

Determining the amount of volatile matter in a sample

Introduction

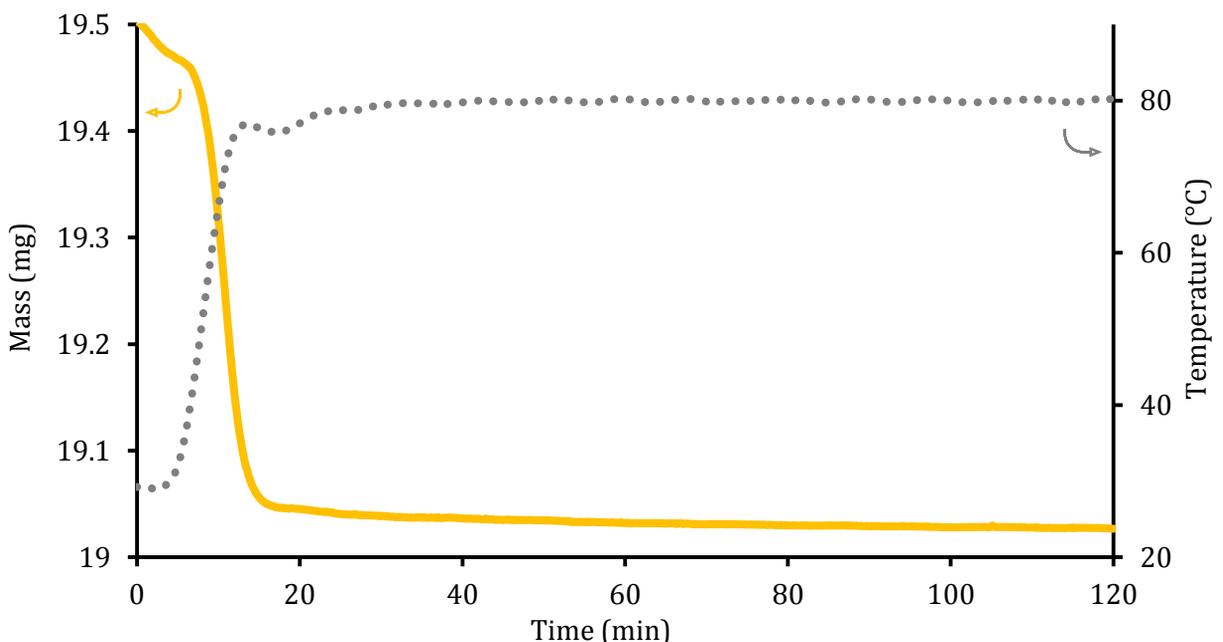
A loss-on-drying test aims to determine the amount of any kind of volatile matter in a sample when it is dried under specific conditions. **It is useful for quality control but also in research, e.g. to quantify the moisture or any solvent contained in the material.**

The procedure is straightforward: a specimen is maintained at a constant temperature while

its mass is monitored. When the mass no longer decreases or after a specific amount of time, the mass loss of the specimen is recorded: it corresponds to the amount of volatile matter extracted. The experiment is performed in a thermogravimetric analyser (TGA), which monitors mass and temperature in function of time.

Experimental details

- Method: ASTM E1868-10 standard method for loss-on-drying by thermogravimetry [1]
- Instrument: *Setaram* Setsys Evo 16/18, equipped with suspensions for TGA alone
- Thermal profile: heating ramp at 5 K/min to 80 °C, then isotherm at 80 °C for 110 min.
- Atmosphere: dry air flow at 20 mL/min
- Sample: poly(methacrylic acid-co-ethyl acrylate) 1:1
- Amount of sample: 19.50 mg
- Sample holder: open crucible of 170 µL in alumina



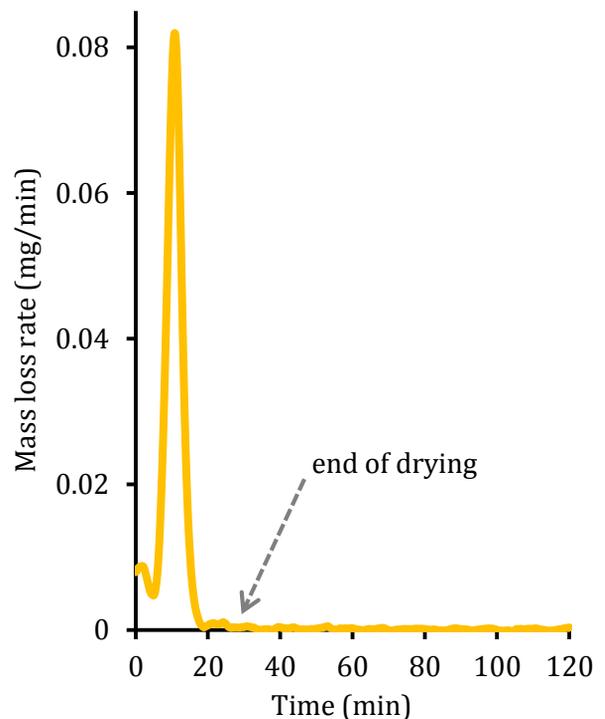
Evolution of the sample mass with time, as the temperature reaches and stays at 80 °C. The mass drops after 10 min then is stable from 20 min. The curve was corrected to remove the contribution of buoyancy effect, by subtracting the data from a identical run with the sample holder left empty.

Results

The total mass loss is evaluated from the previous curve when the mass no longer decreases. To detect it most accurately, the mass loss rate is monitored, as plotted on the right: it is simply the negative derivative of mass with time. The peak observed at 10 min corresponds to the mass loss step in the previous curve. Then the mass loss rate decreases continuously, and remains less than 0.02 mg/min from 14 min.

A common criterion for the detection of the end of drying is to wait until the mass loss rate is less than 0.1 %/min (i.e. 0.02 mg/min here) for 15 min [1]. Therefore the drying process is complete after 29 min. At this moment the sample mass is 19.04 mg, corresponding to the dry mass, so it has lost in total 0.46 mg.

The content of volatile matter in the sample is then 0.244 % in mass. The test must obviously be performed several times to estimate the uncertainty on the final result.



Conclusion

- Loss-on-drying is evaluated by maintaining a sample material at a given temperature and measuring the mass loss until a given minimal mass loss rate is reached.
- The thermogravimetric instrument used in this experiment must have a microbalance capable of measuring mass variations down to 10 µg and a furnace capable of maintaining a temperature within ± 2 °C [1].
- The isothermal temperature is chosen with respect to the thermal stability of the material, which can be evaluated by a preliminary thermogravimetric test.
- This test method is applicable to a large variety of materials, either solids or liquids, even mixtures, so long as the major component is stable at the temperature of drying.