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Detection of packaging adhesives

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Introduction

Multilayer packaging of food and pharmaceutical products often contains adhesion promoters based on epoxysilanes. They enable the stable bonding of inorganic substrates and organic materials, for example in the case of aluminium tubes with plastic coating. A prominent representative is the substance 3-Glycidyloxypropyltrimethylsilane (GLYMO).

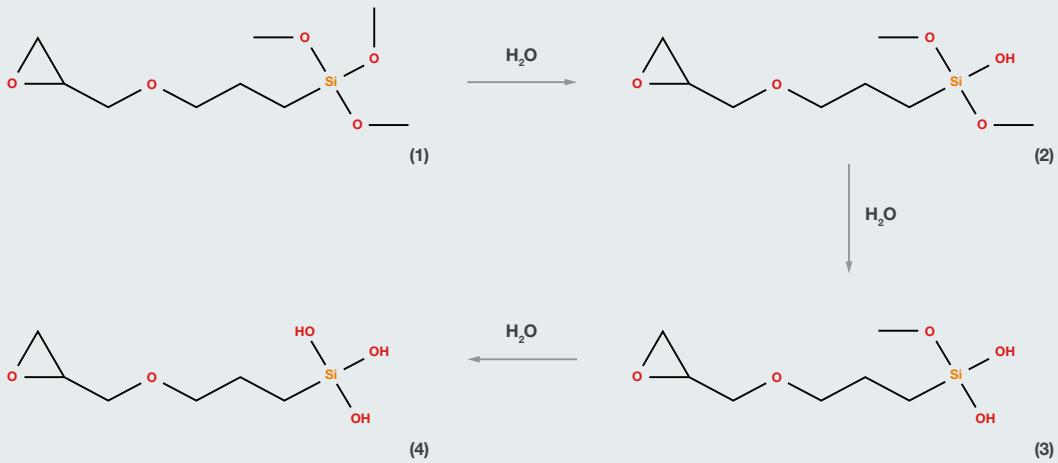
Main part

GLYMO is used especially for composites that are exposed to high temperatures. For example, food packaging intended for sterilization or hot filling. Among others because of its frequent use, the European Food Safety Authority (EFSA) carried out a safety assessment of the substance. In the document published in 2017, GLYMO and certain reaction products are classified as potentially genotoxic[1]. Currently, the use of GLYMO as an adhesion promoter is still permitted but subject to the following conditions [1, 2]:

- As an additive for the treatment of glass fibres embedded in plastics with low diffusivity. These include polyethylene terephthalate, polycarbonate, polybutylene terephthalate, thermosetting polyesters and epoxy bisphenol vinyl esters.
- The corresponding packaging is not allowed to be used for the filling of fat-based products.
- In the case of water-based products, no GLYMO must be detectable, the burden of proof being on the manufacturer. Specifically, the following limit values must be observed [1, 3]:
  - GLYMO (treated glass fibre): 10 µg/Kg
  - reaction products GLYMO (treated glass fibre): 60 µg/Kg

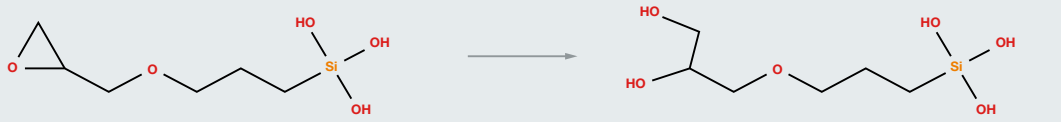
UPLC-HRMS methods are suitable for detecting the absence of GLYMO in aqueous products. The substance decomposes in aqueous media towards its metabolites. Therefore, the hydrolysis products of **graphs 1 and 2** should also be taken into account in the analysis.

Graph 1:
Stepwise hydrolysis of the three trimethoxy groups from GLYMO 1 with cleavage of methanol to trihydroxysilane 4



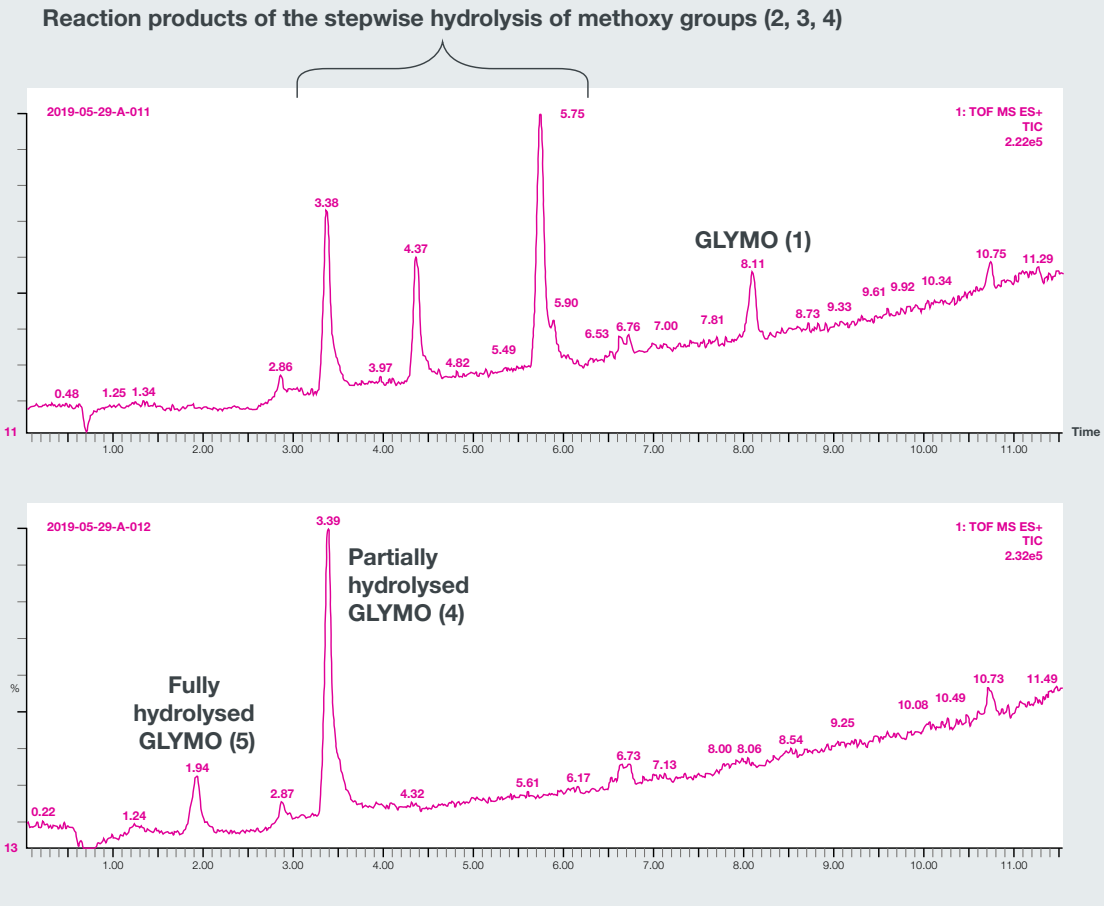
The reaction steps are strongly pH-dependent. Complete hydrolysis in neutral medium at room temperature takes about 24 hours. The starting compound GLYMO can be detected for about eight hours. The last reaction step, i.e. the opening of the epoxy ring to trihydroxyglycol, is much slower. A complete reaction can only be expected after a storage period of more than one month. In acidic solution the reaction rates are higher. Thus, complete hydrolysis is completed after about one hour and water addition to the epoxy group after about 24 hours. In summary, in aqueous media mainly the degradation products 4 and 5 are to be expected. Both compounds can be reliably detected by UPLC-HRMS (**Graph 3**).

Graph 2:
Addition of water to the epoxy ring and formation of a glycol group



(4) (5)

Graph 3:
UPLC-HRMS chromatograms of partially hydrolysed aqueous GLYMO solutions



Conclusion

The risk assessment of packaging materials in contact with food or pharmaceuticals is a complex process. Extensive knowledge of starting materials, additives and manufacturing processes is essential. The example of the adhesion promoter GLYMO shows that analytical know-how and procedures beyond routine analysis are also required. This is typical for studies on extractables and leachables. The identification and quantification of the corresponding substances often requires sophisticated procedures such as high-resolution mass spectrometry. It is therefore recommended to develop an individual analytical concept for the respective packaging before the product launch. □

References

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2. *https://www.labelpack.de/mit-epoxysilan-freien-klebstoffen-neuer-gesetzgebung-zuvorkommen/*
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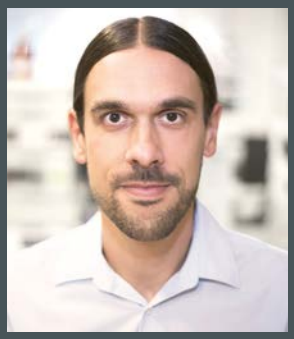
Author



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