Biolin Scientific
[Progress Together]

Q-Sense | [Application Note] 18

Analysing vapor uptake & release with QCM-D

This application note illustrates how Q-Sense Quartz Crystal Microbalance with Dissipation (QCM-D) can be used to characterize changes in film properties exposed to moist air. Measurements are easily conducted in the Q-Sense Humidity Module - the relative humidity can rapidly be changed and controlled without using other equipment. Humidity measurements are of importance in a wide range of applications, for example pulp and paper research, drug encapsulation for storage and delivery, and moisture absorption in polymers e.g. photoresists. Here, an analysis on how water uptake affects properties and thickness of different starch films, is presented.

Introduction

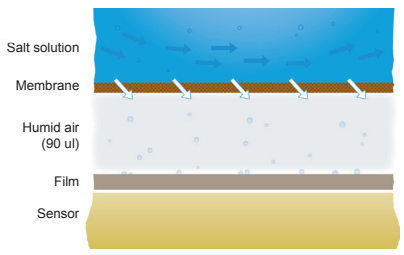
QCM-D is a surface sensitive technique, which provides real-time information on structure, thickness, swelling and mass of your sample. For rapid and easy analysis of vapor uptake and release from thin films, Q-Sense has developed a specific measurement chamber - the Humidity Module. Simultaneous measurements of both mass and structural properties in this module give a thorough understanding of how the substrate behaves under different humidity conditions.

The relative humidity can rapidly be changed and controlled without using other equipment. The humidity is controlled by salt solution in the flow system. The technical design includes a GORE™ membrane that separates a small air pocket above the sensor from liquid flow, see figure 1.

Approach

Starch is an important ingredient in the paper industry and the effect of humidity on paper strength and quality is a central question. To conceptually illustrate how the humidity QCM-D setup can be used, moisture uptake in two different kinds of starch were evaluated at different humidities. Native potato starch (NPS) and glycerol doped starch (NPS/Glycerol) were used in these experiments. To initiate the humidity QCM-D measurements, starch solutions were spin coated at 2000 and 4000 rpm for 20 seconds under normal laboratory conditions onto Q-Sense gold coated sensors. The coated sensors were analysed and all measurements were started at 11% air humidity. The humidity was then increased incrementally, as illustrated in table 1. To achieve 0% humidity the chamber can be filled with nitrogen gas.

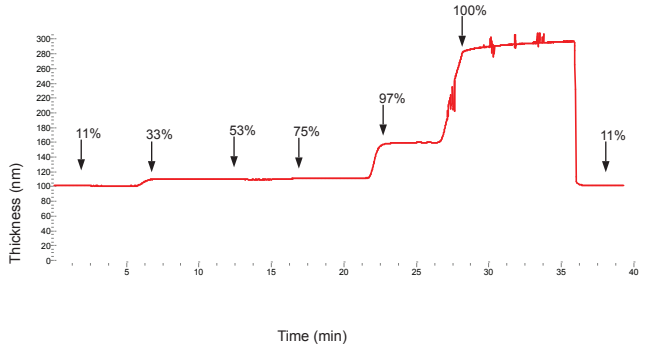
The Q-Sense humidity module



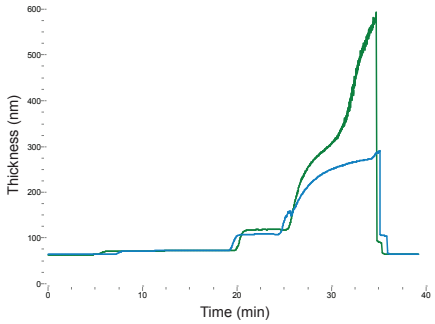
[Figure 1]: The Q-Sense Humidity Module in cross-section.

Solution	Relative humidity
LiCl	11%
$MgCl_2$	33%
$MgNO_3$	53%
NaCl	75%
K_2SO_4	97%
H_2O	100%

[Table 1]: Relative humidity table at 23°C



[Figure 2]: Thickness of NPS as a function of air humidity.



[Figure 3]: Comparison of the swelling of a NPS/Glycerol (green) and NPS (blue).

Results & discussion

The obtained raw data were quantified as starch film thickness and viscoelasticity using QTools[1] analysis software , see figure 2. The analysis shows that thickness and viscoelasticity[2] of starch increase with the humidity in a non-linear fashion.

Further, a relatively high humidity level (above 75%) was required before a substantial swelling of the films could be observed, see figure 2. This might be due to starch molecules coupling to each other, rather than to water, meaning that water molecules start to adsorb from the top of the film at lower humidity levels. As the humidity level is increased resulting in saturation of the surface and air, the water molecules are forced down into the starch film, leading to a drastic swelling. This effect was seen in both NPS and NPS/Glycerol films, see figure 3.

Measurements comparing NPS with NPS/Glycerol shows that starch with glycerol binds considerably more water. This behavior is expected since glycerol fictionalization of starch makes it more hydrophilic. The NPS/Glycerol film swells ten times its original thickness, from 60 to about 600 nm, see figure 3.

Notably, the starch films also collapse back to original thickness in a matter of seconds, when the humidity is changed back from 100% to 11%, see figure 2, 3. Measurements at 100% humidity are inherently unstable due to saturation effects and droplets forming on the sensor, but here included to illustrate analysis possibilities. For all other humidities reproducibility is very high.

Conclusion

The unique design of the Q-Sense Humidity Module enables investigations of your sample at different humidities in a controlled and simple way, without using other equipment. Both thickness and viscoelastic properties can be analyzed simultaneously in real-time. By analyzing film thickness and structural properties at different humidities, it was found that the starch films showed a non-linear relationship between air humidity and water uptake. Further, the chemical composition of the starch affects its ability to bind water. The reproducibility of these measurements was high.

References:

Measurements by Q-Sense/Biolin Scientific in collaboration with the Department of Polymeric Materials, Chalmers University of Technology, Sweden.

[1)] QTools is an analysis software included in your Q-Sense QCM-D system; it is used for viscoelastic modeling of QCM-D data.

[2)] Viscoelastic properties of the film are possible to measure thanks to Q-Sense instruments' unique simultaneous measurement of frequency and dissipation.

Biolin Scientific
[Progress Together]

E-mail:info@biolinscientific.com
biolinscientific.com