TRACE DETERMINATION OF TETRAHYDROCANNABINOL (THC) IN COSMETIC PRODUCTS BY STIR BAR SORPTIVE DISPERSIVE MICROEXTRACTION-LIQUID CHROMATOGRAPHY-TANDEM MASS SPECTROMETRY

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INTRODUCTION

- The use of **cannabidiol** (CBD) and **Cannabis Sativa L. extracts** has recently become a new trend as a cosmetic ingredients

- **THC** can be present in cosmetic products at trace levels coming from the raw materials or by isomerization of CBD

- The **aim** of this work is to develop a sensitive method to determine traces of **THC** in cosmetic products
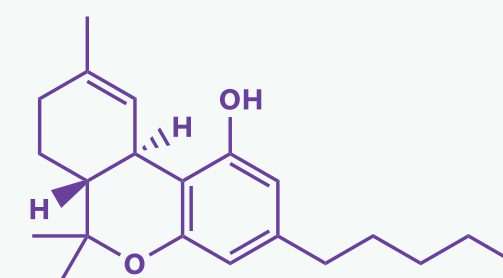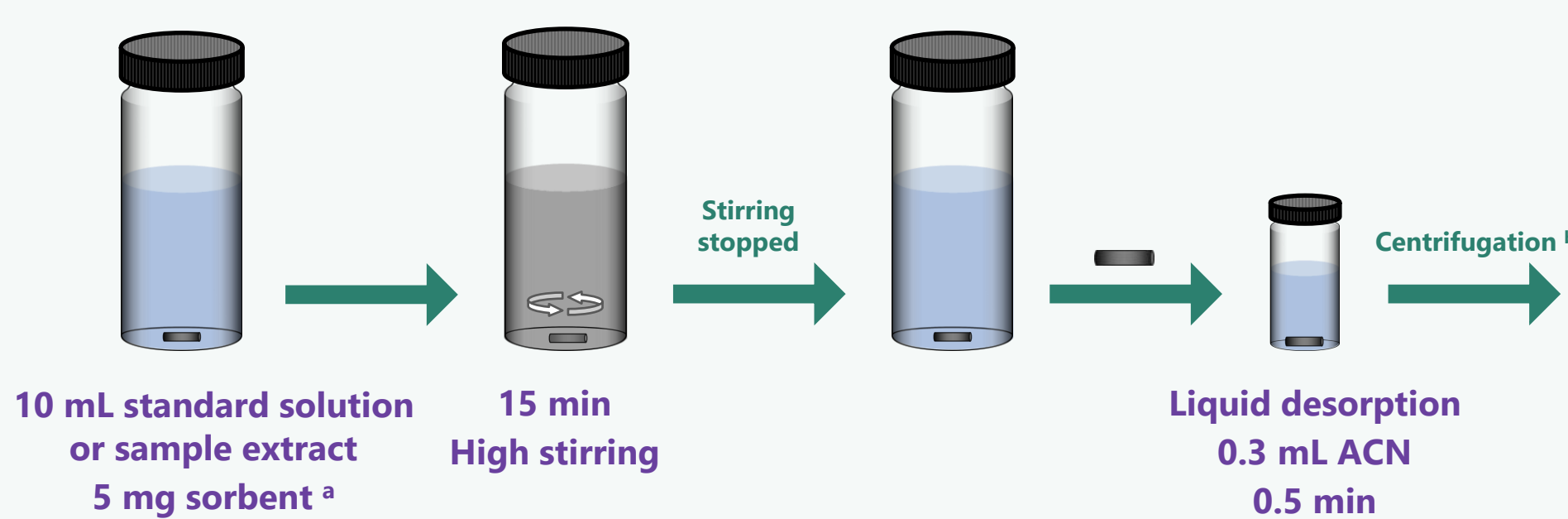
Δ^9-**Tetrahydrocannabinol**

- **Complex matrices** (as cosmetics) and the **low concentration levels** expected for THC make necessary a **sample preparation step** where the analyte is extracted and preconcentrated

- **Stir bar sorptive dispersive microextraction (SBSDME)** [1] was developed a few years ago by our group combining the principles of stir bar sorptive extraction (SBSE) and dispersive solid-phase extraction (DSPE). In this technique, a magnetic sorbent material, which is used as extraction phase, is dispersed by magnetic stirring and retrieved onto a neodymium stir bar surface. The analytes are then desorbed in a small amount of solvent and injected into LC-MS/MS

EXPERIMENTAL

10 mL standard solution or sample extract 5 mg sorbent a
→ **15 min High stirring** → **Stirring stopped** → **Liquid desorption 0.3 mL ACN 0.5 min** → **Centrifugation** b

LC-MS/MS
- **Injection volume:** 5 µL
- **Column temperature:** 35 ºC
- **Mobile phase:** MeOH (0.1% formic acid):H$_2$O (0.1% formic acid) (80:20)
- **Flow:** 0.4 mL min^{-1}
- **Column:** Zorbax SB-C18 (50 mm length, 4 mm I.D., 1.8 µm)
- **Gas temperature:** 310 ºC
- **Gas flow:** 12 L min^{-1}
- **Nebulizer gas pressure**: 35 psi
- **Acquisition mode:** ESI + (MRM)
- **Capillary voltage:** +6000 V

a CoFe$_2$O$_4$ MNPs embedded into Strata-X^{TM} RP polymer

b Retention of THC in polypropylene was observed. Centrifugation was carried out using a glass insert inside of a centrifuge microtube
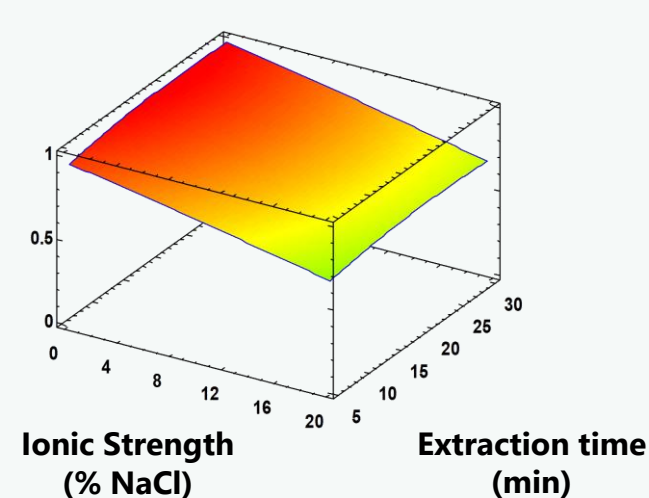
RESULTS AND DISCUSSION

Selection of the experimental variables for the extraction procedure by the Response Surface Methodology

Desirability
0.0 / 0.1 / 0.2 / 0.3 / 0.4 / 0.5 / 0.6 / 0.7 / 0.8 / 0.9 / 1.0
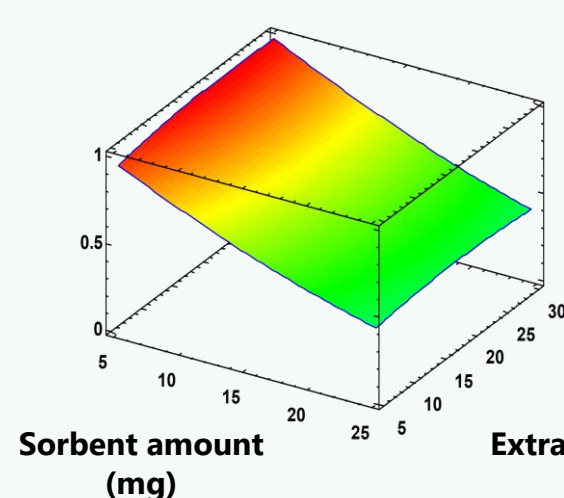
pH: 5.0
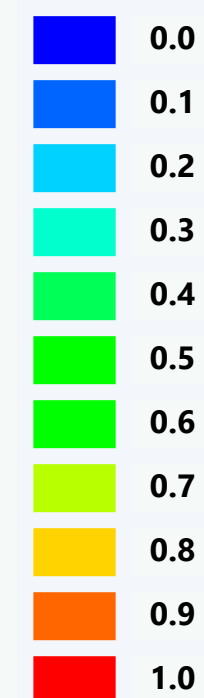**Ionic Strength:** 0% NaCl
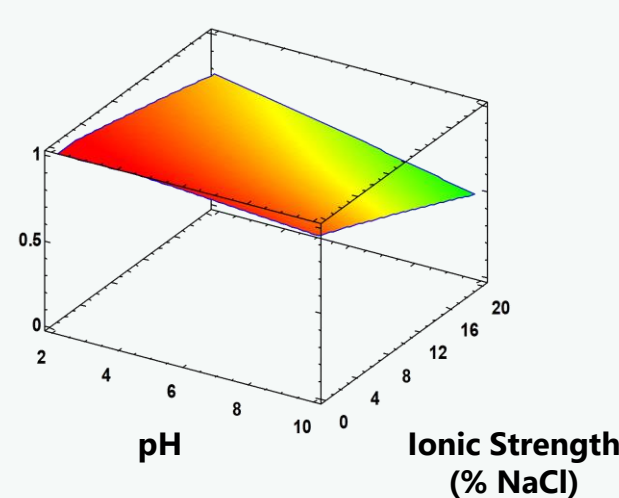
Sorbent amount: 5 mg
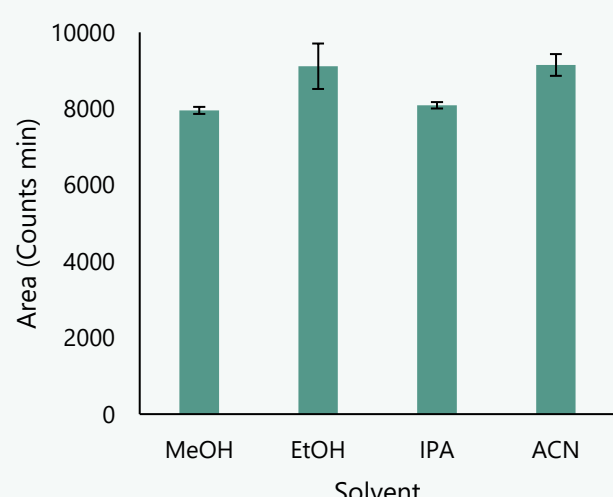**Extraction time:** 15 min

Sorbent amount: 5 mg
pH: 5.0

Optimization of the liquid desorption procedure

Selected solvent: ACN
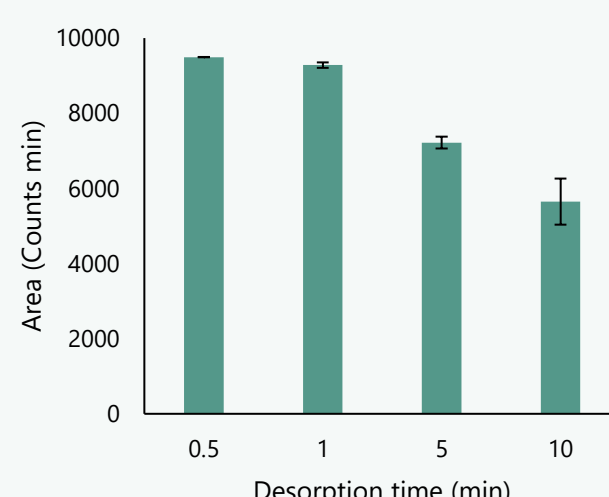
Selected desorption time: 0.5 min

Chromatogram

m/z: 315.2 → 193.1
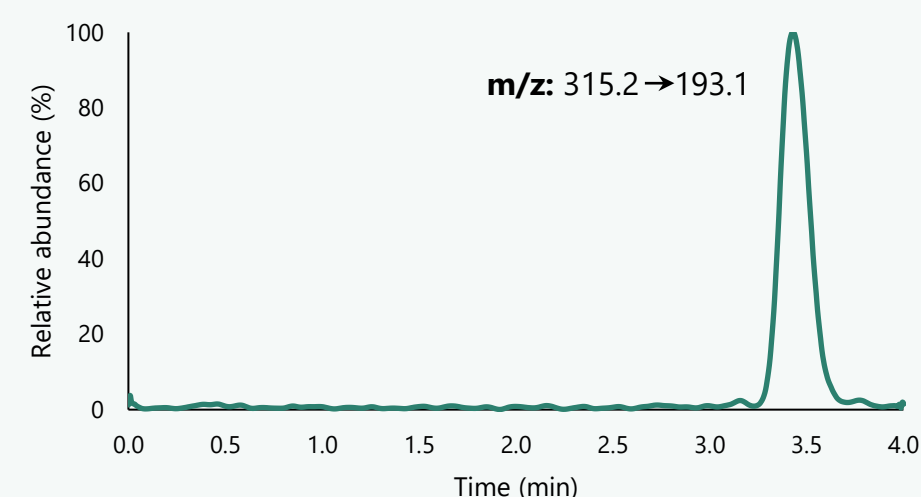
Chromatogram of a 500 ng L^{-1} standard solution obtained by the proposed method

- High level of **linearity**, that reached at least 10 ng mL^{-1}, was observed
- Low instrumental **limit of detection** (6.7 ng L^{-1}) and good **enrichment factor** (16) were achieved
- Experiments with sample extracts are still under study

CONCLUSIONS

- A new SBSDME-LC-MS/MS method has been developed for the **extraction and preconcentration** of trace levels of THC
- The proposed method constitutes a **simple**, **rapid** and **efficient** procedure for its application in **cosmetic samples**

[1] V. Vállez-Gomis, J. Grau, J.L. Benedé, D.L. Giokas, A. Chisvert, A. Salvador, Anal. Chim. Acta 1153 (2021) 338271