Moisture and Fragrance Release from Hydrogel Air Fresheners

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ABSTRACT SUMMARY

Moisture and fragrance release was measured from κ-carrageenan hydrogel beads containing a model fragrance using ThermoGravimetric Analysis (TGA) coupled with GC-MS. This approach was used for the evaluation of potential fragrance mixtures in hydrogel air fresheners. It can determine if the fragrance character/composition released from the hydrogel differs from the original fragrance and if it changes during drying. Knowledge on the release of complex fragrances in hydrogels can aid in designing fragrances that perform well and predictably in air freshener applications.

INTRODUCTION

Hydrogel air fresheners have been available commercially for many years and generally contain high levels of water (> 90% by weight), a gellant—often carrageenan—and fragrance. These air fresheners dehydrate, shrink and release fragrance when exposed to typical environmental conditions. Generally, a 200-300 gram product will last around 30 days, albeit with diminishing fragrance intensity [1]. As water evaporates, it tends to generate a dry, occlusive zone on the surface that hinders the further evaporation of volatiles; leaving fragrance trapped in the core of the air freshener [2]. Furthermore, the composition of the fragrance can change over time. Thus, there is a need to design so-called linear fragrances, with minimal changes to its character [3]. Additionally, an ideal fragrance has minimal interaction with the air freshener carrier, resulting in a similar fragrance character compared to the liquid fragrance. This study aims to understand the fundamentals of mass transfer in hydrogel air fresheners. Rather than working with actual—often cone-shaped—air fresheners, we scaled the problem down to a small spherical model system that allows for the rapid determination of the fundamental mass transfer properties under carefully controlled conditions in TGA. First, equilibrium moisture sorption isotherms (MSI) of κ-carrageenan hydrogel beads (Figure 1) were determined.

EXPERIMENTAL METHODS

Materials: κ-Carrageenan (Ticaloid® 710 H, TIC Gums) was used to prepare carrageenan solutions (2.85 wt%). A model fragrance consisting of equal weight parts of linalool, hexyl acetate, benzyl acetate and isoamyl acetate was optionally added to the carrageenan solution (1 wt%). Small spheres of hydrogel (diameter of several millimeters) were made by prilling hot carrageenan solution in cold NEOBEE® (medium chain triglycerides). Beads were washed in 1% SDS solution and rinsed to remove surface oil.
**MSI determination:** unscented spheres were equilibrated over saturated salt solutions (~23, 53 and 75% RH) at 25°C. Moisture content was measured gravimetrically using a vacuum oven (~70°C, 100 mmHg, 16 hours).

**Moisture release:** Thermo Gravimetric Analysis (TGA) was conducted (Q50 TGA, TA Instruments) at 25°C for 2 hours. Roundness and diameter of spheres were measured before TGA using image analysis.

**Fragrance release:** A TGA-GC-MS (TG 209 F1 Libra from Netzsch-Agilent 7890B GC system 5977A MS) was used for fragrance release analysis. Spheres were exposed to 40°C for 45 min using dry helium. Abundance of prominent ions of the 4 compounds was measured by MS after baseline subtraction.

**RESULTS AND DISCUSSION**

**Moisture transfer:** The moisture sorption isotherm data was fitted with the well-known Guggenheim-Anderson-deBoer equation (GAB) yielding: \( C_1=2.082, k=0.00198, m_0=0.277 \).

The apparent moisture diffusion coefficient \( D_{app} \) (m²/s) was calculated from TGA drying data by applying the equation [4]:

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\frac{M_t}{M_\infty} = 6 \left( \frac{D_{app}}{r^2} \right)^{1/2} \left( \pi^{-1/2} + 2 \sum_{n=1}^{\infty} ierfc \left( \frac{nr}{\sqrt{D_{app}t}} \right) \right) - 3 \frac{D_{app}}{r^2} \]

\( D_{app} \) was determined as a function of moisture content \( X \) in range of ~12.5 to 22 g water/100 g solids. The diffusion coefficient increased with increasing moisture content and yielded the following relationship: \( D_{app} = 5 \times 10^{-13} e^{15.955X} \).

**Fragrance Transfer:** Combined moisture and fragrance release from a scented hydrogel sphere is presented in Figure 2a. This data confirms the rapid decline of this model fragrance release with falling moisture content. Figure 2b presents the compositional fragrance release from the hydrogel compared to data generated on the liquid ‘gold-standard’ measured under similar conditions in TGA-(GC)-MS (at t=10 min). The relative ion abundance for each compound is represented by the area percentage of the individual slices. It shows that the headspace composition of the hydrogel was considerably different from the liquid. The fragrance released mostly linear with time. A more non-linear release is expected from future experiments on complex mixtures with more dissimilar compounds.

**CONCLUSION/FUTURE WORK**

The approach presented here allows for the rapid determination of moisture and fragrance release from model hydrogel air fresheners under carefully controlled conditions. We found that the character of the fictional model fragrance was affected by the κ-carrageenan gel, but that the fragrance composition was reasonably linear over time. In future work we aim to simulate the mass transfer of moisture and fragrance in hydrogel air fresheners using the results obtained in this study. This rapid and standardized procedure can be used to optimize fragrances for hydrogel air fresheners—or possibly—other applications; ultimately aiming to improve, extend and linearize fragrance release in commercial air care products.

**REFERENCES**