

Comparison of Three Extraction Systems for Determining Surface Lipid Content of Thickness-Fractionated Milled Rice

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ABSTRACT

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The surface lipid content (SLC) of rice is often used to objectively measure the degree to which bran has been removed from rice kernels, commonly known as degree of milling (DOM). This study was conducted to evaluate new, rapid extraction technology for potential timesaving measurements of SLC of milled rice. The SLC of two long-grain rice cultivars, Cypress and Drew, were determined using three extraction systems: Soxtec, accelerated solvent extraction (ASE), and supercritical fluid extraction (SFE). Before milling, rough rice was separated into three thickness fractions (<1.84, 1.84–1.98, and >1.98 mm) and samples from

each thickness fraction were milled for durations of 10, 20, and 30 sec. Head rice collected from each milling duration was extracted using each of the three methods. Results showed that regardless of the extraction method, thinner kernels had lower SLC measurements than thicker fractions. In most cases, both the ASE and Soxtec produced SLC greater than that of the SFE. The ASE also showed SLC measurements at least as great as those from Soxtec extraction, suggesting that the ASE is as thorough in extracting lipids as commonly used methods.

Milling produces white rice kernels with the embryo and most of the bran layers removed. Rice bran contains 12–23% oil and accounts for >60% of the nutrients within a rice kernel (Juliano and Bechtel 1985). Bran remaining on rice kernels after milling can potentially result in lowered quality, appearance, and stability due to oxidation of the oil in the bran. Thorough milling produces rice with less bran retention; however, overmilling to minimize remaining bran can result in head rice yield (HRY) reduction (Andrews et al 1992; Bennett et al 1993). Head rice yield refers to the mass percentage of rough rice that remains as head rice (>75% of the original whole kernel length) throughout the milling process. The degree of milling (DOM) of rice is the extent to which bran has been removed from the surface of rice kernels during the milling operation (Bennett et al 1993) and is important in determining HRY (Sun and Siebenmorgen 1993), paste viscosity (Perdon et al 2001), and sensory quality (Piggott et al 1991).

Limited research has been conducted on evaluating kernel size effects on milling performance. Because rough rice includes kernels of various sizes, each size fraction could mill differently, therefore affecting the DOM. Previous research has included separating milled kernels into thickness fractions and determining the surface lipid content (SLC) as an index for quantifying DOM (Chen et al 1998). The SLC is the ratio of the mass of surface lipids extracted from a sample of milled kernels to the mass of the original sample. Chen et al (1998) found that when rice was milled as an unfractionated bulk, thin kernel SLC was higher than thicker kernels, especially at low overall DOM levels. These findings indicated that measurements involving the SLC of kernels should account for varying kernel sizes.

Several chemical analyses have been employed to determine DOM. However, these methods are typically time-consuming and laborious and require large volumes of solvent. A common method to evaluate DOM is by measuring the SLC of milled rice through a solid-liquid solvent extraction (e.g., petroleum-ether) (Hogan and Deobald 1961; Mathews and Spadaro 1980). Generally, the extraction is accomplished with a system such as a Soxhlet, which is the technique routinely accepted for determining oil from oilseed crops (AOCS 1964) and for the extraction of many different analytes from

various matrices (Molkentin et al 2001; Manila et al 2002; Fatoki and Awofolu 2003). There are several disadvantages inherent with Soxhlet extraction, such as requiring 14–24 hr for extractions, as well as extensive setup, including glassware, heating mantles, and recirculating chillers. Soxhlet extraction is also a solvent-intensive method with several hundred milliliters of organic solvent required for each sample during the extraction process (Ayuso-Garcia et al 1999).

An updated Soxhlet extraction system, the Soxtec system, has recently been utilized for a wide variety of applications in the food, feed, environmental, and industrial sectors. The Soxtec system currently employed in this study includes an extraction unit and a control unit. The weighed sample is placed into thimbles and inserted into the extraction unit. Solvent is dispensed into the cups of the closed system and the cups are heated by an electrical heating plate. The four-step extraction procedure consists of predrying the sample to be extracted, immersing the sample in boiling solvent, rinsing the sample with condensed solvent, and postdrying of the extraction cup containing the lipids. The Soxtec system has recently been used to determine oil content in soybeans (Morrison 1990) with no significant difference compared with Soxhlet extraction. This would suggest that the Soxtec system is as accurate as the widely used Soxhlet system.

One limitation of the Soxtec manual system is that the operator is required to load new samples because only six samples can be simultaneously extracted; whereas, with more automated extraction technology such as pressurized liquid extraction, or more commonly, by its trade name, accelerated solvent extraction (ASE), 24 samples can be loaded onto the system and subsequently left unattended to complete the extraction. ASE emerged in the mid-1990s as a fully automated extraction system that utilizes elevated temperatures and pressures that enable solvents to be heated to temperatures in excess of their boiling point, which results in a more efficient extraction process. ASE has been applied to environmental contaminants, food matrices, and medicinal plants (Ong et al 2000; Huie 2002) with the ability to extract samples in <30 min with solvent volumes <50 mL/sample.

Another automated extraction system, supercritical fluid extraction (SFE), has been used in food, agricultural, pharmaceutical, and environmental applications owing to its versatility. Similar to ASE, SFE utilizes high temperatures and pressures, is also less labor-intensive than Soxhlet, with the ability to perform rapid extractions (often in <30 min) of target analytes, resulting in recoveries comparable to traditional extraction methods (Huie 2002; Erstfeld and Chen 1998). Carbon dioxide is commonly used as the extraction solvent in SFE extractions, which greatly reduces risks associated with hazardous solvent contact or vapors. The

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SFE system also allows for the addition of a solvent modifier for increased extraction efficiency.

An alternative method recently reported by Lam and Proctor (2001) extracts the surface lipids from milled rice by vortexing for 2 min using isopropanol, followed by 2 min more of vortexing, and then centrifuging for 10 min to remove bran particles. Automation of this technique for high sample output may be difficult.

While the Soxtec system is a traditional method of lipid extraction, it may not be the most convenient and efficient technique for the extraction of lipids to determine milled rice DOM. Surface lipids could also be extracted by newer instrumentation such as the ASE and SFE that have the advantages of reduced organic solvent consumption, reduced extraction durations, less handling, and fewer steps to be performed by the operator, and more samples loaded and extracted per analysis. The objective of this study was to investigate the automated techniques of ASE and SFE, both of which allow a high throughput of samples, relative to the standard Soxtec system in measuring SLC for quantifying the DOM of thickness-fractionated milled rice.

MATERIALS AND METHODS

Sampling Techniques

Two long-grain rice cultivars, Cypress and Drew, were harvested from the Northeast Research and Extension Center, Keiser, AR, at moisture contents (MC) of 17.2 and 18.5% (expressed on a wet basis), respectively. Cypress represents a leading cultivar from a milling quality standpoint, while Drew is currently one of the most prevalently grown long-grain rice cultivars in the mid-South. Immediately after harvest, the rice was cleaned using a dockage tester (model XT4, Carter-Day Co., Minneapolis, MN) and gently dried by placing the rice onto screen trays in a controlled temperature and relative humidity chamber (21°C, 53% rh) to achieve ≈12% MC. After drying, rough rice samples were separated into three kernel thickness fractions (<1.84 mm, 1.84–1.98 mm, ≥1.98 mm) using a precision sizer (ABF2, Carter-Day) starting with the 1.98-mm screen. Rice passing through the 1.98-mm screen was then fed to the 1.84-mm screen. The resulting mass thickness distributions are given in Table I.

Samples from each thickness fraction and the unfractionated bulk of Cypress and Drew rice were milled to obtain head rice. The milling procedure consisted of dehulling ≈150 g of rough rice using a rice sheller (THU, Satake, Tokyo, Japan). The resulting brown rice was milled in a laboratory mill (McGill No. 2, Rapsco, Brookshire, TX) for bran removal and rice collection. Placing a 1.5-kg mass on the lever arm 15 cm from the middle of the mill chamber controlled the pressure on the rice during milling. Samples of each thickness fraction of both Cypress and Drew rice were milled for durations of 10, 20, and 30 sec. Milled head rice mass was recorded and HRY were determined from each milling duration using a shaker table with a 4.76-mm screen (Grainman Machinery Mfg., Miami, FL). Average HRY of the thickness fractions for Cypress and Drew are given in Table I. Head rice samples from each milling duration of each thickness fraction were placed in plastic freezer bags, purged with nitrogen, and stored at -10°C until subsequent extraction using the Soxtec, ASE, and SFE systems.

Surface and Total Lipid Extraction

Soxtec surface lipid extraction. Surface lipids were extracted from head rice using a Soxtec Avanti 2055 manual extraction unit (Foss Tecator, Eden Prairie, MN) with petroleum ether (Mallinckrodt Baker, Paris, KY) as the extracting solvent. Before extraction, 5 g of head rice from each milling duration of the three thickness fractions from both cultivars were predried by placing into cellulose extraction thimbles (33 mm i.d. × 80 mm) (Foss North America, Eden Prairie, MN). A defatted cotton plug was placed on top of the sample to prevent any sample from boiling out of the thimble during extraction. The samples were then placed in a convection oven at 100°C for 1 hr (Hogan and Deobald 1961). Petroleum ether (70 mL) was measured into each extraction cup and the thimble was lowered to immerse the sample into the solvent for 20 min of boiling. The boiling temperature on the unit was set at 135°C, which was the recommended temperature setting by the manufacturer for extraction of lipids from ground rice. The thimble was then manually raised above the solvent surface and rinsed for 30 min by the condensed solvent to extract lipids remaining on the surface of the kernels. After rinsing, the solvent flow was manually discontinued. The solvent from the extraction cup was then evaporated for 5 min and collected inside the Soxtec. The total extraction required 50 min/sample. The extraction cups were dried at 100°C for 30 min to remove any residual petroleum ether, leaving only dry material, which represented the extracted surface lipids. After drying, the cups were transferred to a desiccator to cool for 30 min, and the mass of remaining lipids in the cups was used to calculate SLC as a percentage of the original head rice mass (4–5 g).

Soxtec Total Lipid Extraction

For total lipid content (TLC) determination (AOCS 1964), ≈20 g of head rice were ground into flour using a laboratory grinding mill (Cyclotec, Udy Corp., Ft. Collins, CO) equipped with a 0.5-mm screen. A 4–5 g subsample of ground head rice was extracted with the Soxtec Avanti 2055 following the procedure described above. The mass of remaining lipids in the cups was used to calculate TLC as a percentage of the original head rice mass (4–5 g).

Accelerated solvent extraction. Extraction of surface lipids from head rice that was predried for 1 hr as described above, was accomplished using an accelerated solvent extractor (ASE 200, Dionex, Sunnyvale, CA). The pressure during extraction was maintained at 10,342 kPa (1,500 psi) at a temperature of 105°C, which was recommended for extraction of oil from oilseeds (Anonymous 2001). Each predried sample (≈5 g) was placed in an extraction cartridge containing a cellulose filter pad, loaded onto the ASE, and extracted three times with 25 mL of petroleum ether with a static duration of 10 min. The total extraction was 30 min/sample for six samples for each thickness fraction extracted. The extract (25 mL) was evaporated under a nitrogen flow until no petroleum ether was detected. The vials were placed in a drying oven (100°C) for 30 min to evaporate residual solvent and transferred to a desiccator to cool for 30 min. The mass of the remaining lipids in the vials was used to calculate SLC as a mass percentage of the original head rice mass (5 g).

Supercritical fluid extraction. Surface lipids from head rice that was predried for 1 hr as previously described were also extracted

TABLE I
Rough Rice Mass Distribution and Head Rice Yields (HRY)^a of Thickness-Fractionated Cypress and Drew Rice

Thickness Fraction (mm)	Cypress		Drew	
	Mass Fraction (%)	HRY (%)	Mass Fraction (%)	HRY (%)
<1.84	10	40	20	42
1.84–1.98	53	67	66	72
>1.98	37	70	14	70
Unfractionated	...	64	...	67

^a Each HRY is the average of three milling determinations.

by a supercritical fluid extractor (SFE 3560, Isco, Lincoln, NE) at 51,710 kPa (7,500 psi) at 105°C, the temperature recommended by the manufacturer (S. Fraas of Isco, *personal communication*). The extraction involved carbon dioxide (ultra pure grade, 99.9%) as the supercritical fluid with a maximum flow rate of 1.5 mL/min with 15% petroleum ether as the modifier. An aliquot of 7 mL of petroleum ether was added to the collection vial before extraction for use as the collection solvent. Each predried head rice sample (3 g) was placed in the extraction cartridge containing a filtered end, loaded onto the SFE, and extracted for 30 min/sample, with six samples of each milling duration of each thickness fraction extracted. Supercritical CO₂ was passed through the extraction cell and a restrictor, which controlled the extraction pressure. Extracted lipids were collected in the collection vial, while CO₂ was vented to ambient air. After extraction, the petroleum ether (<15 mL) containing the lipid extract was evaporated under nitrogen flow until no petroleum ether was detected. The collection vials were placed in a drying oven (100°C) for 30 min to evaporate any residual petroleum ether. After drying, the vials were transferred to a desiccator to cool for 30 min, and the mass of the remaining lipids was used to calculate SLC as a percentage of the original head rice mass (3 g).

Statistical analysis. To determine SLC differences among thickness fractions and extraction systems, statistical analysis was performed using a Student's *t*-test with $\alpha = 0.05$ in the general linear model procedure by one-way analysis of variance (JMP IN 5.0, SAS Institute, Cary, NC). Significant difference between means was taken at $P < 0.05$.

RESULTS AND DISCUSSION

Table I shows the mass thickness distributions of Cypress and Drew rough rice. The mid-thickness fraction comprised the majority

of rice in each cultivar. The HRY of the thinner kernel fractions (40–42%) was lower than the mid-thickness kernel fractions (67–72%), which is supported by previous findings (Sun and Siebenmorgen 1993). The lower HRY of the thin kernel fractions are attributed to lower mechanical strength of thin, immature kernels (Lu and Siebenmorgen 1995). Because rice fractionated by kernel thickness before milling allows for a more uniform kernel size distribution during milling than that of an unfractionated, diversely sized bulk, higher HRY of the thicker fractions would be expected. This was observed in the current study in that the HRY of the thickest rice fractions (70%) of Cypress and Drew were greater than the thinnest fractions. The mass fraction percentage of mid-thickness rice was greater than that of the thickest fraction, but the two fractions had similar HRY.

Surface Lipids of Fractionated Rice

Figure 1 shows the average SLC measurements of thickness-fractionated Cypress rice, with each fraction milled for three durations. The SLC extracted from the thinnest kernels were lower ($P < 0.05$) within all milling durations compared with the mid-thickness and thicker kernels of Cypress rice when using any extraction system. SLC measurements from the mid-thickness and thicker fractions of Cypress were similar in value, although the ASE and Soxtec system mid-thickness values were statistically greater ($P < 0.05$) than those of the thickest kernel fractions for the 30-sec milling duration. Results for Drew were similar to those for Cypress in that significantly lower SLC measurements were found for the thinner kernels compared with thicker kernels (>1.84 mm) for all milling durations when using any extraction system (Fig. 2). It was assumed then, that as the milling process progressed, thinner kernels were milled at a greater bran removal rate than thicker kernels as demonstrated by lower SLC measure-

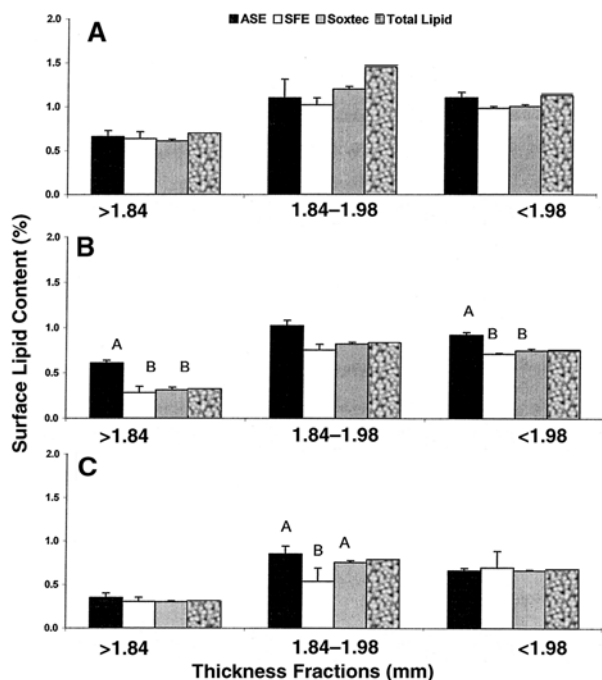


Fig. 1. Surface lipid content (SLC) of thickness-fractionated Cypress rice, with each fraction milled for 10 sec (A), 20 sec (B), and 30 sec (C), as determined by three extraction methods. Groups of columns with different capital letters are significantly different ($P < 0.05$). The first three columns in each group represent the average SLC of six extracted rice samples and the final column represents the average total lipid content of six extracted samples as determined by Soxtec analysis. Error bars on each column represent standard deviations of mean lipid extraction values.

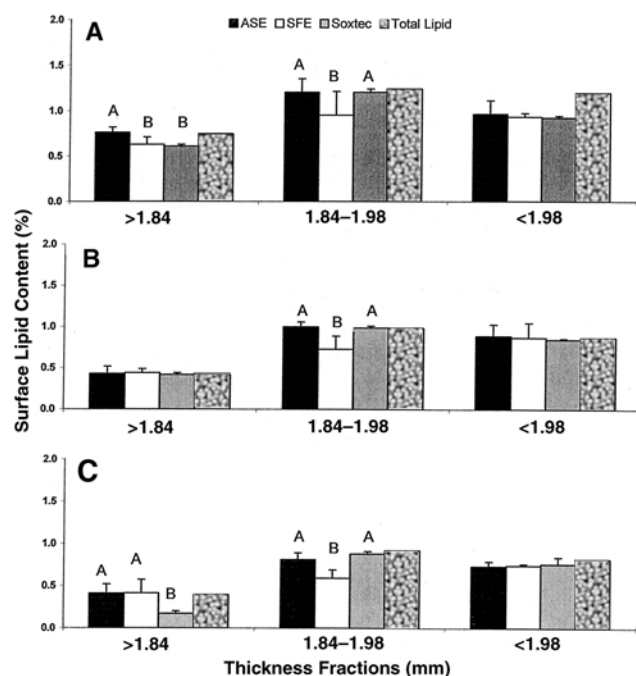


Fig. 2. Surface lipid content (SLC) of thickness-fractionated Drew rice, with each fraction milled for 10 sec (A), 20 sec (B), and 30 sec (C), as determined by three extraction methods. Groups of columns with different capital letters are significantly different ($P < 0.05$). The first three columns in each group represent the average SLC of six extracted rice samples and the final column represents the average total lipid content of six extracted samples as determined by Soxtec analysis. Error bars on each column represent standard deviations of mean lipid extraction values.

ments from thinner kernels compared with thicker ones. The current results are consistent with those of Wadsworth et al (1982), who reported that after fractionating bulk rough rice into several size fractions and milling each fraction separately, thicker kernels retained more bran on the kernel surface than thinner kernels.

For all thickness fractions of Cypress rice, SLC decreased as the milling duration increased from 10 to 30 sec (Fig. 1). For example, accelerated solvent extracted SLC of mid-thickness (1.84–1.98 mm) kernels were 1.0% at the 10-sec milling duration and decreased to 0.85% at the longest milling duration of 30 sec. This was expected because rice lipids are predominantly located in the bran layers that are progressively removed as the milling process proceeds (Andrews et al 1992). These trends of lower SLC in thinner kernels and decreasing SLC as milling durations increased were similar for all three extraction methods for both Cypress and Drew.

Surface lipid content compared with total lipid content. Total lipid analyses were conducted on rice flour based on the AOCS method (1964) using the Soxtec extraction system to determine whether the extractions performed on whole rice kernels removed only surface lipids or were removing lipids from the interior of the kernel as well. The TLC is expected to be greater than the SLC due to the fact that the TLC would include lipids from the inside and the surface of the kernel, whereas the SLC theoretically removes only the surface lipids. In most cases, the average TLC as determined with the Soxtec (Figs. 1 and 2) was similar to or greater than the values of SLC determined when using the Soxtec, ASE, or SFE. In some cases, however, particularly for the 20-sec milling duration in Cypress rice, the SLC as determined by the ASE was greater than the TLC as determined by the Soxtec. This difference may be due to the ability of the ASE to extract lipids from deeper inside the kernel because it involves a pressurized extraction, while the Soxtec procedure is not pressurized. In addition, the pressurized extraction could be removing other constituents from the kernel such as starch or protein. These results demonstrate that differentiation of TLC from SLC using the current extraction procedures may be difficult. Further research comparing surface lipid analysis of head rice to total lipid analysis of rice flour needs to be conducted using a variety of extraction techniques to clarify the discrepancy between SLC and TLC measurements.

Extraction System Comparison

Comparing the three extraction systems, greater SLC values were produced from Cypress rice when using ASE for the thinnest (< 1.84 mm) and thickest kernels (>1.98 mm) milled for 20-sec durations compared with the other two extraction methods (Fig. 1). The SLC measured from Drew using the ASE were greater from mid-thickness kernels milled for all durations than those measured by SFE and from thinner kernels at 10- and 30-sec milling durations compared with the Soxtec (Fig. 2). This is consistent with other reports (Ong et al 2000) where pressurized liquid extraction was superior to conventional extraction methods (ultrasonic and Soxhlet extraction) for the extraction of berberine and aristolochic acids in medicinal plants, and Wang et al (1999), who concluded that polycyclic aromatic hydrocarbon recoveries by ASE were comparable to or better than those obtained by Soxhlet extraction.

When using SFE, significantly lower SLC were measured for mid-thickness Cypress kernels milled for the 30-sec duration and from thinner and thicker kernels milled for 20 sec compared with the ASE extraction method (Fig. 1). It was observed that SLCs of Drew rice were lower from SFE extraction for mid-thickness kernels milled for 10, 20, and 30 sec compared with the other two extraction methods. The low SLC obtained when extracting head rice using SFE may be attributed to a high susceptibility to matrix effects, which were problematic when using SFE (Huie 2002). Previous observations have noted that various matrices inherent in food and plant materials are highly complex, and factors such as the water content and particle size of the matrix can severely limit the capacity of SFE in extraction efficiency and rapid kinetics (Huie 2002).

Practical Considerations

Given the extraction values obtained with the three systems, other operating factors should be considered. For measurement precision, Figs. 1 and 2 show that in nearly all cultivar/thickness fraction/milling duration treatment combinations, the ASE and SFE SLC had much higher standard deviations than the Soxtec SLC. For example, the standard deviation of SLC means ranged from 0.03–0.21 for Cypress and Drew when using the ASE system compared with 0.01–0.08 when using the Soxtec system. This observation could have a significant impact on the adoption of this technology in routine SLC measurement.

Regarding the duration required for extraction, the Soxtec system used in this study (capacity of six samples) required 50 min for actual sample extraction. This compares to 30 min for the ASE and SFE systems; however, 24 samples can be extracted per analysis in both the ASE and SFE compared with only six samples in the Soxtec. The ASE also required less attendance by the user while the extractions were completed, and reduced solvent consumption (25 mL/sample with the ASE and 70 mL/sample with Soxtec). However, even though the automated method of the ASE did reduce time and solvent usage, the precision of the Soxtec was superior to that of the ASE or SFE as demonstrated by the high standard deviations obtained when using the ASE (Figs. 1 and 2).

Using SFE for surface lipid extractions resulted in lower lipid values compared with the other two methods. This may be due to the experimental conditions: extraction duration, flow rate, and modifier concentration that were not fully optimized to maximize the recovery, kinetics, and selectivity of the SFE method. For example, during optimization of the SFE conditions that are necessary to effectively extract active ingredients from plant matrices, a study found that the density of CO₂ and the fluid volume passing through the material are important factors affecting extraction efficiency, and also optimizing the SFE variables of temperature, pressure, modifier concentration, static extracting duration, and CO₂ dynamic extracting volume will allow faster extraction kinetics (Huie 2002). In addition, another notable drawback was that the SFE conditions were more complex (many more factors need to be optimized [flow rate of CO₂, purge time, calibration of restrictor, restrictor flow rate] for selective extractions) when compared with fewer required parameters (extraction duration, solvent pressure, temperature) when using the ASE system.

CONCLUSIONS

This study demonstrated that regardless of the extraction method used, SLC measurements from thinner rice kernels were lower than those of thicker rice kernels. Longer milling durations (30 sec) for all thickness fractions of the two rice cultivars studied resulted in decreased levels of SLC using any extraction method. Overall, SLC obtained by ASE were comparable to or greater than those obtained by Soxtec or SFE. Lower lipid values were obtained when using the SFE compared with the Soxtec, which indicates that optimization of the many operating conditions is necessary when utilizing this system. These results indicate that the ASE is as thorough in extracting surface lipids as commonly used conventional methods for DOM determinations, with the advantages of reduced extraction time, reduction in the amount of organic solvents required for extraction, and less handling required by the operator, although less superior in precision than the conventional Soxtec.

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