Cryogenic grinding of brown rice with RETSCH’s CryoMill

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Rice is Japanese staple food and one of the most frequent samples of food analysis. Dr. Koji Baba of the National Institute of Agro-Environmental Sciences in Ibaraki Prefecture develops analytical methods for the detection of toxic substances in soil and plants and examines their dynamic states. Dr. Baba uses RETSCH’s CryoMill for sample preparation prior to analyzing brown rice.

The number of people poisoned by arsenic in Asia shows no sign of declining. Much attention is paid on arsenic intake through rice, Asia’s main diet, as well as contaminated drinking water. Dr. Baba is studying analytical methods of arsenical brown rice in order to determine its species and contents in agricultural environment. Arsenic appears mainly in two inorganic forms, arsenite (As (III)) and arsenate (As (V)). The valence of arsenic is unstable and easily oxidized by heating. Therefore, it is important to grind samples under low temperature prior to analysis. For this reason Dr. Baba chose RETSCH’s CryoMill which features an integrated cooling system ensuring that the grinding jar is continually cooled during the grinding process.

Grinding Process

Instrument: CryoMill
with 50 ml grinding jar of stainless steel
and 25 mm diameter ball of stainless steel

Parameters: Pre-cooling = automatic
Grinding = 25 Hz; 150 seconds; 2 cycles
Sample volume = 10 g

Result:
d50 = 31.954 μm

Result
By comparing grinding at ambient temperature and cryogenic grinding under the
same conditions (10 g, 25 Hz, 150 seconds, 2 cycles), Dr. Baba found the former often did not provide the required fineness. Reducing the volume to 7.5 g and conducting the same comparison, he found the cryogenically ground particles were obviously finer with a lighter color. (Fig. 1) The lighter color means that the surface of the brown rice with dark color was also finely ground.

The CryoMill also improved the efficiency of sample preparation. Cryogenic grinding of 10 g more sample can achieve the same result as 5 g at ambient temperature. This means the former can process twice of the sample in comparison with the latter.

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Fig 1: Difference of ground samples

Ambient grinding (7.5g)  Cryogenic (10g)  Ambient (5g)
Particle distribution graphs below (Fig. 2) show two peaks both with ambient and cryogenic grindings. It suggests brown rice has different substructures with different grind ability.

Fig 2: Particle distribution

Upper: Ambient grinding (5 g, 150 seconds, 2 cycles)
Lower: Cryogenic grinding (10 g, 150 seconds, 2 cycles)
Figure 3 below shows results of ICP analysis of brown rice cryogenically ground by CryoMill. Comparing the standard sample, arsenic acid, arsenic trioxide and dimethylarsenic acid were detected while monomethylarsenic acid was not.

Dr. Baba chose RETSCH’s CryoMill because it can be used both for cryogenic and ambient grinding. Thus it is possible to compare the results of the different grinding modes and see if the heat has an effect on the sample. In addition, the mill is safe to use because the user does not get into contact with the liquid nitrogen. Furthermore liquid nitrogen circulates only around the grinding chamber, which avoids extra condensation that easily causes mechanical and electrical damages.

The present focus is on arsenical compounds, but toxic substances which harm agricultural environment are not only arsenic but also cadmium, residual pesticides, POPs and so on. Not a few of them are temperature-sensitive and volatile. It is necessary to cryogenically grind samples prior to analyzing such substances.

Fig 3: Result of ICP analysis
From left to right: Arsenic acid, Arsenic trioxide, Monomethylarsenic acid, Dimethylarsenic acid
Dr. Baba has a grinding jar of stainless steel and is keen to use a jar of zirconium oxide newly developed by Retsch because molybdenum content in stainless steel has some risks to interfere analyzing Cd by ICPMS.

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1) Assessing risk in agricultural environments and developing risk management technologies.

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3) Basic research to help elucidate the functions of agricultural ecosystems.