Formulation-specific consumer products analysis for alleviating ion-suppression in MS

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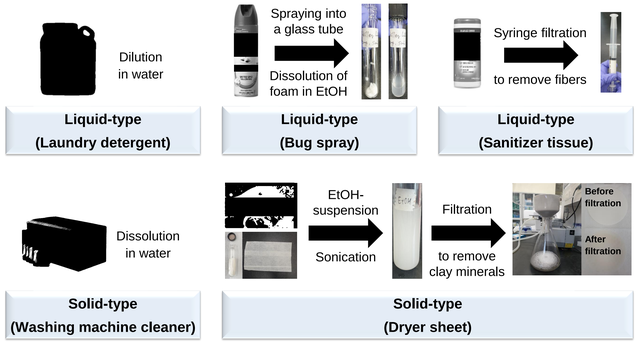
Introduction

- **Widespread use of consumer products (CPs):** A number of CPs are widely used in our daily life, and their annual usage is increasing.
- **Unreported information on chemicals contained in CPs:** Despite the routine use of CPs, there is a lack of toxicity information and safety guidelines. The chemicals consumed may pose risks to humans due to their potential toxicities and various exposure routes.
- **Necessity of performing formulation-specific CPs analysis:** Common CPs like detergents and hand sanitizers often contain ion-suppressing interferences such as surfactants and salts. This can significantly reduce the signal response when directly analyzing extracts from these products using MS. Notably, existing studies on MS analysis of CPs have not addressed the need to remove these interferences during sample preparation.

Methods

Sampling of various formulation of CPs

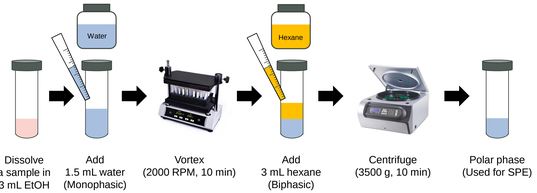


Listing up disclosed chemicals in CPs

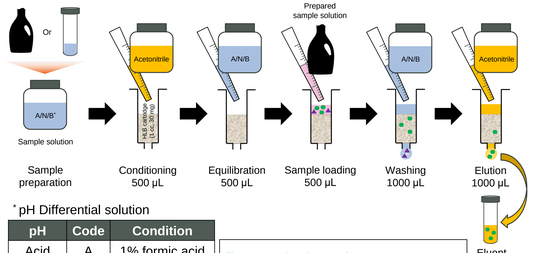
➢ Information on the major ingredients was available on the packaging of the CPs or the manufacturer's website.

Analytes extraction

[LLE]



[SPE]



* pH Differential solution

pH	Code	Condition
Acid	A	1% formic acid
Neutral	N	HPLC water
Base	B	2% NH_4OH

● : Neutral polar analytes
▲ : Anionic or cationic surfactants or salts

LC-ESI-MS/MS analysis

➢ Instrument: Agilent 1290 Infinity II UHPLC system coupled to 6546 Q-TOF mass spectrometer

LC Parameters		Conditions					
Column		Zorbax C18 (2.1 x 150 mm, 5 µm)					
Mobile phase		A: 0.1% acetic acid in water:ACN (4:6) B: 0.1% acetic acid in IPA:ACN (9:1)					
Flow rate		0.4 mL/min					
Injection volume		5 µL					
Gradient	Time (min)	0	12	15	22	23	30
	Solvent B (%)	2	45	80	80	2	2

MS Parameters	Conditions
Ion polarity	Positive/negative
Mass range	70 – 1,000 m/z
MS and MS/MS resolution	30,000 / 7,500
Acquisition mode	Data-dependent (Top 5 precursors)
ID database	NIST20 CID library

Results and discussion

Formulation-specific sampling

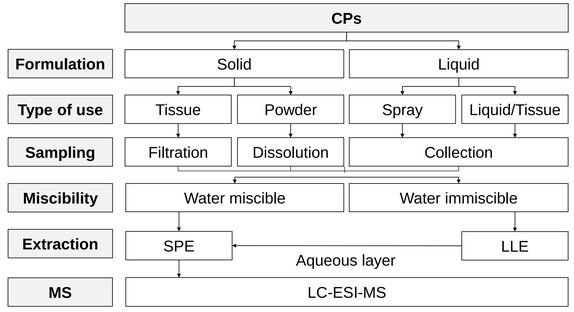


Figure 1. Classification of sample preparation methods depending on the physical properties of CPs.

➢ The sampling method was chosen based on the sample formulation. For samples with pH-dependent properties, SPE was used. Additionally, samples extracted using LLE underwent further SPE to remove ion-suppressing interferences.

➢ The sanitizer comes in the form of a wet sheet, composed of a nonwoven fabric made of pulp and cellulose, and a liquid that contains an active ingredient functions as a disinfectant. Therefore, it was classified as a liquid.

➢ A major challenge during sampling was the presence of insoluble particles, likely bentonite clay from the dryer sheet, which could interfere with the analysis. To address this, the extracts obtained from the wet wipes were filtered through a glass microfiber filter.

Efficiency of pH-dependent SPE

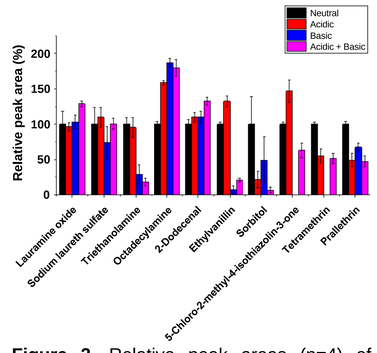


Figure 2. Relative peak areas (n=4) of detected compounds from major ingredients of CPs and identified using the NIST20 database.

Solvent selection in LLE

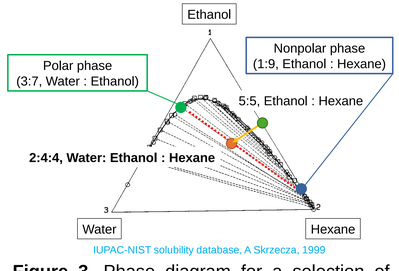


Figure 3. Phase diagram for a selection of solvents in LLE.

➢ The results suggest that the ionized fraction of the compound in solution, which varies with pH, influenced its interaction with the HLB cartridge.

➢ Lauramine oxide and sodium laureth sulfate exhibited similar peak areas across three distinct pH conditions, suggesting a lack of pH-dependent effects. This finding indicates that the partitioning of compounds with long alkyl chains is dominated by nonpolar interactions.

➢ The miscibility boundary in a ternary mixture determines whether it is in a monophasic or biphasic state. For polarity-selective analysis, a biphasic system is necessary, allowing the dissolved CP solution to separate into two phases. Hexane was selected as the non-polar solvent for the development of an ethanol-based biphasic solvent system.

Conclusions

➢ Since some CPs contain silicate minerals that must be removed before MS analysis, sample preparation should be tailored to the characteristics of each CP.

➢ Although pH-dependent SPE was performed to reduce ESI interferences, the pH conditions also affected other compounds of interest. Therefore, selective SPE, considering the specific compounds to be analyzed, is required.

Acknowledgements

This work was supported by Korea Environment Industry & Technology Institute(KEITI) through Technology Development Project for Safety Management of Household Chemical Products, funded by Korea Ministry of Environment(MOE)(KEITI: 2020002960003, NTIS: 1485017761)