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Preparing a Sample for Determining the Size of Particles

De-agglomeration with Ultrasonic Homogenizers

Complete de-agglomeration of particulate materials is a prerequisite for good results in particle size analysis. Treatment in ultrasonic baths is often insufficient. This article compares several methods of de-agglomeration.

Since particles are often present as agglomerates, it is necessary to use homogenization methods that facilitate complete dispersion of the particles. In comparison with the common ultrasonic baths, higher energy densities can be transferred to the medium by using ultrasound homogenizers (up to 1500 W / cm^2 compared to 1-5 W / cm^2). Examples for two matrices (dietary supplements and catalytic oxides) are described here in detail.

The Methods

In the following experiments, homogenization was carried out using an ultrasound machine (Sonopuls HD 3100, Bandelin). The micro tip MS 73 and the cone tip KE 76 served as sonotrodes. Amplitude 50-100%, sonication time 1-15 min without pulsation. Some of the samples were cooled during the treatment.

Dietary Supplements

In the method developed for de-agglomeration of dietary supplements containing SiO_2, three different methods for homogenizing powders in aqueous solution (mechanical dispersing rod, ultrasonic bath and ultrasonic sonotrode) were tested and compared to each other.

In each case, 0.15 mg of SiO_2 was dispersed in 2 mL of water, the times and intensities of homogenization being varied. For the separation of larger aggregates and agglomerates through sedimentation, the dispersions were allowed to stand for a few hours. Subsequently the particle size distribution was measured using asymmetric flow-field-flow fractionation (AF4) in combination with UV and light scattering detectors.

It was found that the most effective homogenization can be achieved with the ultrasound sonotrode (Table 1 and Figure 1). When using a dispersing rod or an ultrasonic bath, larger particle agglomerates are still present.

The necessary duration of sonication was determined for sufficient homogenization of a solid dietary supplement using the ultrasound sonotrode. It showed that 15 minutes are sufficient.

Depending on the duration of sonication, cooling is recommended. The sonotrodes must be regularly checked for erosion (wearing away of metal particles), and replaced if necessary.

Catalytic Oxide Based Materials

An integral part of vehicle catalyzers are oxide based materials. As the initial size of the oxide particles has to be adjusted before use, it is of great importance that the particle size distribution is determined exactly. The particle size distribution is measured using static light scattering.

The de-agglomeration of the particles is often not ensured by using the ultrasound bath treatment, especially since this treatment is carried out with a highly diluted sample. Therefore an external ultrasonic homogenizer (USH) is recommended. The influence of the USH is shown in Figure 2a.

It can be seen that the ultrasound treatment causes the two peaks on the right side of the distribution curve to disappear. However, what seems to be of little relevance in the volume-based distribution curve, changes the d99 values significantly (see Table 2). In the example shown, the particle size d99 is considered to be the limit. It can be seen how different the values are at 7 µm, with and without US treatment.

By means of an ultrasonic homogenizer, it is also possible to determine measuring values for the hardness of particulate raw materials. The samples have to be the same in terms of solids content, volume, sample vessel, sonotrode type and sonication time or intensity. Figure 2b shows an example result. There were two different oxides with different grain sizes and different behaviors with regard to the treatment in the ultrasonic homogenizer.

The coarser oxide (1) shows no change in the particle size distribution through the ultrasonic treatment. The finer oxide (2) shows a significant decrease in particle size.

The possible relevance always depends on the use or the process. If, for example, it is combined with a grinding process, then it can be assumed that oxide (2), as a “softer” material, is reduced faster than oxide (1).

The Practical Process

In order to obtain reproducible results, it is always advisable to weigh the amount of sample to be dispersed and to treat it with ultrasound in a so-called rosette cell. The solids concentration should not be too high, so that the mobility of the particles is not restricted.

It has been found that for oxides, a sonotrode with a larger cross-sectional area leads to better results in terms of the duration of the dispersion. The sonication itself takes place continuously. For heat-sensitive samples, the cell can be placed in a water bath. The dispersion should then be pulsed to promote heat removal.

The sonotrode is placed so that the tip is in the upper third of the filling level. The filling level itself should be so high that the “arms” of the rosette cell are just covered.

Particles tend to sink to the bottom depending on their density and particle size. The sampling for the particle size determination should therefore be carried out as soon as possible after dispersion in order to prevent segregation.

Conclusion

In sample pre-treatment using an ultrasonic homogenizer, it is possible to obtain reproducible measurement results, which represent the actual particle structure of the samples. For unknown samples, the measurement should always be done with and without pre-set sonication to verify the influence and general need for the procedure. The simple handling and cleaning of the smooth rod (Fig. 3) are further advantages of ultrasonic sonotrodes. In addition to de-agglomeration, other proven

fields of application are homogenizing, emulsifying, suspending various substances, accelerating chemical reactions or disrupting cells in the field of sample preparation and processing.

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