Tough? Fatty? Fibrous? Quick and reproducible homogenisation of complex food samples

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Food comes in different shapes and textures and is not uniform. However, for analyses such as finding out the nutritional value or identifying harmful substances, samples that are homogeneous and representative are needed to ensure reliable and consistent results. Therefore, the samples need to be made uniform and smaller in size. This crucial step in the analytical process can be facilitated by using various kinds of laboratory mills as provided by Retsch. For most analyses, only a small amount of sample is required which needs to reflect the whole original sample. Depending on the part of the original sample from which the analysis sample was taken, results can vary substantially which affects reproducibility. The sample preparation process in general needs to suit the sample properties and the requirements of the chosen analysis method to avoid false results. The selection of grinding parameters and accessories should not change the sample properties that are to be analysed. The type of sample preparation and the required particle size are determined by the analysis method. For most digestion and extraction processes a particle size of 0.5 mm is ideal. As food differs greatly in terms of hardness, moisture or fat content, Retsch offers a range of appropriate mills and grinders for the specific requirements of the different analysis methods.

Figure 1: Knife Mill GRINDOMIX GM 200.

Food with a high water, oil or fat content

In food laboratories all over the world, Retsch's Knife Mill GRINDOMIX GM 200 (*Figure 1*) is the standard mill for the preparation of food samples. The cutting effect of the steel blades ensures thorough homogenisation of samples with a high water, oil, sugar or fat content. The GM 200, which accommodates up to 700 ml sample material, covers a wide application range from granular materials like rape seed, rice or soybeans to fibrous or tough samples like plants, meat, fish, candy, or cheese. Example: Thanks to the powerful 1000 W drive with increased traction, the GM 200 is able to homogenise difficult samples like very fatty bacon within 30 seconds without blockages (*Figure 2*). For this kind of sample, a special serrated knife is used which is much better suited for cutting through the tough meat than a conventional knife. By using the volume reduction lid, the sample is continuously forced against the knife blades, thus ensuring complete homogenisation.

Figure 2: bacon before and after homogenisation in a knife mill

Granular samples like grain, rice, or corn

Granular food that is medium-hard and has small grains is best ground into fine particles with a mix of forceful impact and shearing. The impact effect makes the grain burst and the shearing effect further reduces the samples to smaller pieces. For this purpose, the ZM 300 is very suitable, a high-speed rotor mill that can reach a maximum speed of 23,000 min^{-1} and grinds samples by impact and shearing between rotor and fixed ring sieve. The sample stays in the grinding chamber of the ZM 300 for a very short time so that the properties that need to be analysed are not altered in any way.

Application example: 200 g corn are fully homogenised at a speed of 18,000 min^{-1} in less than 30 seconds without blocking the rotor. When using a 0.5 mm ring sieve, the whole sample is ground to a fineness below 0.5 mm.

Figure 3: corn before and after homogenisation in a rotor mill.

To prevent clogging of the sieve when homogenising fatty or temperature-sensitive samples, a distance sieve should be used which creates a small gap between sieve and rotor.

Preliminary grinding of large sample pieces

Cutting mills are used for preliminary size reduction of soft, medium-hard or fibrous samples such as roots, nut shells or bones. The speed is either fixed or can be set from 100 to 3000 min^{-1}, depending on the model. The final grind size is determined by the aperture size of the exchangeable bottom sieve, which ranges from 0.25 mm to 20 mm, and by the sample properties. Three rotor types ensure optimum cutting of different types of material. By using a cyclone the sample is discharged more quickly from the grinding chamber, and the sample and grinding tools are cooled by the air stream.

Cryogenic grinding of samples with volatile components

For samples that require cooling during the grinding process, the GM 200 or the larger GM 300 are suitable. Cooling may be necessary when, for example, volatile components like pesticides have to be detected, or when the sample material is soft and elastic and needs to be made brittle to break more easily (*Figure 4*). This can be done, for example, by mixing the sample with twice the amount of dry ice. Once the mixture is sufficiently cooled, it is put in the grinding container of the knife mill and homogenised. To release the overpressure, Retsch has designed a special lid with a large opening.

Figure 4: fruit gum before and after grinding with dry ice in a knife mill.

If small amounts of several millilitres need to be embrittled and remain cool during grinding, the Retsch CryoMill or the MM 500 control are the best option. These ball mills produce finer particle sizes than the knife mills. Sample and grinding balls are placed into the jar which performs horizontal oscillations with a frequency of 30 Hz. The grinding jar is continuously cooled with liquid nitrogen from an autofill system before and during grinding, making the sample material hard and brittle so that it can be easily pulverised. This method can even fully homogenise heterogenous samples like chorizo sausages to a fineness of around 300 µm.

Small volumes, high throughput

In mixer mills like the MM 400, different adapters can be used, allowing for processing of, for example, single grains in 20 x 2 ml single-use tubes or 2 ml steel tubes. The advantage of the steel tubes is that they won't break even under cryogenic conditions. Larger models like MM 500 nano or MM 500 control accommodate up to 36 x 2 ml single use vials or 18 x 2 ml steel tubes (*Figure 5*). Thus, mixer mills are perfectly suited for grinding very small sample amounts at high throughput rate, at room temperature but also under cryogenic conditions.

Paste making procedure

Sometimes food samples like chocolate or other sweets need to be warmed to improve, e. g., the viscosity. Retsch's MM 500 control offers the option to heat

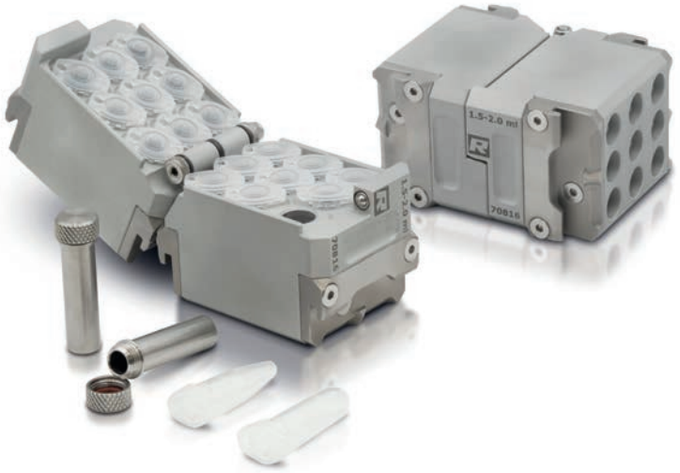

Figure 5: Adapters for 2 ml tubes used in the Mixer Mill MM 500.

the samples up to 100°C during a homogenisation or mixing process. This can be very helpful, for example, if oils need to be mixed to increase viscosity. For such applications, stainless steel jars up to 125 ml size, filled, for instance, with 8 x 20 mm steel balls for mixing, are suitable.

Sample preparation for pesticide analysis with the QuEChERS method

The so-called QuEChERS method ('Quick, Easy, Cheap, Effective, Rugged, and Safe') was developed to make sample preparation for pesticide analysis more efficient. For this method, 10 g of the homogenised food sample is extracted with 10 ml acetonitrile, diluted with a salt mixture, by shaking for 1 to 3 minutes. To simplify the lab work and ensure representative results, the shaking processshould be carried out in a modern lab mill like Retsch's Mixer Mill MM 400. Up to eight 50 ml Falcon tubes can be placed in an adapter and moved with a frequency of 30 Hz, ensuring thorough mixing and consequently a good extraction. Finally,the organic phase is dried and submitted to chromatographic analysis to detect the pesticides.

Summary

Retsch provides a range of laboratory mills for fast, accurate and neutral to-analysis preparation of virtually any food sample. With a variety of accessories allowing for flexible adaptation to the sample material and volume in question, samples with high water, sugar, oil or fat content as well as granular materials are easily homogenised. Cryogenic grinding with liquid nitrogen or dry ice is suitable to process soft, elastic materials or those with volatile substances. Heating can enhance the mixing effects of sticky samples. With the correct sample preparation process and suitable laboratory mills, the effectiveness and reliability of food analysis can be greatly increased.