APPLICATION OF THERMOGRAVIMETRIC METHOD IN CEMENT SCIENCE

EMPA Materials Science & Technology

Author: Belay Z. Dilnesa
Contact: belay.dilnesa@empa.ch

Introduction

❖Thermogravimetric analysis (TGA) is commonly employed in research and testing to determine characteristics of materials, to determine the rate of degradation, absorbed moisture content of materials, the amount of inorganic and organic components in materials or the kinetics of a reaction based on weight changes.

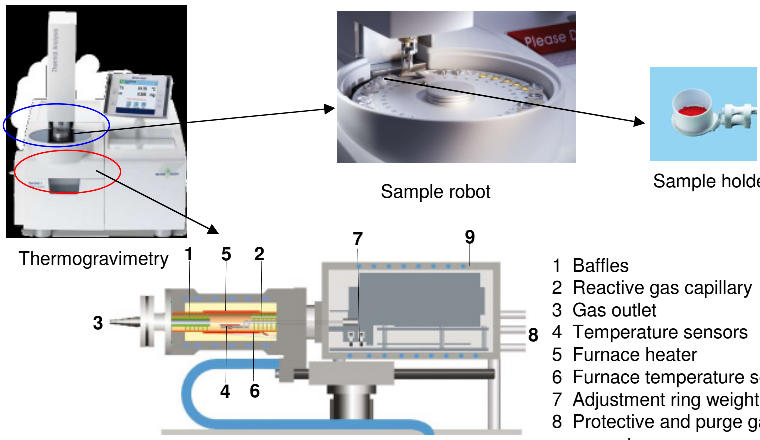
❖TGA provides quantitative measurement of mass change in materials associated with thermal degradation
.

❖Thermogravimetric curves are given for specific materials and chemical compounds due to unique sequence from physicochemical reactions occurring over specific temperature ranges and heating rates.

❖Characterization of pure materials using TGA enables :
- ❖Thermal stability investigation
- ❖ Identification
- ❖Quantification in a complex matrix

Measurement Principle

Figure 1: TGA



1 Baffles
2 Reactive gas capillary
3 Gas outlet
4 Temperature sensors
5 Furnace heater
6 Furnace temperature sensor
7 Adjustment ring weights
8 Protective and purge gas connector
9 Thermostated balance chamber

The analyzer usually consists of a high-precision balance with a pan (generally platinum) loaded with the sample. The pan is placed in a small electrically heated oven with a thermocouple (temperature sensor) to accurately measure the temperature. The atmosphere may be purged with an inert gases to prevent oxidation or other undesired reactions. Analysis is carried out by raising the temperature gradually and plotting weight against temperature.

Relevance for Our Field

❖Quantification of the amount of water

❖Characterization and identification of phases from complex cement matrix

❖TGA and derivative curve of thermogravimetric analysis (DTA) curves of cement paste can be divided into three major parts, representing two different kinds of reaction:

❖Up to 300℃ -> removal of water from hydrated products, which are likely to include most cement phases, most of the C-S-H. Several minor steps are likely to take place in this area, attributed to capillary pore water, interlayer water and adsorbed water. The corresponding peaks overlap each other because of the dynamic heating process

❖400℃–500℃ -> Dehydroxylation

❖Above 600 ℃ -> decarbonation

Example

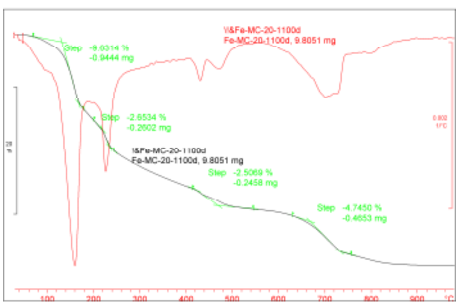


Figure 2. TGA and DTA for synthesized Fe-monocarbonate phase that shows quantifications

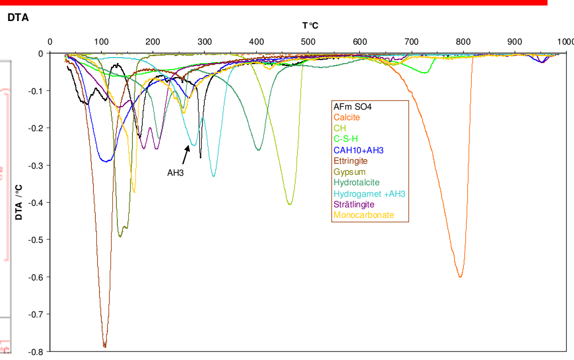


Figure 3: DTA curves for synthesized pure phases which exists in hydrated cement

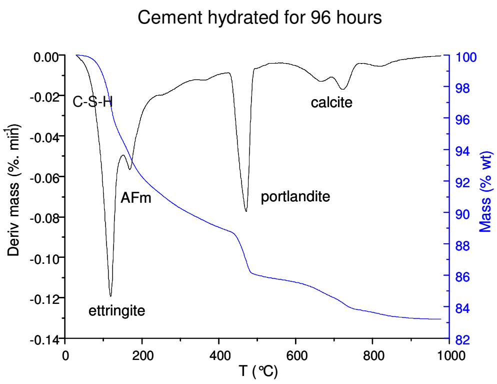
Applications & Potentials

❖ Phase quantification related to mass change

❖Phase identification in a complex cement paste

❖To study the kinetic formation of phases in cement

❖Estimates the amount of water in the hydrated cement.



Limitations

❖Difficulty of distinguishing different phases having the weight loss at the same temperature in a complex cement matrix. Example AFm phases and C-S-H

❖Small sample volume measured => large error in mortar sample

❖Total water loss is a sum parameter, influenced by the relative amount of different hydrates

❖Sensitive to drying condition

❖Data interpretation not always straightforward. Analysis in combination with other techniques is often helpful.

EMPA Concrete / Construction Chemistry Laboratory