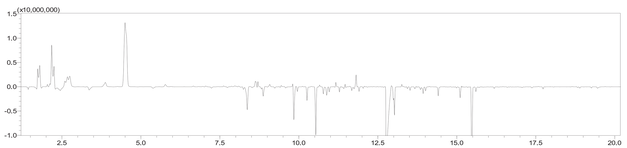

Fig. 5 Chromatogram of the packaging subtracting the coffee powder sample.

- Using the Shimadzu Smart Aroma Database, it was possible to identify the main compounds of coffee aroma during the same E&L analysis **(Fig. 7).**

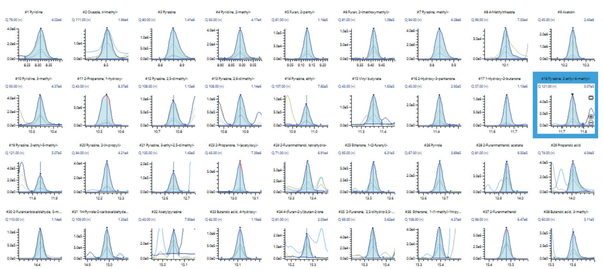

Fig. 7 Mass chromatogram of coffee aroma.

4. Conclusion

- The use of HS-20 NX in Trap mode with GC/MS enabled the identification of over 100 compounds, comparing the spectra with the National Institute of Standard and Technology (NIST) library. Among these compounds are potential E&L substances, as well as coffee aroma components.
- This technique demonstrates high versatility and efficiency by providing high analytical sensitivity capable of identifying compounds even at low concentrations, something unachievable by conventional methods. This evolution represents a notable advancement in coffee and food quality control, enabling precise detection of essential characteristics and components to ensure the excellence of the final product and consumer health.

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