Influence of Cryo-Grinding on Particle Size Distribution of Proso Millet Bran Fraction

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Abstract— Cryo-grinding is an ultra-fine grinding method used in the pharmaceutical industry, production of herbs and spices and in the production and handling of cereals, due to its ability to produce powders with small particle sizes which maintain their favorable bioactive profile.

The aim of this study was to determine the particle size distributions of the proso millet (Panicum miliaceum) bran fraction ground at cryogenic temperature (using liquid nitrogen (LN₂) cooling, \( T = -196 \, ^\circ C \)), in comparison to non-cooled grinding. Proso millet bran is primarily used as an animal feed, but has a potential in food applications, either as a substrate for extraction of bioactive compounds or raw material in bakery industry. For both applications finer particle sizes of the bran could be beneficial.

Thus, millet bran was ground for 2, 4, 8 and 12 minutes using the ball mill (CryoMill, Retsch GmbH, Haan, Germany) at three grinding modes: (I) without cooling, (II) at cryo-temperature, and (III) at cryo-temperature with included 1 minute of intermediate cryo-cooling step after every 2 minutes of grinding, which is usually applied when samples require longer grinding times. The sample was placed in a 50 mL stainless steel jar containing one grinding ball (Ø 25 mm). The oscillation frequency in all three modes was 30 Hz. Particle size distributions of the bran were determined by a laser diffraction particle sizing method (Mastersizer 2000) using the Scirocco 2000 dry dispersion unit (Malvern Instruments, Malvern, UK).

Three main effects of the grinding set-up were visible from the results. Firstly, grinding time at all three modes had a significant effect on all particle size parameters: \( d(0.1) \), \( d(0.5) \), \( d(0.9) \), \( D[3,2] \), \( D[4,3] \), span and specific surface area. Longer grinding times resulted in lower values of the above-listed parameters, e.g. the averaged \( d(0.5) \) of the sample (229.57±1.46 µm) dropped to 51.29±1.28 µm after 2 minutes grinding without LN₂, and additionally to 43.00±1.33 µm after 4 minutes of grinding without LN₂. Only exception was the sample ground for 12 minutes without cooling, where an increase in particle diameters occurred (\( d(0.5)=62.85±2.20 \) µm), probably due to particles adhering to one another and forming larger particle clusters.

Secondly, samples with LN₂ cooling exhibited lower diameters in comparison to non-cooled. For example, after 8 minutes of non-cooled grinding \( d(0.5)=46.97±1.05 \) µm was achieved, while the LN₂ cooling enabled collection of particles with average sizes of \( d(0.5)=18.57±0.18 \) µm. Thirdly, the application of intermediate cryo-cooling step resulted in similar particle diameters (\( d(0.5)=15.83±0.36 \) µm, 12 min of grinding) as cryo-milling without this step (\( d(0.5)=16.33±2.09 \) µm, 12 min of grinding). This indicates that intermediate cooling is not necessary for the current application, which consequently reduces the consumption of LN₂.

These results point out the potential beneficial effects of millet bran grinding at cryo-temperatures. Further research will show if the lower particle size achieved in comparison to non-cooled grinding could result in increased bioavailability of bioactive compounds, as well as protein digestibility and solubility of dietary fibers of the proso millet bran fraction.

Keywords— ball mill, cryo-milling, particle size distribution, proso millet (Panicum miliaceum) bran.

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