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## Reducing the number of analyses by efficient sample preparation

Analytical techniques are on the fast track to quicker and more accurate results. The limiting step in the analytical pathway, particularly in food analysis, is sample preparation. The power of new analytical methods cannot be fully appreciated if preparation of the sample is not efficient. This article discusses the relevance of the mixing, grinding and homogenization process and their influence on the accuracy of the sample analysis.

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# Mixing Process – The Critical Step in Sample Preparation

## A comparative study of four mixers

Keywords: Sample preparation, mixer, sunflower seeds, particle size distribution, homogeneity, mixing, grinding

*The aliquot size for chemical analysis is often only a few grams or milligrams of homogenized sample. If the sample is not sufficiently homogenized, the aliquot may not be representative of the whole sample. Reliable results are thus only obtained by adequate homogenization during the mixing process.*

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### Introduction

The following experiments concentrate on the mixing process of sunflower seeds with four different mixers. The main focus was on the particle size distribution and the homogeneity of the processed sample. All parameters were equal for all mixers, except for the sample amount, which was optimized for each instrument. The tested mixers are: Büchi B-400; Moulinex Moulinette SE, Retsch Grindomix GM200 and Robot coupe Blixer 3 plus.

### Visual impression

With each mixer, samples were processed for 3, 10 and 30 seconds.
The differences shown in fig. 1 are significant. After 3 seconds

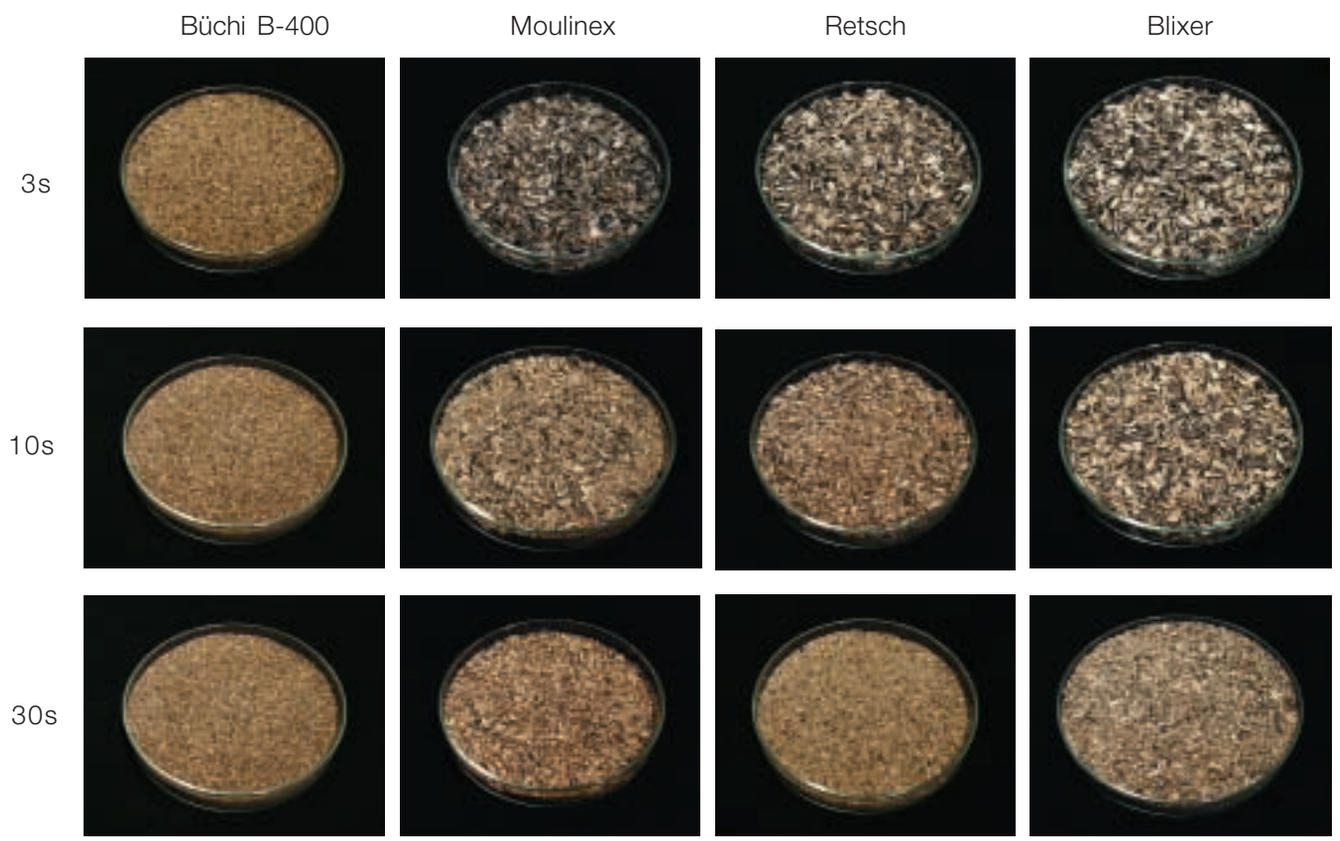


*Fig. 1 Sunflower Seeds. Pictures taken after 3, 10 and 30 seconds mixing with Büchi B-400, Moulinex, Retsch and Blixer.*

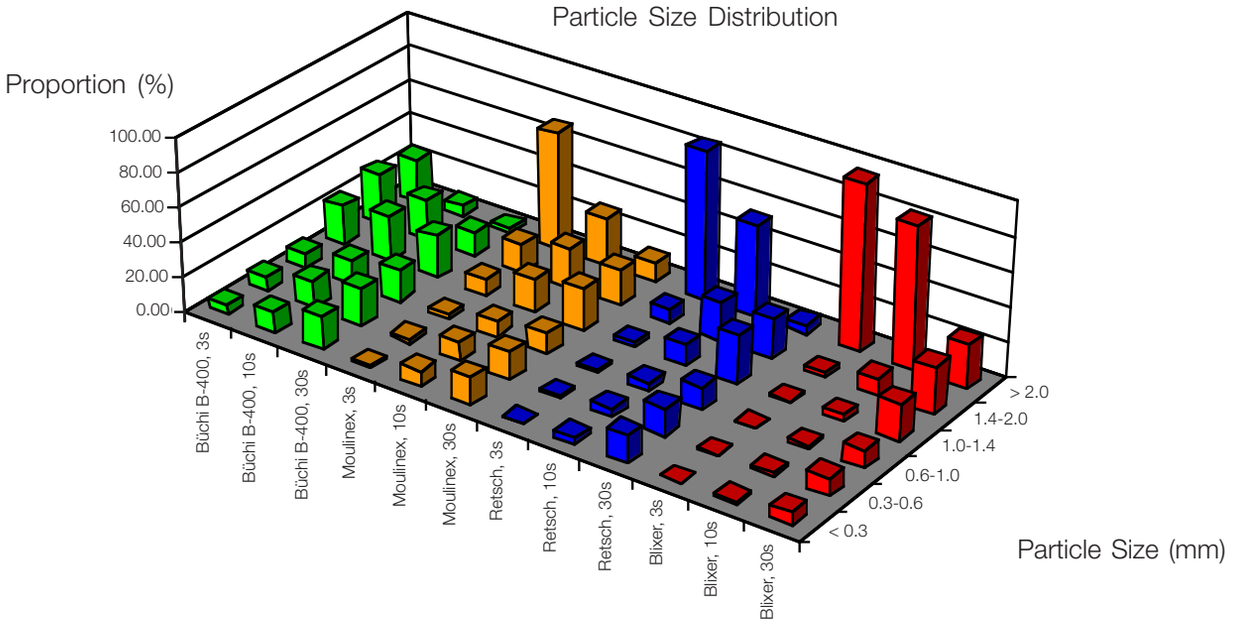


*Figure2: Particle Size Distribution. Proportion of particles with size of<0.3mm, 0.3-0.6mm, 0.6-1.0mm, 1.0-1.4mm, 1.4-2.0mm and >2.0mm for each mixer after 3, 10 and 30 seconds.*

of processing with the Büchi B-400, the sunflower seeds already have a very fine-grained consistency, whereas the samples of the other three mixers still contain many large particles. The particle size of samples processed with the other three mixers visibly decreased after longer mixing times. Comparable results are obtained if samples are processed for 3 seconds with the Büchi B-400, and 30 seconds with the Moulinex, Retsch and Blixer. It is desirable to minimize the mixing time, thus minimizing heat generation during mixing.

## Particle size distribution

The particle size distribution of the mixed samples was determined with the aid of a sieving tower and compared using laser diffraction measurements (fig. 2). These measurements confirm the visual impression of fig. 1.

After a processing time of 3 seconds with the Büchi B-400, the proportion of particles >2 mm is below 26 %, whereas with the other instruments the proportion of particles >2mm is 67% (Moulinex), 86 % (Retsch), and 96 % (Blixer), respectively. The only mixer with a considerable amount of particles <1mm after mixing for 3 seconds was the Büchi B-400. The Moulinex needed 10 seconds and the Retsch and Blixer needed 30 seconds to obtain comparable results.

## Homogeneity

In order to optimize the parameters (number of mixing intervals and time per interval) in only a few steps, the software Experimental Design (Design Expert Version 5 Software) was chosen. This software suggests carrying out 13 experiments with different numbers of mixing intervals and different mixing times.
The fat content was determined in duplicate for all samples. The samples were processed according to the design suggestions.
Fig. 3 shows the analyzed fat content and its deviation.

Target fat contents are obtained by mixing the sample as follows:

6....96 sec. with Büchi B-400
24...35 sec. with Moulinex
24...96 sec. with Retsch
70...96 sec. with Blixer

Good results with small deviations are obtained after mixing for only 6 seconds with the Büchi B-400. Results from samples processed with the Moulinex show rather large deviations and no improvement after longer mixing times. Retsch and Blixer give comparable results to the Büchi B-400, but need 5 to 10 times longer.

As seen from the results of samples processed with the Moulinex for more than 35 seconds, long mixing times may be harmful to the sample. The effect of heating has not been analyzed further, but the sample appears to start to decompose. This is also confirmed by the results after 35 seconds mixing time. The mean value of the 8 results is within the target amount, but the standard deviation is very large. This indicates that the fat is no longer evenly distributed and many more samples need to be analyzed to obtain an accurate result.

Digression: Importance of the mixing process for NIR analysis
For classical analyzing methods, where sample sizes in the range of a few grams can be taken, it might be sufficient to have particles <2 mm.
However, experiments on NIR measurements have demonstrated that it is advantageous to have small particles. Obtained results thus correspond better to the reference measurements. Larger particles disrupt the calibration and the measurements, so that uncertainty factors and standard deviations are increased.

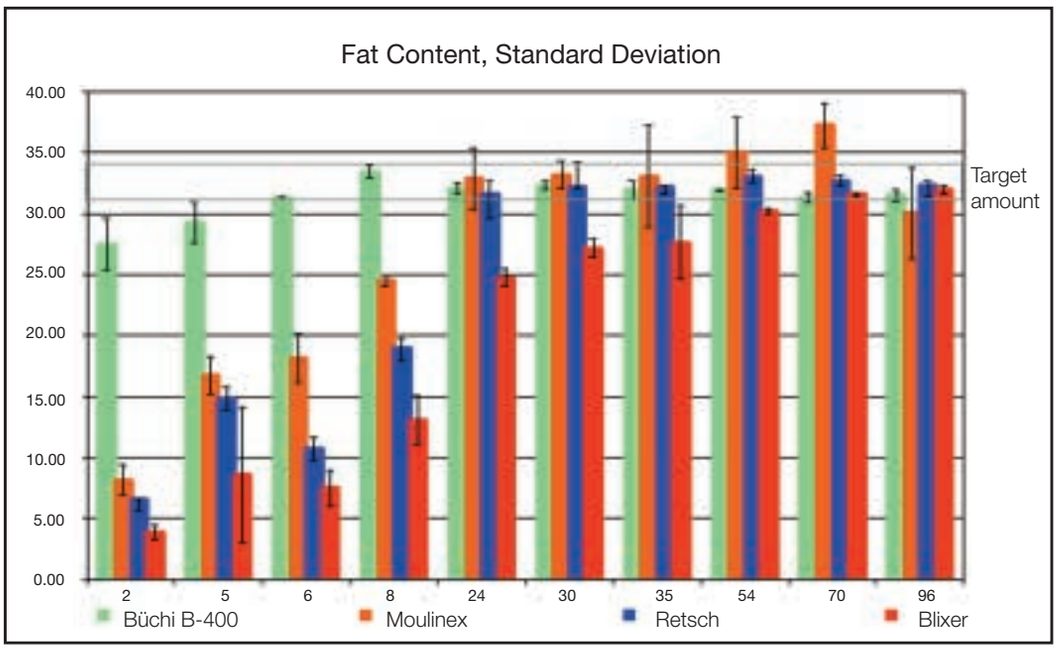


*Fig. 3 Fat Content and RSD. The fat content and absolute standard deviation is based on two samples run in parallel (Exception: at 35 seconds, 8 samples in parallel).*

## Discussion

Through evaluation of the results with Experimental Design, the working range of the tested mixers can be shown (see fig. 4). The colored area represents the range where a fat content between 32 and 34g/100g and a RSD below 1% has been reached.

Büchi B-400 is the instrument with the largest colored area and therefore the most effective and most flexible mixer in the test.
Retsch and Blixer fulfill both conditions (fat content of 32...34g/100 g and RSD < 1%) in a narrow working range. This means that the mixing procedure has to be optimized for each sample, otherwise a representative and reliable result will not be obtained. Samples processed with Moulinex are not homogeneous, RSDs below 1 % are impossible to achieve and the results are unreliable.

## Conclusion

These experiments show that analytical results and their standard deviation directly depend on the mixing time and the number of mixing intervals. It can also be seen from the Moulinex example, that small particle sizes (shown in fig. 2) do not necessarily correspond to well homogenized samples

The results of the experiments also show that well homogenized samples are obtained if the samples are treated with more mixing intervals and less mixing time per interval. In practice, such studies for ideal mixing parameters would have to be done for each kind of sample. As time and money is valuable, it is preferable to use an instrument which gives homogeneous samples over a broad range, so that it is easy to find parameters which fulfill the demands.

Good sample preparation is essential for accurate results with only few measurements. The Büchi B-400 is the only one of the tested mixers which satisfies the demand for small particle size, homogeneity, and short mixing time.



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