Author: Dr. Kerstin Dreblow

Contact:
Michaela Buffler
Product Manager

Berghof Products + Instruments GmbH
Harretstraße 1
72800 Eningen | Germany

T +49.7121.894-233
F: +49.7121.894-300
michaela.buffler@berghof.com

May 2017

Article series GIT Journal

Topic series 3: Technical article

Safety-relevant Aspects of Microwave Digestion

When it comes to the preparation of samples, the main focus is on a functioning application and that the results correspond to the analytical requirements. But for applications to function safely when working with acids, it is important to not only understand the chemistry behind the digestion but the construction-relevant aspects as well.

1. SENSOR TECHNOLOGY

In microwave digestion, the sample is heated with an acid mixture up to 260°C in a microwave-transparent pressure vessel. In the process, the sample disintegrates completely and goes into solution. The advantage of microwave heating is that the sample solution is heated very quickly and selectively. To minimize safety risks caused by spontaneously induced exothermic reactions, monitoring the pressure and temperature parameters is particularly important. Modern sensor systems do not just measure pressure and temperature, they provide important data for controlling the microwave performance.

General requirements

The parameter that is actively influenced by the microwave is the temperature. The pressure is only a by-product. But, in terms of safety, it is a critical one. The following requirements are necessary for a sensor system in general:

- **No absorption**
  Even a strong microwave field must not affect the sensor. Shielded sensors have the disadvantage that they do not always operate reliably and are also bulky.
- **Fast reaction time**
  In order to effectively control exothermic reactions, the measuring speed of the sensors must be high and without any delay.
- **No contamination**
  The sensor must not contaminate the sample.
- **No corrosion**
  All components in the oven must be absolutely corrosion resistant. Coated sensors and hose systems are a disadvantage.
- **Direct measurement**
  The reaction parameters must be detected in every vessel within the shortest possible time to ensure efficient and safe control of performance.

Temperature measurement

The temperature measurement of digestion vessels can be performed in a variety of ways.

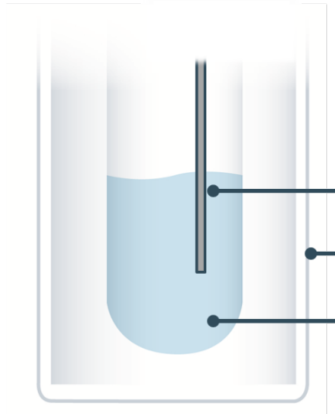

Figure 1: Type of temperature measurement

Putting sensors in reference vessels has become popular in the last few years. Temperature probes sheathed in fluorine polymer are immersed directly into the sample solution. The reaction control, however, is limited to only the one vessel - also for cost reasons. This means that for strongly inhomogeneous samples or samples with different reactivity, there is insufficient temperature monitoring. In addition, the sensors often have multiple sheaths which limits the reaction time because of heat transference in the sheath. The result is delayed detection which makes it difficult to monitor spontaneous exothermic reactions. For this reason, additional IR sensors are often used to measure the external temperature of the digestion vessel. However, this gives no information whatsoever about the current actual probe temperature.

In-situ IR temperature processes measure the probe temperature directly and are characterized by the following advantages.

- ***Fast reaction time***
  From a physical point of view, the speed of the measurement is limited only by the speed of the IR radiation from the sample to the detector. Since there is no material between the sample and the sensor, the temperature is measured without delay in real time.
- ***No contamination***
  The sensor is located outside of the digestion vessel and the microwave field.
- ***Ease of handling***
  The sensor does not need to be awkwardly mounted.

Microwave control with exothermic reacting samples

Spontaneously reacting samples offer a good way to see how quickly thermometers must function. Typically, the exothermic reactions appear quickly in the heating-up phase. The digestion of polymer granules can illustrate this. As the acid attacks and the particles begin to melt, the temperature begins to rise quickly. By continuously recording the temperature data, the microwave performance can be adjusted to ensure cautious venting.

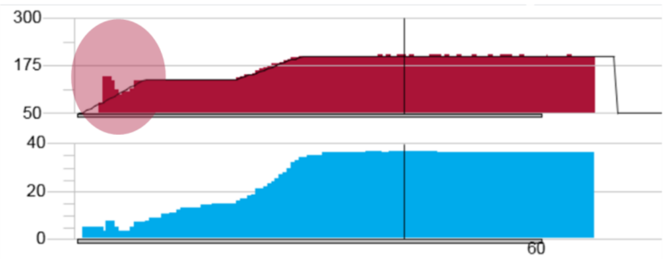

Figure 2: In-situ measuring principle for determining the sample temperature

In addition, the temperature rises along with the increase of pressure (blue curve, Fig. 2). This produces a total pressure in the vessel that is the sum of the steam from the acid mixture at the current temperature and the partial pressure from the gaseous substances that are formed. In the example given, pressure is formed by the disintegration of the polymer granules and the creation of CO_2 as a byproduct of the decomposition. Controlling the pressure is an important parameter and necessary for regulating microwave performance and safely controlling the reaction during digestion.

Pressure measurement

Pressure can also be measured similarly to temperature using external reference probes. However, the risk of contamination and leakage is also the biggest disadvantage which is why a contact-free pressure measurement with optical process is preferred.

2. VESSEL MATERIAL

Digestion vessels, or pressure digestion vessels, are a key element of modern microwave digestion systems in sample preparation and must meet a series of requirements.

- ***Microwave transparency***
  To guarantee quick, delay-free in-situ IR temperature measurement, no material that which can absorb the IR radiation may be between the sensor and the probe. The material itself must be permeable to microwave radiation.
- ***Corrosion resistant***
  All materials that come into contact with the media must be 100% resistant to chemicals because wet-chemical digestion works with aggressive reagents (acids).
- ***Pressure and temperature resistant***
  In closed systems, temperatures well above the boiling points of the reagents are created. The vessels must be able to withstand high pressures and temperatures in order to guarantee a safe digestion process.

- ***Ease of handling***
  Simple and tool-free handling is an advantage considering the fact that increasingly more samples must be processed in increasingly shorter time.
- ***No contamination***
  The risk of contamination must be minimized when working in trace and ultra-trace analytics. An optimized surface quality and reduced absorption or adsorption effect is particular important.

Vessel materials in general

There are numerous materials which can be used for storage vessels for digestion solutions or as pressure vessels for digestions (glass, PP, PE, PFA, PTFE...). PTFE and quartz have been proven in regards to the above requirements. In addition, both Teflon and quartz glass are transparent for IR radiation in a certain specific spectral range and make it possible to monitor the interior vessel temperature with modern sensors. Because of their chemical resistance, including to acids such as HF, fluorine polymers (PTFE, TFM, PFA) are especially suitable for the manufacture of digestion vessels. PTFE also has insulation properties. The material is only indirectly heated via the sample, which minimizes material stress and makes long cooling off periods unnecessary.

All other plastic materials have reduced chemical resistance compared to the fluorine polymers, which is why using them for digestion vessels must be critically assessed. PEEK (Polyetheretherketone) is particularly sensitive to nitric acid and can be attacked and, in unfavorable cases, will also absorb microwave radiation (conditional microwave transparency). Vessel overheating can occur and even lead to the vessel melting.

Press photos:

Verification

In case of publication, please send a copy.

Contact to Berghof Products + Instruments GmbH:

Michaela Buffler
Harretstraße 1
72800 Eningen | Germany
T +49.7121.894-0
F +49.7121.894-300
Email: michaela.buffler@berghof.com

Publishing office:

Berghof GmbH
Harretstraße 1

72800 Eningen | Germany
www.berghof.com

Information about Berghof Products + Instruments GmbH:
Berghof has been developing customized solutions and products of top quality polytetrafluoroethylene (PTFE / Teflon™) since 1966. As a development partner and certified series supplier for the international automotive industry, among others, Berghof Fluoroplastics has extensive knowledge of materials and manufacturing, in particular in the area of the isostatic presses. Through our use of homogeneously compressed materials from this process, our customers benefit from the experience, the exceptional material properties, the top quality and manufacturing precision.