Preparation of Soil, Sewage Sludge and Sediment Samples in a Wastewater Treatment Laboratory

The preparation of a mixture of organic and inorganic samples holds some difficulties: whereas sand, clay and stones can usually be ground to homogeneous samples with suitable laboratory mills, the high energy input can cause samples with organic components such as fat or starch to cake. Carsten Bunn, a laboratory technician at the waste water treatment laboratory BRW, has to deal with this problem every day. He treats samples which are taken from the sand traps of the wastewater treatment plants and consist of exactly that mixture. The sediments of household and industry waste water not only contain sand, clay or leaves but anything that people nowadays dispose of through the sewer system: cellulose, hair and especially food residues.

Analytics in the context of waste water treatment and water maintenance cover a wide spectrum. The German BRW laboratory which is located near Duesseldorf, analyzes approximately 5,000 samples a year. Part of their responsibilities is the general water maintenance and wastewater treatment which includes the disposal of sewage sludge and other solid substances. Although the analysis of water is their main task, the analysis of solids becomes more and more important. The BRW prepares three different types of solid samples:

1. **Soils:** e.g. from storage basins or construction projects
2. **Sediments:** from the preliminary purification of biological sewage works
3. **Sewage sludge:** from biological waste water treatment

What is left after the preliminary purification steps in the sand traps of waste water works is a mixture of various organic and inorganic materials. These sediments are usually disposed of at dumpsites. The disposal costs depend on the content of contaminants of the waste material. Therefore, these types of sample can only be dehydrated with gentle drying methods in order to prevent the escape of volatile substances. In the BRW laboratory this is done by freeze-drying.

The laboratory does not only analyze sediments but also sewage sludge which is either used as fertilizer or intended for thermal utilization. These samples are only analyzed for their heavy-metal content so drying them at 105 °C is not a problem.

"Never Had Any Down Times"

As part of the preparation for the subsequent analysis, the dried samples are pulverized in Retsch's Planetary Ball Mill PM 400. This mill generates a very high energy input which keeps the grinding time short. Thus, a heating up of the sample material can be avoided which could otherwise falsify the analytical results with regard to the volatile components. Moreover, planetary ball mills are not only suitable for hard materials; they also homogenize fibrous, abrasive and soft materials which are typically found in sand traps. To ensure a high sample throughput in his lab, Carsten Bunn considers reliability and operational convenience the most important features of a laboratory mill. "When the samples come in in fits and starts, everything must go smoothly", he describes his daily routine. "With the PM 400 we have never had any down times. The fact that it is very easy to use helps to speed up the sample processing." A high throughput is also ensured by the four grinding stations of the PM 400 ball mill. As most of the samples are analyzed for volatile substances, the grinding process must not lead to a temperature increase of the material. The BRW achieves optimum results in the PM 400 with a speed of 200 rpm and a grinding time of only 5 to 10 minutes.

How to Avoid Caking of the Sample

The most difficult components of the mixture are food residues. Starch and fat tend to cake during grinding. To remove these adhesions manually is a wearisome and time-consuming task. The laboratory assistants at the BRW know how this can be avoided: after the sample has been ground, the grinding jars are simply filled with silica sand and ground glass which act as cleaning agent. If it doesn't influence the subsequent analysis, a few drops of alcohol added to the sample can also help to reduce caking.

"No Sign of Wear After One Year of Use"

Most of the soil and sewage sludge samples are analyzed for heavy metals, therefore the grinding tools must be heavy-metal free. Grinding jars made of agate, which would normally be the material of choice, are not suitable here as the samples have an abrasive effect and cause grooves in the material. This can lead to the accumulation of material residues causing sample to sample contamination. To avoid this risk, the BRW uses heavy-metal-free jars of zirconium oxide. Carsten Bunn is convinced of the high quality and solidity of these grinding tools. Even after one year of regular use, he cannot detect any sign of wear.

The consideration of a few basic aspects when using planetary ball mills can lead to a significant reduction of wear:

- **Filling degree:** Follow the recommendations for jar filling – if the level is too low, the strain on the material will increase as will the wear.
- **Speed:** Operate the mill at the lowest speed that will achieve the desired result. High speed increases the energy input and puts a strain on the grinding tools.
- **Number and diameter of balls:** The manual includes recommendations for the number and size of balls that should be used. Using too many balls as well as mixing different sizes will significantly increase wear.
- **Grinding time:** Do not grind for longer than is necessary since longer grinding times increase wear.

Subsequent Analysis

The range of components which are then analyzed is wide: Starting with heavy metals such as lead, cadmium, mercury or chromium the list goes on into the depths of organic chemistry. TOC, PAK, PCB, EOX or BTEX are common pollutant categories in the analytics of water and solids.

Conclusion

With just one laboratory grinder – the planetary ball mill PM 400 – the BRW produces representative samples for subsequent analysis, regardless of type and origin of the sample! The only exception are soil samples which are subjected to primary size reduction in a jaw crusher (type Retsch BB 51).

AUTHOR DETAILS

Ute Vedder,
Marketing,
Retsch GmbH
Rheinische Str. 36,
D-42781 Haan, Germany.
Tel: 0 21 29/55 61-155
Fax: 0 21 29/55 61-213
E-mail: u.vedder@retsch.com
Web: www.retsch.com