

Determination of the Migration of Acetaldehyde from PET Bottles into Non-carbonated and Carbonated Mineral Water

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Introduction

Poly(ethylene terephthalate) (PET) bottles are widely used for beverages especially for mineral water. For this application, high levels on the neutrality of odour and taste of the packaging materials are required. For mineral water in PET bottles, the migration of acetaldehyde is of special interest, because acetaldehyde is detectable in low concentrations in mineral water as a fruity off-flavour. Acetaldehyde is a degradation product of the PET polymer generated during preform production. After cooling down, acetaldehyde is trapped in the PET bottle wall and migrate into the mineral water after filling and storage. The proposed acetaldehyde formation mechanism is given in Figure 1. The taste threshold limit of acetaldehyde in mineral water is reported to be in the range of 10 to 20 ppb [1].

For the quality control of the mineral water, a suitable method for quantitative determination of acetaldehyde in mineral water should be available. The detection limits should be far below the odour and taste thresholds of acetaldehyde in mineral water. Several methods are given in the literature [2]. However, in most cases derivatisation reagents are used which transforms acetaldehyde into a higher molecular weight compound. Sample preparation pose the risk of a loss of the highly volatile acetaldehyde. In addition derivatisation reagents are difficult to use in a automated systems for routine control at the mineral water filling companies.

Aim of this study was therefore the development of a sensitive, fast and automated method for the quantitative determination of acetaldehyde in mineral water without derivatisation of acetaldehyde.

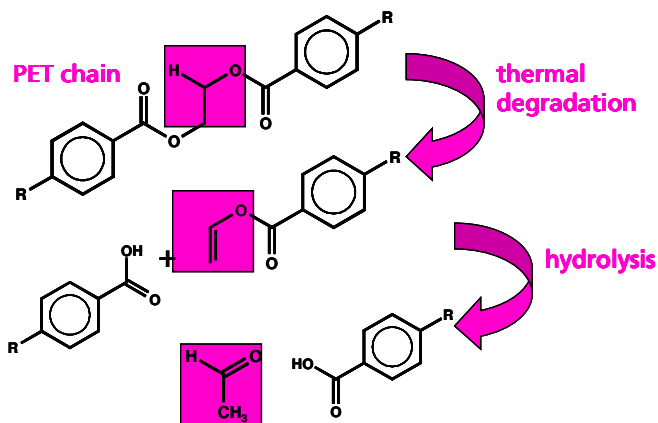


Figure 1: Proposed mechanism of the formation of acetaldehyde from PET polymer during preform production

Purge an Trap Gas Chromatography Method

Acetaldehyde was determined quantitatively in mineral water by purge and trap gas chromatography using the following procedure: The mineral water was cooled down to 1 °C and stored in minimum for 1 d in order to minimize the loss of acetaldehyde during opening of the bottle. 0.8 ml of a 20% (w/v) solution of NaOH were given into a 22 ml headspace vial. Subsequently 10.0 ml of cold mineral water were added. The vial was immediately sealed and analysed by purge and trap headspace gas chromatography. Gas chromatograph: Carlo Erba 5300 Mega, column: combination of DB VRX and DB 624, each 30 m long, 0.32 mm i.D., 1.8 µm film thickness, pressure: 120 kPa helium, temperature programme: 35 °C (6 min), rate 10 °C min⁻¹ up to 90 °C, rate 20 °C min⁻¹ up to 260 °C (10 min). Purge and trap autosampler: IMT PTA 3000, sample temperature: 40 °C, purge time: 20 min, purge flow: 20 ml min⁻¹, trap temperature: -120 °C, desorption temperature: 200 °C, desorption time: 10 min, water trap: -15 °C, trap: Tenax®. Calibration was achieved by external calibration with acetaldehyde standard in distilled water between 4.35 ppb and 43.5 ppb (linearity R²: 0.9998). The detection limit was determined to be 0.2 ppb.

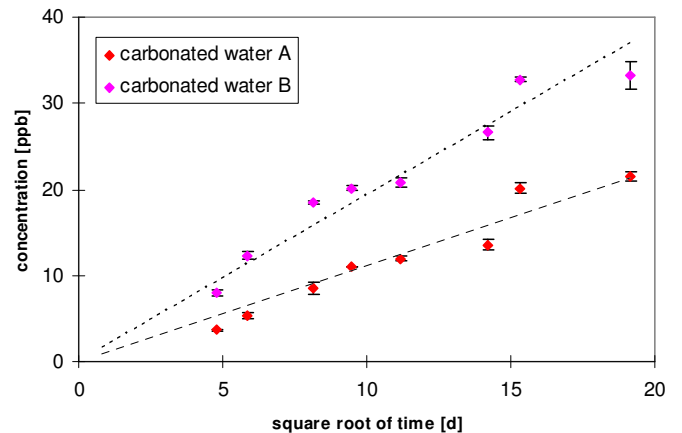


Figure 2: Migration kinetics of acetaldehyde from refillable PET bottles into carbonated mineral water at 23 °C determined over the shelf life of 365 d

Conclusions

The purge and trap method developed in this study was found to be a suitable method for routine control of acetaldehyde in mineral water. The detection limit is a factor of 50 to 100 below the odour and taste thresholds. Migration kinetics for mineral water show, that the migration of acetaldehyde in carbonated mineral water follows Fickian laws for diffusion (Figure 2), resulting in a linear correlation of the migration with the square root of storage time. On the other hand, for non-carbonated mineral water, acetaldehyde was not stable in the mineral water matrix resulting in a significant lower concentration of acetaldehyde in the mineral water as expected from migration theory [3]. Therefore PET bottles for non-carbonated mineral water might have a higher amount of acetaldehyde in the PET bottle wall. On the other hand, for carbonated mineral water the bottle wall concentration of acetaldehyde should be minimized. These results show, that from the long-term monitoring of the acetaldehyde migration the final concentration of acetaldehyde in the mineral water is difficult to estimate because the mineral water itself, especially the concentration of carbon dioxide, plays a crucial role for the concentration of acetaldehyde in the mineral water over the shelf life of the mineral water bottles. In addition, carbon dioxide permeate through the bottle wall, which might change the stability of acetaldehyde during storage. Therefore, the prediction from the bottle wall concentration of acetaldehyde using migration models might fail and can only be used for a "worst case" estimation.

As a conclusion, the packaging companies and mineral water fillers have to test their PET bottles in regular terms in order to evaluate if the acetaldehyde odour and taste thresholds are exceeded after a certain storage time. The purge and trap method developed in this study is a suitable method for such a routine determination of the migration of acetaldehyde into mineral water.

References

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