

Evaluation of Mineral Oil Hydrocarbons Contamination in Dry Foods Using Advanced Chromatographic Techniques (LC-GC/FID and GC×GC-QToF/FID)

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INTRODUCTION

Petroleum distillation and refining generate a complex mixture of chemical compounds known as Mineral Oil Hydrocarbons (MOH). MOH can enter the food chain through multiple pathways, including their use as lubricants in food production machinery, processing aids, food or feed additives and food packaging materials.

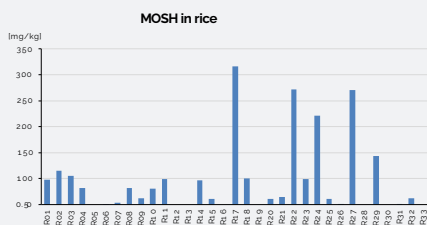
They are classified into Mineral Oil Saturated Hydrocarbons (MOSH) which consist of linear, branched, and cyclic saturated hydrocarbons and Mineral Oil Aromatic Hydrocarbons (MOAH) which comprise aromatic structures with varying degrees of ring condensation and alkylation.

Given the potential genotoxic and carcinogenic effects associated particularly to the MOAH fraction¹, MOSH/MOAH detection and quantification are of growing concern.

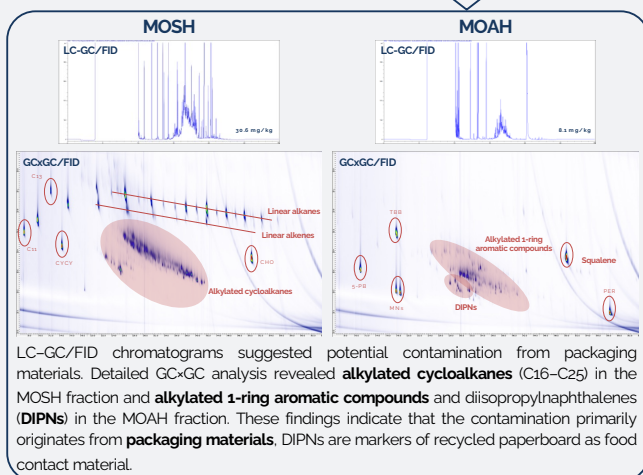
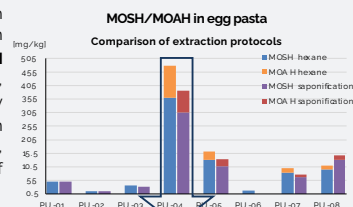
The present study aimed to evaluate MOSH and MOAH levels in a range of commercially **cereal-based dry foodstuffs**, a potential daily source of exposure.

RESULTS

Rice samples contained only MOSH, ranging from <0.50 to 3.2 mg/kg, consistent with literature data. **MOAH levels in all rice samples were below the limit of quantification** (LOQ: 0.50 mg/kg for foods with <4% fat/oil).



Egg pasta was analyzed using both extraction protocols: *n*-hexane extraction and saponification. **MOSH and MOAH levels were similar with both methods**, suggesting that contamination is mainly superficial. Values were higher than those observed in durum wheat pasta, likely due to the higher fat content of egg pasta, as reported in the literature⁴.



CONCLUSIONS

- **No critical contamination of MOHs** was detected in any sample.
- Complex foods, such as pasta and cereals, required more **in-depth analytical methods**. Including an initial step of **alkaline saponification resulted in higher yields** compared to other tested protocols.
- According to the literature, **egg pasta is more susceptible to MOHs contamination** due to its higher fat content; however, our deeper characterization revealed that the main source of **contamination derives from packaging**.
- Breakfast cereals, due to their compositional complexity, required **additional purification steps**, and they showed **multiple sources of MOHs contamination**.

MATERIALS AND METHODS

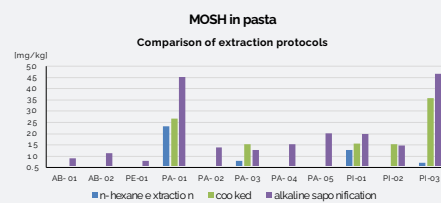
Samples were collected from Italian supermarkets to represent a wide range of products available to consumers. The selection included: durum wheat semolina **pasta** (PA), whole wheat pasta (PI), **egg pasta** (PU), early childhood pasta (AB), **rice** (R), soy and rice **noodles** (PE), and **breakfast cereals** (CB).

To extract superficial contamination, samples were soaked in *n*-hexane overnight. For more complex matrices (pasta and cereals), an **alkaline saponification** step – prior *n*-hexane extraction – was applied to break down the matrix, release internal contamination, and purify the fat content. In both protocols, an **internal standards mixture** was added for quantification.

MOSH and MOAH fractions, within the C10–C50 range, were determined using online liquid chromatography coupled with gas chromatography and flame ionization detection (**LC-GC/FID**)³. Further qualitative and compositional insights were obtained through comprehensive two-dimensional gas chromatography with high-resolution time-of-flight mass spectrometry and flame ionization detection (**GC×GC-QToF/FID**).

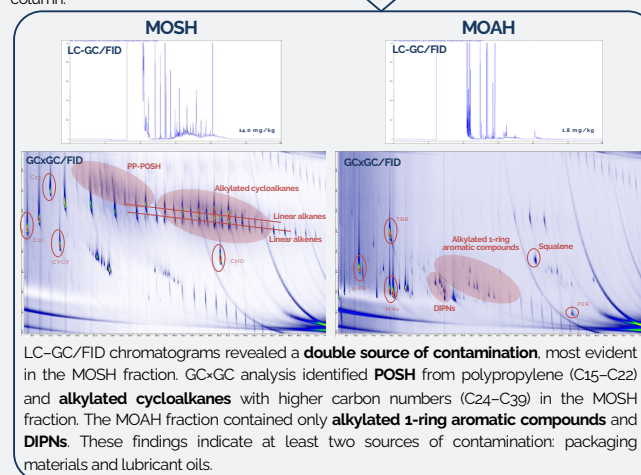
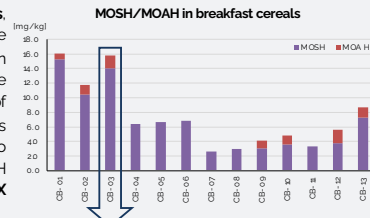
Superficial testing showed that only a few **pasta** samples had MOSH levels above the LOQ, while **MOAH were undetectable in all samples**.

Due to the structure complexity of pasta, two protocols were compared to extract internal contamination: samples were subjected to **cooking in water** or **alkaline saponification** before *n*-hexane extraction.



The results indicated the presence of internal contamination, with the saponification protocol being the most effective method for extracting MOSH and MOAH.

Different types of **breakfast cereals**, including muesli and cornflakes, were subjected to alkaline saponification followed by MOSH/MOAH *n*-hexane extraction. Due to the presence of vegetable oils, some samples required **epoxidation** reaction prior to LC-GC analysis. In addition, the MOSH fraction was purified using an **ALOX** column.



LC-GC/FID chromatograms revealed a **double source of contamination**, most evident in the MOSH fraction. GC×GC analysis identified **POSH** from polypropylene (C15–C22) and **alkylated cycloalkanes** with higher carbon numbers (C24–C39) in the MOSH fraction. The MOAH fraction contained only **alkylated 1-ring aromatic compounds** and **DIPNs**. These findings indicate at least two sources of contamination: packaging materials and lubricant oils.

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