SOP No: SOP-CHEM-001	SOP Description: Surface and Total Lipid Content Determination by Extraction
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SOP-CHEM-001: Surface and Total Lipid Content Determination by Extraction

Scope:

This procedure is used to measure lipid content of rice. Rice lipids are primarily found in the bran and germ layers of the kernel. When rice is milled, these layers are removed, thus removing a majority of lipid. Consequently, surface lipid content is used in the rice processing industry to indicate degree of milling. Surface lipid content is determined by analyzing whole kernels of milled rice. Total lipid content of brown rice (germ and bran layers intact) or milled rice is determined using a ground sample.

Principle:

The Soxtec system is based on principles of the conventional Soxhlet fat extraction method. Samples are weighed into porous sample thimbles and placed in an extraction unit, where they are exposed to a solvent. Soluble lipid material is extracted into the solvent during a two-stage process of boiling and rinsing. The distilled solvent is then condensed and collected. A final drying step evaporates the last traces of solvent from the extraction cups. The dried extraction cups are weighed and mass percentage of lipid content is calculated.

Equipment:

Soxtec Avanti 2055, Foss North America, Eden Prairie, Minnesota
Soxtec accessories: thimble handler, thimble holder, extraction cups, and extraction cup rack
Clean, dry extraction thimbles
Glass boiling beads
Defatted cotton
Petroleum Ether

Procedure A – Surface Lipid Content: for whole-kernel brown or milled rice samples

1. Turn on Soxtec controller, water bath (recirculator), and hood.
    a. Press the Hot Plate button on the controller to initiate the pre-heat cycle.
2. Using the magnetic thimble handler, place a thimble into the metal holder. Place the unit onto a balance and tare. (Do not touch the thimbles with bare hands, as oil from your skin can skew results. Always use the magnetic handler or latex gloves.)
3. Weigh 4-5 g of sample into the thimble and record the sample weight as W_1.
4. Transfer the thimble to a drying rack and repeat steps 1-3 for up to five additional samples.
5. Place a piece of clean defatted cotton on top of each sample to keep it from boiling out of the thimble.
6. Dry the samples at 103 ± 2°C for one hour.
7. While samples are drying, verify that there is sufficient water in the recirculator. The tank should be filled to within approximately 3 inches (one finger-length) of the top.
    a. Add deionized water to the appropriate depth.
    b. On a weekly basis (ex. each Monday), add five drops of algicide to the water.

8. Cool the samples for approximately 10 minutes in the dessicator, until the drying rack is cool to the touch.
9. While the samples are drying, place 3 or 4 glass boiling beads in each extraction cup. Record the weight of the cup and beads as W_2.
    a. Cups may be stored in the dessicator until the samples are dry and cool.
10. Move the left handle of the Soxtec to its lowest position and the right handle to its highest position.
11. Attach the dried thimbles to the thimble magnets at the base of the extraction units. Always wear gloves when handling the thimbles, taking care not to touch the cellulose material with bare hands.
12. Move the left handle into the upper position; place extraction cups into the rack, and place the rack onto the hot plate.
13. Move both handles to their middle positions.
14. Add approximately 70 mL of petroleum ether by connecting the dispensing hose to the connectors at the top of each extraction chamber. (The dispenser is set to dispense 70 mL. Simply raise and lower the dispensing tube.)
15. Move both handles to their lowest positions.
16. Press the Start button**.** If/when the hot plate is at the pre-set temperature, the controller will beep, indicating it is ready to run the samples.
17. Press the Timer button, which will initiate the first phase of the program, the boiling phase (135°C, 20 min).
18. When the timer sounds, move the left handle to the middle position and press the Timer button to initiate the rinse phase (30 min).
19. When the timer sounds again, move the left handle to the top position, and press the Timer button to initiate the solvent recovery phase (5 min).
20. When the timer sounds a third time, press the Timer button, move the right and left handles to their top positions, and remove the cups.
    a. Verify that all of the solvent has evaporated from the cups. If there is liquid remaining, put the cups back on the Soxtec and press the Fan button to facilitate evaporation for a few more minutes.
21. When all solvent is evaporated, place the cups in a convection oven set at 103±2°C for 30 minutes.
22. Move the cups from the oven to the dessicator and allow to cool for at least 30 minutes.
23. Weigh the resultant sample + cup and record the weight as W_3.
24. Move the left handle of the Soxtec to its bottom position.
25. Remove the thimbles and place them back in a holder. Leave them under the hood until they are dry to the touch. When they are dry, remove the defatted cotton and discard the sample at the bottom of the thimbles. (The cotton may be reused if it is clean.)
26. To clean the thimbles, use a vacuum to remove any remaining sample residue. Thimbles may be reused, provided that they are clean and no discoloration is present.
27. Empty the boiling beads from the extraction cups and wash the cups with detergent. It is often necessary to let the cups soak for a short time to remove the lipid residue. Place the cups in the oven to dry completely.
28. After running all samples, allow the unit to cool to below 50°C. Turn off the water bath, the Soxtec controller and the fume hood. Rinse out the ether dispenser with water and empty the collected solvent from the Soxtec into a waste bottle. Store all solvents and chemical waste in the fume hood or in the cabinet under the hood.

Procedure B – Total Lipid Content: for ground rice samples

1. Follow instructions in Procedure A, except that rice should be ground into a fine flour with a Cyclone Mill prior to lipid extraction. Sample size should be approx. 5 g.

Calculation:

$$\% \text{ Lipid Content} = [(W_3 - W_2)/W_1] \text{ X } 100$$

Where W1 is the initial mass of the sample; W2 is the mass of the extraction cup (with beads) prior to extraction; W3 is the mass of the extraction cup (with beads) and extracted lipids.

Reference:

Matsler, A. L., and Siebenmorgen, T. J. 2005. Evaluation of operating conditions for surface lipid extraction from rice using a soxtec system. Cereal Chem. 82:282–286.

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