Phase Transformation Temperature in Calcium Sulfate Hemihydrate Measured by Vapor Sorption Analysis

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ABSTRACT

Vapor sorption (VS) can be used to distinguish structural characteristics of powdered materials. Calcium sulfate hemihydrate ($CaSO_4 \cdot 0.5H_20$) can have free anhydrite CaSO4 (III) molecules that will adsorb increased amounts of moisture below a given temperature. The isohume and temperature programming capabilities of the Q5000 SA make it possible to observe this conversion in calcium sulfate materials.

INTRODUCTION

Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) are commonly used to measure the dehydration of gypsum or calcium sulfate dihydrate ($CaSO_4 \cdot 2H_20$) (1,2). Vapor Sorption (VS) instruments are designed to measure gravimetrically the effect of moisture adsorption and desorption, which can lead to hydration or dehydration of select materials. The tendency of a material to absorb water upon exposure to a humid environment can provide important information related to the structure of that material. Formulations of calcium sulfate hemihydrate can have free anhydrite $CaSO_4$ (III) molecules that will adsorb increased amounts of moisture below a given temperature (3). This increased moisture uptake leads to the phase transformation of $CaSO_4$ (III) into calcium sulfate dihydrate, which is not recommended during product manufacturing processes. The Q5000 SA was used to aid in the characterization of a stucco material.

EXPERIMENTAL and RESULTS

The stucco used was taken from freshly opened containers and exposed to ambient conditions only for a minimal time period (5 min). Reduced equilibration time is required for smaller sample masses and it is recommended to use between 1-10 mg samples for VS experiments. Two separate samples of the same stucco were investigated to measure sample size effects. The larger sample (green) and the smaller sample (blue) weighed 12.18 mg and 1.64 mg respectively. Figure 1 shows the results of a dynamic temperature ramp experiment, with the humidity maintained at 50 RH%. The temperature of the furnace was first equilibrated at 85 °C and held for 60 min, after which it was linearly decreased at a rate of 0.1 °C/min to 25 °C. Results from both experiments show that the stucco sample began to uptake moisture at a higher rate after about 550 min (Figure 1), at which time the furnace temperature reached approximately 36 °C. From the similarity in the two curves it was determined that sample size does not affect the kinetics

required to convert the $CaSO_4$ (III) phase. The shift in weight signal observed at 75 °C in Figure 1 was due to the activation of the external water circulation system required to cool the thermoelectric heating elements.

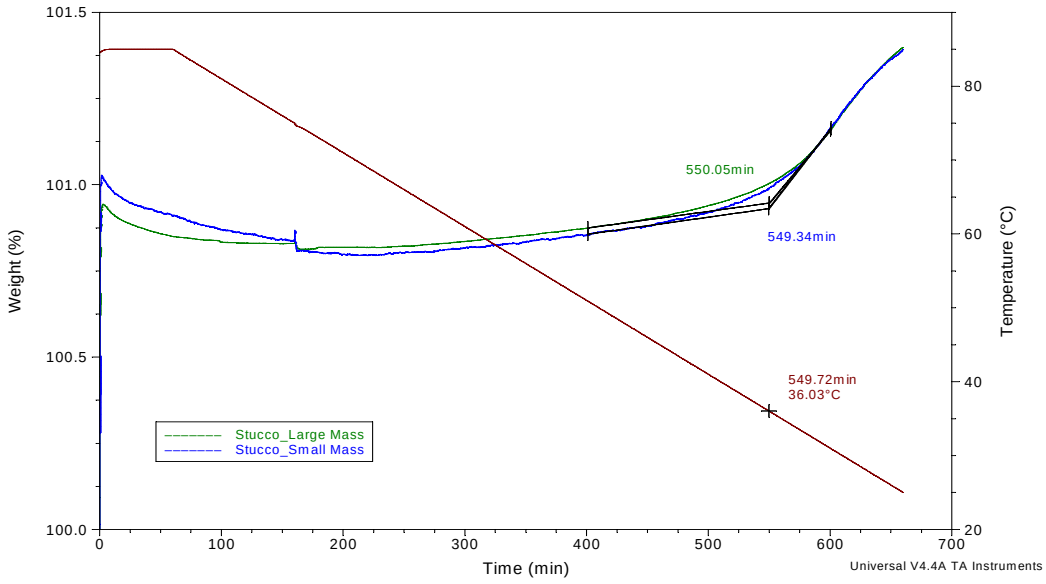

Figure 1. Sample mass effect of an isohume temperature ramp VS plot.

CONCLUSIONS

Vapor sorption is an important analytical tool for characterization of powdered materials. Dynamic temperature isohume experiments were used to investigate the temperature of known physical changes in stucco formulations. The results indicate that this stucco material should be kept above 36 °C at 50 % RH to prevent the conversion of the $CaSO_4$ (III) phase.

REFERENCES

1. TGA Characterization of Gypsum in Stucco, TA Instruments, Thermal Solutions (TS 29)

2. Determination of Calcium Sulfate Hydrates by DSC, TA Instruments, Thermal Solutions (TS 26)

3. Gartner E. M., Gaidis J. M. Hydration Mechanisms, I. In: Jan Skalny, editor. Materials Science of Concrete 1. USA: The American Ceramic Society, Inc.: 1989. p 95-126.

KEY WORDS

Q5000 SA, VS, Vapor Sorption, Humidity, Relative Humidity, Calcium Sulphate Hemihydrate, Anhydrite

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