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Extraction of Phthalates from Textile Analyzing with GC-MS

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Introduction

In textiles, phthalates (plasticizers) are used to increase the flexibility of prints on for instance T-shirts. Some phthalates are associated with hormone disturbance (endocrine disruptive effects) and more and more phthalates are banned by the European Union. As our skin is continuously in direct contact with clothing, migration of chemical compounds from the fabric into the skin is likely. Therefore, it is important to investigate the presence (and concentrations) of phthalates in clothing. In addition, the migration of phthalates into artificial sweat is also of importance for a final risk assessment on phthalates in clothing. In this project a method is optimized for extracting phthalates from textile, by making use of a ultrasonic bath and reflux method with different extraction solvents.

Plan of Action

First Part:
- Constructing regression models by analyzing calibration standards, containing different concentrations of four phthalates: DIBP, DBP, BBP, DEHP
- Reproducing different extraction methods from the previous project groups with a (95% polyester, 5% elastane) T-shirt

Second Part:
- Optimizing the method with different extraction solvents: dichloromethane (DCM), ethyl acetate and artificial sweat

Materials & Method

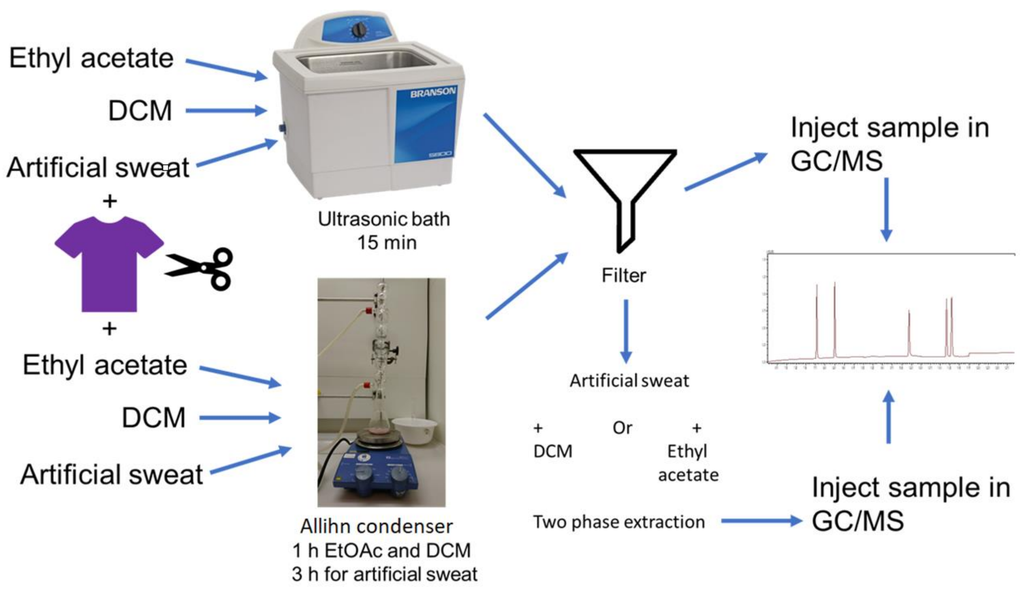

Figure 1: Scheme of the used methods

Used equipments:

- GC-MS: Shimadzu QP 2010
- Column: Agilent CP-sil 50m 0,25mm 0,25µm CB-MS (apolair)
- Auto-Injector: AOC-20i
- MS: Quadrupool (EI ionisatie)

Parameter	Value
Injection temperature	300 °C
Injection modus	Split
Split ratio	1:20
Injection volume	1,0 µL
Column flow	0.88 mL/min
Temperature programme	1. 180 °C - hold for 3 min 2. 255 °C – with 25 °C/min 3. 300 °C – with 50 °C/min Run time: 13 min

Figure 2: GC MS setup and settings

Results

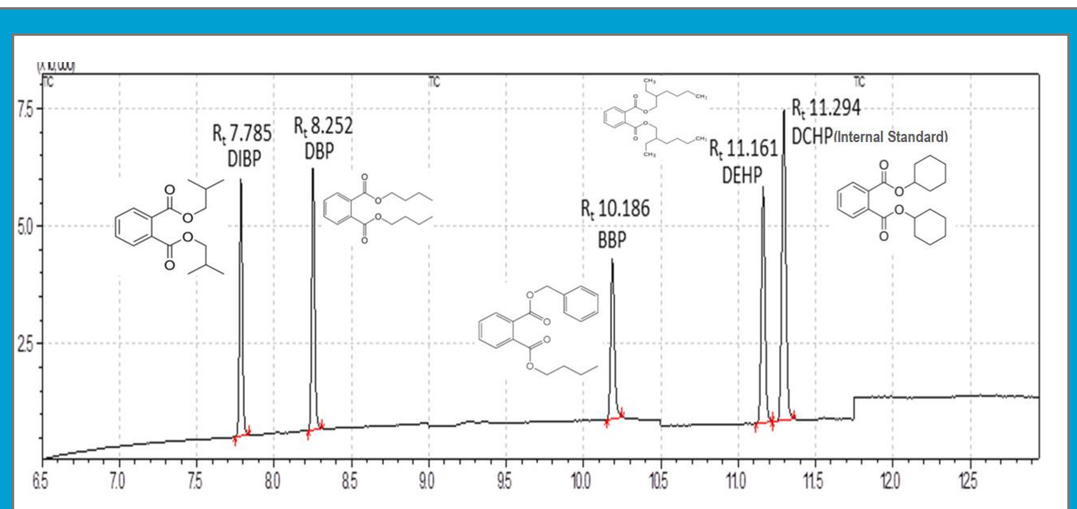

Figure 3: Seperation of the standards DBP, DBP, BBP, DEHP and DCHP (internal standard)

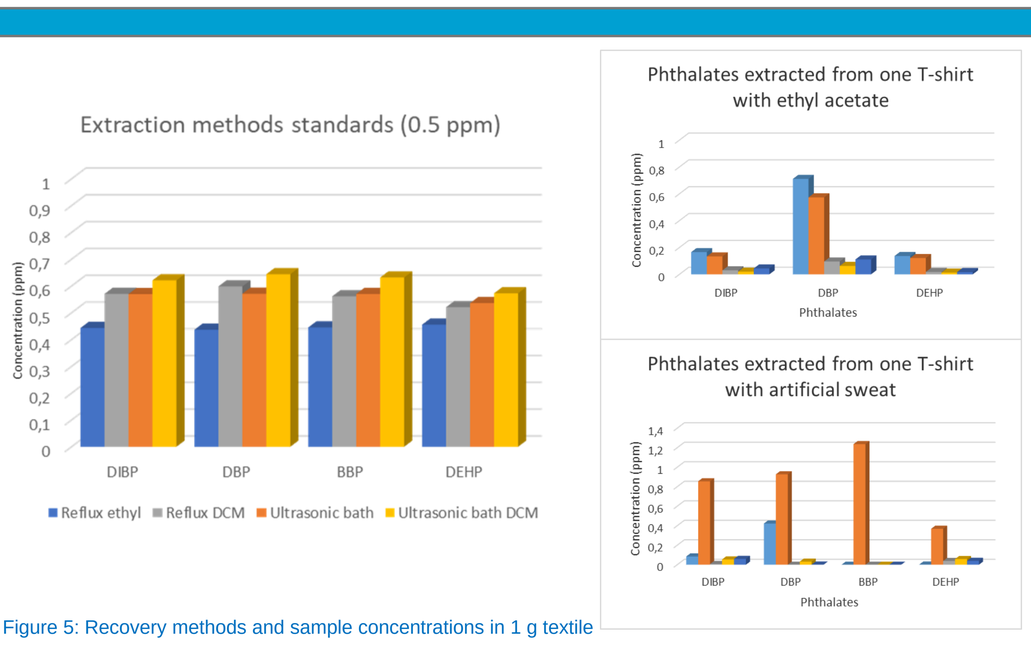

Figure 5: Recovery methods and sample concentrations in 1 g textile

Regression models

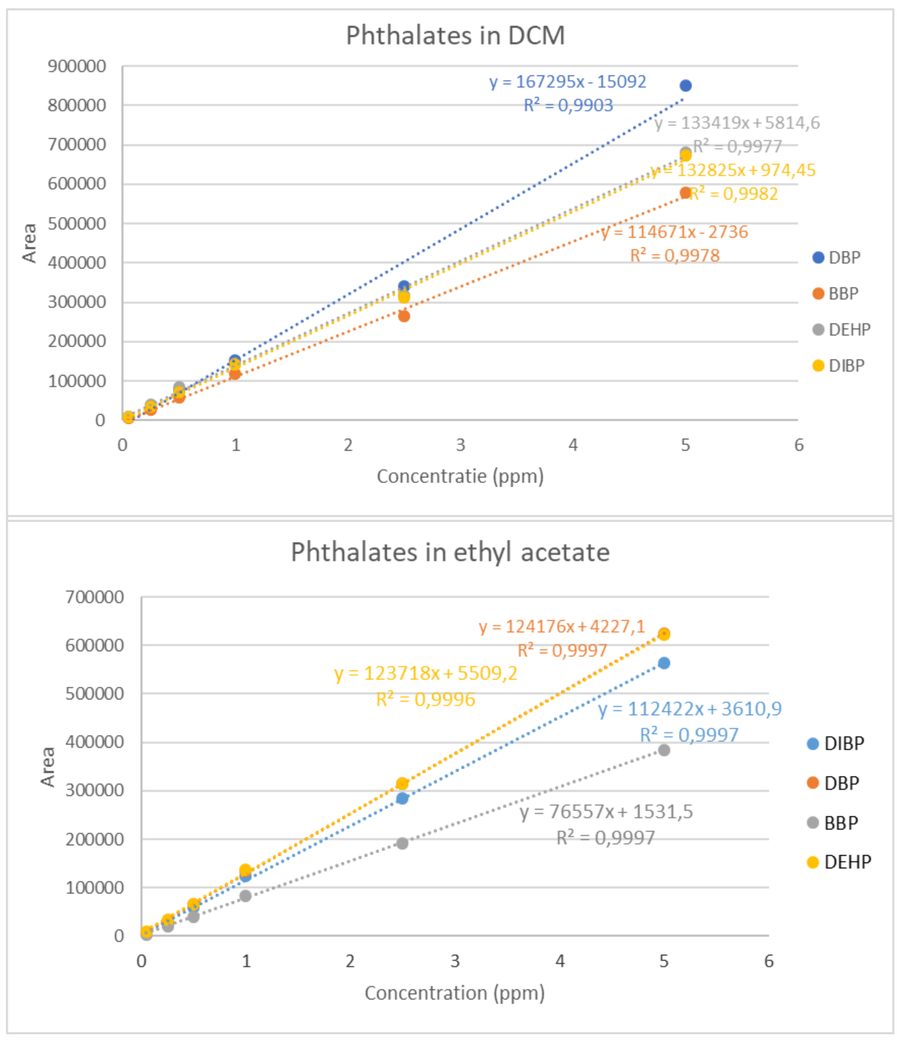

Figure 4: Regression models of phthalates with two different solvents

Conclusion

Phthalates are detected in the T-shirt and a method has been optimized for the extraction of four phthalates (DIBP, DBP, BBP, DEHP). The preferred method in this project is the ultrasonic bath with ethyl acetate as solvent. Also, the artificial sweat monster works both for DCM and ethyl acetate. The sample preparation with the ultrasonic bath method is shorter and more samples can be processed. Furthermore, ethyl acetate has more reliable results than DCM and is less harmful to health and environment.

Future Work

The chosen method can be validated with the GC-MS. To have more statistic certainity about which solvent gives more reliable results, more extractions with DCM and ultrasonic bath could be performed.
Furthermore, the sensitivity of the GC-MS method can be improved by changing parameters such as split and splitless injection. Also, the phthalates elute at 7 minutes which could be faster.