Measurement of water sorption isotherms of materials using combined real-time humidity and microwave resonance measurements

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Background

Material surface and bulk moisture content affect:
- Mechanical properties – numerous materials such as concrete, paper, powders processing
- Growth/inhibition of bacteria – foods, agriculture, conservation
- Chemical activity – pharmaceutical shelf life and potency, corrosion
- Thermal properties – insulations, combustion
- …and much else.

A range of techniques exist for measuring the water/moisture content of materials but the majority are time consuming/destructive.

Certified reference materials (CRMs) exist for water content which can be used to calibrate/check moisture measurement instrumentation.

Water Sorption Measurement

Water sorption characteristics are determined for many materials normally by studying weight changes after a sample has been exposed to salt solutions of established relative humidities (e.g. COST 90 procedure).

An established technique (dynamic vapour sorption (DVS)) uses a microbalance and controlled gas flow to measure water sorption.

Measurement Summary

The use of a microwave resonance technique has been adapted to provide non-destructive, real-time measurements of moisture transients when the material under test is subjected to flowing air of well-defined humidities.

Initial exploratory work had shown that the sample holder used with the instrument could be adapted to allow flow of air through the test material.

In this project the technique was progressed, refined and used to investigate the water sorption characteristics of materials.

Measurement Principle

The instrument operates in the frequency region between 2 GHz and 3 GHz, and detects shifts in resonant frequency of the order of a megahertz when the microwave field is perturbed by water within a sample.

Progress

Achieving minimal background water content contribution of the sample holder whilst maintaining functionality was an evolutionary process.

Conclusions

The microwave resonance technique has demonstrated to provide the following advances in water sorption measurement:

1. Much faster equilibration times - typically 100 times faster than traditional experimental approaches (COST 90) and similar to those of DVS technique
2. High precision and sensitivity - sample sizes less than 15 mg can be readily studied.
3. Full automation - removes labour intensive nature of sorption measurements.

BCR-302 CRM Water Content Measurements

BCR-302 is a microcrystalline cellulose with certified water content values when exposed to specific humidities.

Water Sorption Isotherm Measurement with Microwave Instrument

10% relative humidity changes were applied in the range 15%rh to 85%rh at four hour intervals.

Figure 1. Study of moisture sorption (DVS) (Source: Surface Measurement Systems)

The water content of a material can be plotted against its water activity or the relative humidity it was exposed to producing a sorption isotherm. Hysteresis is often observed due to different adsorption/desorption characteristics of the material.

Figure 2. An example of a typical sorption isotherm (Source: http://www.fst.ohio-state.edu/olympiad/Laboratories/Olymp/Lab%201_WaterActivity.htm)

Changing from setup b) to c) allowed analysis of smaller sample sizes and greatly reduced moisture equilibration time following humidity changes. Changing to hydrophobic materials used in the fittings connecting the flowing air to the container, c) to d), greatly reduced the background effect that the container had on material moisture measurements.

Figure 3. Schematic diagram of microwave resonance moisture analyser

Figure 4. Effect of inserting moist material into resonator on microwave signal output (Source: Surface Measurement Systems)

Figure 5. Evolution from standard container to current design

Figure 6. Effect of improving container design and moisture absorption characteristics of foam filter

Figure 7. Comparison of water content measurement through evolved vapour analysis as compared with certified values determined through the COST 90 procedure

Figure 8. Microwave moisture value of BCR-302 reference material with stepped RH profile

Figure 9. Typical hysteresis shown by the BCR-302 material