

## THE XXXVII IULTCS CONGRESS 2023 第37届国际皮革工艺师和化学家协会联合会大会

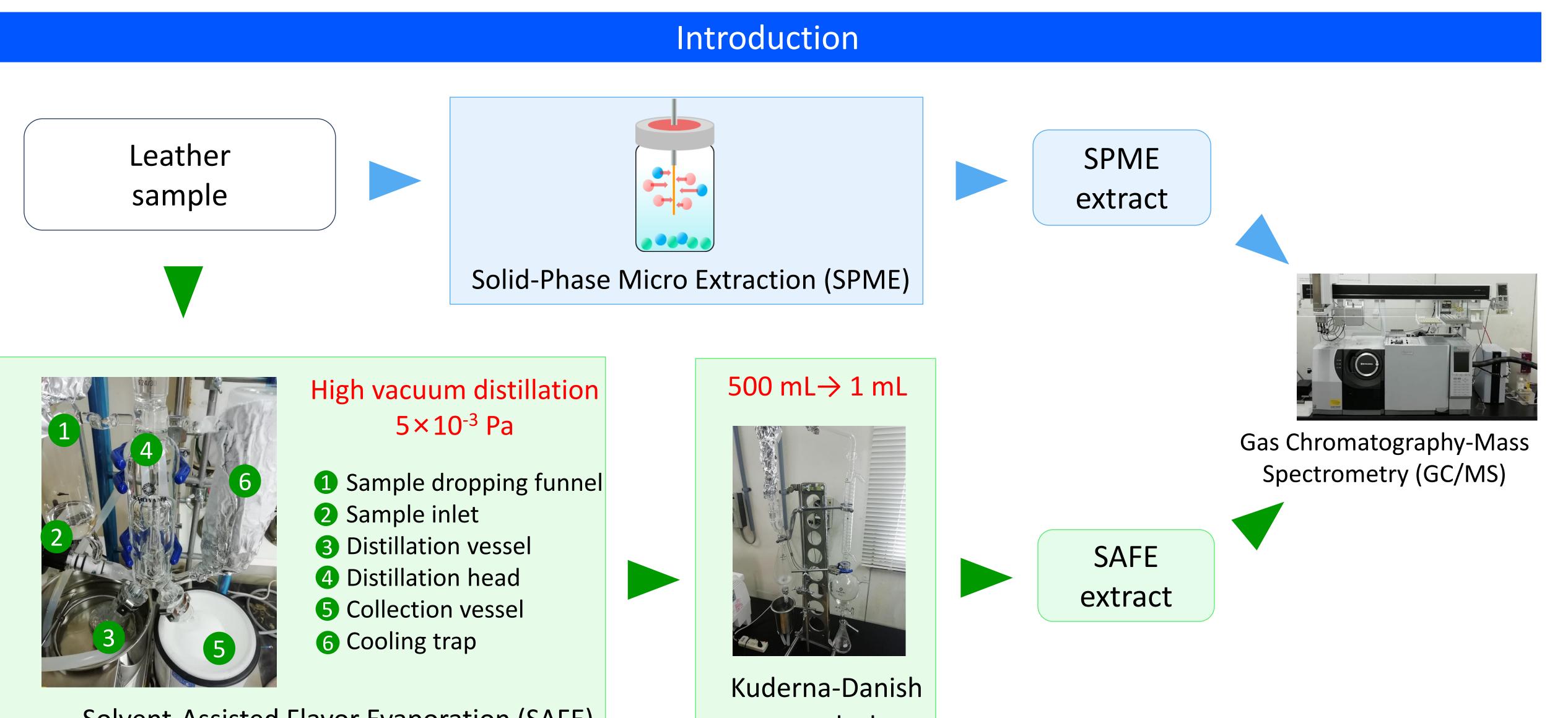
# Comparative analysis of volatile compounds in leather using solvent-assisted flavor evaporation and solid-phase microextraction

Hirohiko Washiya, Osamu Harada

Technical Support Center for Leather Industries, Hyogo Prefectural Institute of Technology, 3 Nozato, Himeji, Hyogo 670-0811, Japan

#### Abstract

The volatile component of leather is a mixture of different compounds. Gas chromatography-mass spectrometry (GC/MS) is used to analyze volatile compounds. Pretreatment is required before sampling. Solid-phase microextraction (SPME) is a solvent-free sample preparation method that uses coated fibers to extract analytes from samples prior to GC. SPME is a simple method, but the adsorbed compounds depend on the type of fiber. Quantitative analysis is difficult. To understand the totality of volatiles in leather, we need to look at solvent extraction. Solvent Assisted Flavor Evaporation (SAFE) distillation is a well-known in the food sector. In combination with a high vacuum pump, high boiling compounds can be removed by high-vacuum distillation. In this study, volatile compounds in leather were extracted using two extractions, SAFE and SPME. 35 volatile compounds were identified by SPME and 31 by SAFE.



concentrator

#### Material and Methods

#### Material

Finished leather with a urethane coating

#### **GC/MS** analysis

GC-MS: GCMS TQ-8040 (Shimadzu Corp., Japan) Column: InertCap-WAX (30 m x 0.25 mm, 0.25 μm, GL Sciences Inc., Japan).

Carrier gas: Helium

Injection mode: Splitless [SPME], Split 10: 1 [SAFE] Oven program 40 °C (5 min.) - 10 °C/min - 240 °C (10 min.) Ionization: El at 70 eV, Scan range: m/z 28-400. Ion source temp.: 200 °C, Interface temp.: 250 °C.

#### **Extraction of volatile compounds by SPME**

Sample: 0.5 g, SPME Fiber: wide range/PDMS fiber (95 μm) Extract temp.: 80 °C, Extract time: 10 min.

#### **Extraction of volatile compounds by SAFE**

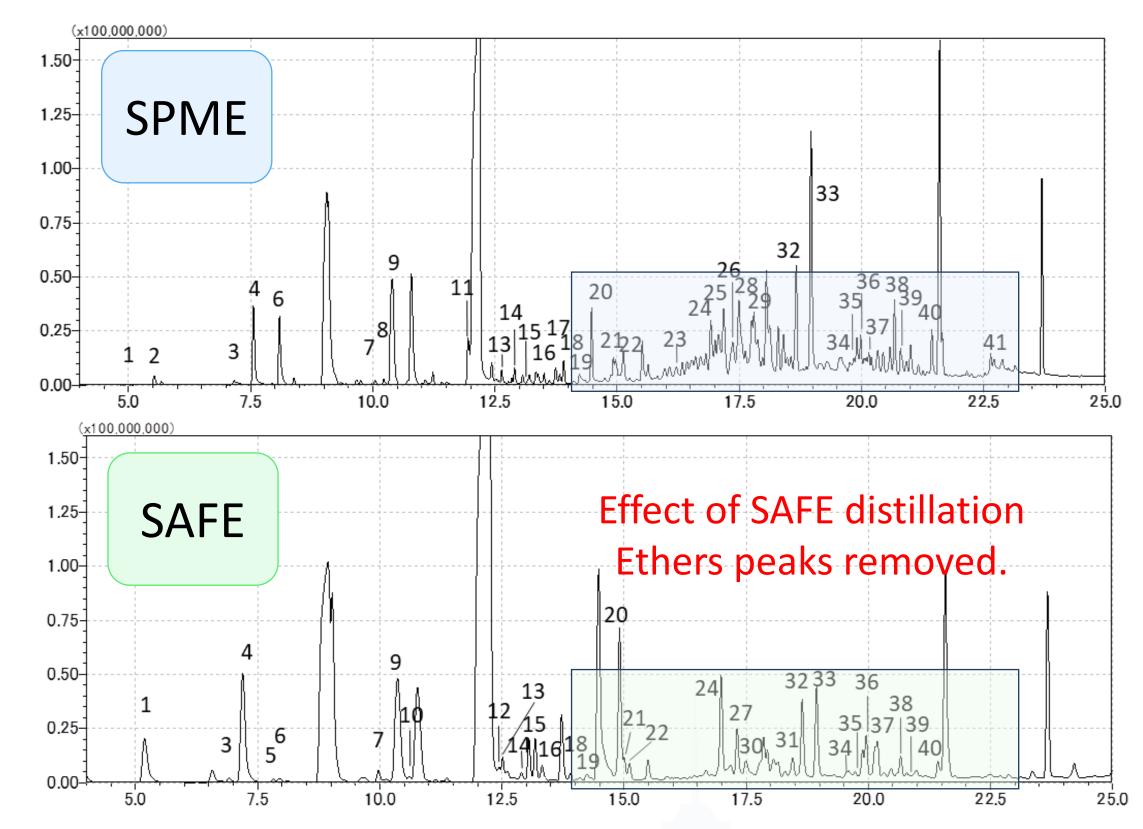
Sample: 10 g, Solvent: Dichloromethane Apparatus: AB25-I-2 (Kiriyama Glass Corp., Japan) Vacuum system: DS-A212Z, TRI-10-N25Y(Diavac Limited, Japan) Extract temp.: 30 °C, Extract time: 30 min. Concentrator: Kuderna-Danish (Kiriyama Glass Corp., Japan).

#### **Results and Discussion**

#### **Evaluation of SPME and SAFE Extracts**

No	o. Volatile compound	Main group	SPME	SAFE
2	hexanal	aldehyde	+	-
8	octanal	aldehyde	+	-
11	nonanal	aldehyde	+	-
18	B (E)-2-nonenal	aldehyde	+	+
17	hexadecane	alkane	+	-
23	b heptadecane	alkane	+	-
25	b hexadecyl nonyl ether	ether	+	-
26	5 nonyl tetradecyl ether	ether	+	-
28	dodecyl nonyl ether	ether	+	-
29	nonyl octacosyl ether	ether	+	-

#### **Comparison of total ion chromatograms**



Aldehydes Alkanes ethers

SPME

No.	Volatile compound	Main group	SPME	SAFE
4	1-butanol	alcohol	+	+
5	3-penten-2-ol	alcohol	-	+
9	2-(2-methoxy-1-methylethoxy)-1-propanol	alcohol	+	+
12	3-methoxy-3-methylbutanol	alcohol	-	+
15	1-(2-methoxy-1-methylethoxy)- 2-propanol	alcohol	+	+
16	2-ethyl- 1-hexanol	alcohol	+	+
19	2-ethyl-2-Hexen-1-ol	alcohol	+	+
20	2-(2-ethoxyethoxy)-ethanol	alcohol	+	+
21	6-methyl-1-octanol	alcohol	+	+
22	1-nonanol	alcohol	+	+
24	2-(2-butoxyethoxy)-ethanol	alcohol	+	+
27	1,1'-oxybis- 2-propanol	alcohol	-	+
30	2-(2-hydroxypropoxy)- 1-propanol	alcohol	-	+
33	1-dodecanol	alcohol	+	+
40	1-tetradecanol	alcohol	+	+

#### Conclusion

SPME showed a good extraction effect on aldehydes, linear alkanes, and ethers. As a result of the removal of high boiling compounds from the leather by SAFE, the TIC of SAFE extraction did not show peak aggregates as SPME.

### SAFE

Alcohols

