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Evaluation of a new extraction system for rapid measurement of surface lipid content of rice for degree of milling estimation

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ABSTRACT

The objective of this research was to evaluate a potential time-saving method for surface lipid content (SLC) measurement of milled rice by utilizing new extraction technology. The SLC is often used as the basis for quantifying the degree to which bran has been removed from kernels during the rice milling process; this quality factor is often referred to as degree of milling (DOM). The SLCs of two long-grain cultivars of rice, 'Cypress' and 'Cocodrie', were determined using an accelerated solvent extraction system (ASE) and compared to the conventional, manual system (Soxtec extraction system) that is typically used for SLC measurement. Both systems were tested at extraction temperatures of 115°C, 135°C, and 150°C with total extraction durations of 30 and 50 min. Results indicated that the longer extraction duration, 50-min, produced the lowest SLCs and higher temperatures generally produced lower SLCs. Overall, the surface lipid levels measured by the ASE were similar to or greater than the Soxtec, suggesting that the ASE is as reliable as conventional methods used for DOM determinations based on surface-lipid extractions, with the added advantages of reducing organic solvent usage, extraction time, and labor.

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MEET THE STUDENT-AUTHOR

Amanda Parker

I was born in Springfield, Mo., where I graduated from Glendale High School in 2000. I am currently a junior at the University of Arkansas and a food science undergraduate. I am a member of Pi Beta Phi sorority, Gamma Beta Phi, and the National Society of Collegiate Scholars. I received the non-resident tuition award and a food science scholarship to help me pursue my goals. Recently, I received the Silo Undergraduate Research Fellowship to help fund my research.

I plan to graduate in 2004 with a bachelor's degree in food science and a minor in communications. I then want to pursue graduate school to further my education in Food Science. My goal is to work in the field of research and development.

In the Food Science Department, I have been given the opportunity to work in rice research. I decided to do this project upon the encouragement of Dr. Cindy Rohrer and Dr. Terry Siebenmorgan who both have been a tremendous help. Through my research I learned many things about degree of milling and surface-lipid content of rice. This opportunity has also helped me improve my laboratory and research skills. This has been a great experience that I feel will assist me in the future.

INTRODUCTION

Milling is a mechanical process during which brown rice is subjected to abrasive or frictional action to remove the germ and bran layers to yield white rice. Due to the high content of oil in bran (up to 24% oil), bran remaining on the kernel after milling can result in off-flavors and odors from oil oxidation. Therefore, it is important that rice be milled sufficiently to remove bran to acceptable levels. The degree of milling (DOM) of rice is the extent to which bran has been removed from rice-kernel surfaces and is important in determining head rice yield (Sun and Siebenmorgen, 1993); viscosity (Perdon et al., 2001); starch gelatinization (Marshall, 1992); and sensory quality (Piggott et al., 1991).

Several methods have been used to estimate DOM, including visual examination, optical measurements, staining techniques, and chemical composition analysis. One commonly used technique for chemical composition analysis is to measure the amount of lipids remaining on the surface of the rice kernel through a petroleum-ether extraction (Watson et al., 1975). A widely used method of petroleum-ether lipid extraction is the conventional Soxtec extraction system. This system is an improvement over previously used systems, such as the Soxhlet system, in terms of saving time, solvent, and labor. For example, the Soxtec extraction system has been found to be as accurate as the conventional Soxhlet system in lipid extraction (Morrison, 1990) with the advantages of reduced extraction durations (often less than 1 h), and lower solvent levels (usually less than 50 mL/sample) compared to the Soxhlet system.

One limitation of the Soxtec manual system is that it requires the presence of the user for manual operation of the lever arm in order for the rinsing step to be completed during extraction. With the increased demand for fast, accurate DOM measurements, alternative methods that are less labor-intensive, use less solvent, are capable of more samples per day, and are more automated than the Soxhlet or Soxtec, have attracted interest. One such extraction system, known as pressurized liquid extraction or more commonly by its trade name, accelerated solvent extraction (ASE), has been utilized to extract oil from several different matrices. Through this system, up to 24 samples can be loaded into the instrument, and using elevated temperatures (up to 200°C) and pressures (up to 3000 psi), extractions are performed quickly in only a small quantity of solvent (<50 mL); also, user

presence is not required to perform any operations on the instrument during the entire extraction process. The completion of 24 samples per day, depending on the extraction duration, can be accomplished at an unhurried pace allowing other laboratory tasks to be finished while the samples are extracting. When compared to conventional methods, i.e., the Soxhlet and Soxtec, this fully automated process produces results in a fraction of the time (< 20 min/sample), with final samples prepared in closed collection vials for further clean-up steps or immediate analysis.

Key parameters to obtaining optimal results with the ASE system are extraction temperature, number of static cycles, and static phases. The static cycle allows the sample to be held for a static or stationary time period in contact with fresh solvent during the extraction process, which aids in maintaining a favorable extraction equilibrium. As the temperature is increased, the viscosity of the solvent is reduced, thereby increasing its ability to wet the matrix and solubilize target analytes. Increasing static phases at elevated temperatures allows compounds of interest to diffuse more quickly into the extraction solvent, thereby enhancing extraction efficiency.

In order to assist the rice industry in providing fast, accurate DOM measurements, this study was conducted to evaluate operating conditions of the ASE system for surface-lipid content (SLC) determination. If the parameters discussed above for the ASE are fine-tuned for accurate and reliable measurements compared to the commonly used Soxtec method, the procedure for quantifying SLC could be standardized and automated for laboratory DOM determination.

MATERIALS AND METHODS

Sampling Techniques

Two long-grain rice cultivars, 'Cypress' and 'Cocodrie', were harvested from the Northeast Research and Extension Center, Keiser, Ark., in September 2002 at moisture contents (MCs) of 17.2 and 18.5% (expressed on a fresh-weight basis), respectively. Immediately after harvest, the rice was cleaned using a dockage tester (Model XT4, Carter-Day Co., Minneapolis, Minn.) and gently dried by placing the rice onto screen trays in a controlled temperature and relative humidity chamber (21°C, 53% RH) to achieve approximately 12% MC. Following drying, a sample of 150 g of rough rice from each cultivar was dehulled using a Satake Rice Machine (Type THU, Satake Engineering Co., LTD, Tokyo, Japan). This was repeated five times in order to obtain sufficient head rice (milled kernels > 75% of original kernel length) quantities for the analysis. The resulting brown rice was milled in a laboratory mill (McGill No. 2, RAPSCO, Brookshire, Tex.) for 30 s. Placing a 1.5 kg weight on the lever arm 15 cm from the middle of the mill chamber controlled the pressure on the rice during milling. Head rice was separated and head rice yields (HRYs) were determined using a Grainman shaker table with a 4.76 mm screen size (Grainman Machinery Mfg., Corp., Miami, Fla.). The HRYs for 'Cypress' and 'Cocodrie' were 70% and 64%, respectively. Head rice samples were placed in plastic freezer bags, purged with nitrogen, and stored at –10°C until subsequent extraction with the Soxtec system and ASE. Thirty-six samples per variety were extracted on the Soxtec system and 54 samples were extracted on the ASE per variety for a total of 180 samples analyzed.

Surface Lipid Extraction

Soxtec. Surface lipids were extracted from head rice using a Soxtec Avanti 2055 Manual Extraction unit (Foss Tecator, Eden Prairie, Minn.) with petroleum ether (ACS grade, Mallinckrodt Baker, Paris, Ky.) as the extracting solvent. Samples were pre-dried prior to extraction on both systems by placing 5 g of head rice into cellulose-extraction thimbles (33 mm i.d. x 80 mm, Foss North America, Inc., Eden Prairie, Minn.) with a defatted cotton plug placed on top of the sample to keep the sample from boiling out, and placed in a convection oven at a constant temperature of 100°C for 1 h (Hogan and Deobald, 1961). Petroleum ether (70 mL) was measured into each extraction cup and the thimble was lowered to immerse the sample in the solvent for two treatment conditions of 15- and 25-min boiling durations. The boiling temperature of the solvent was set on the unit so that three different temperatures of 115°C, 135°C, and 150°C were tested. The thimble was then manually raised by the operator above the solvent surface and rinsed for two durations, 15 or 25 min, by the condensed solvent to extract remaining lipids on the surface of the kernels (Chen and Siebenmorgen, 1997). After rinsing, the solvent flow was discontinued by manual operation and any solvent from the extraction cup was evaporated and collected inside the unit for 5 min. The total extraction length was 30 min and 50 min/sample at each of the three temperature settings. The design of the Soxtec system allowed only six samples to be extracted simultaneously. The extraction cups were dried at 100°C for 30 min to remove any residual petroleum ether, leaving only the dry material, which represented the extracted surface lipids. Following drying, the cups were transferred to a desiccator to cool for 30 min, and the weight of the remaining lipids in the cups was used to calculate SLCs by expressing as a percentage of the original head rice (5 g).

Accelerated Solvent Extractor. Extraction of surface lipids from pre-dried head rice, as described above, was

accomplished by the use of an accelerated solvent extractor (ASE 200, Dionex, Sunnyvale, Calif.). The pressure during extraction was maintained at 1500 psi (10,342 KPa) with temperatures of 115°C, 135°C, and 150°C. Each pre-dried head rice sample (5 g) was placed in an extraction cartridge, loaded onto the carousel holder of the ASE, and extracted for the following durations: two 25-min cycles, two 15-min cycles, and one 30-min cycle (using petroleum ether as the extracting solvent) representing total extraction durations per sample of 50 min, 30 min, and 30 min, respectively. The ASE extracts were collected in 40 mL glass vials and comprised approximately 25 mL of petroleum ether and lipids/sample. After collection, the petroleum ether was evaporated under a nitrogen flow in a laboratory hood until no petroleum ether was detected, and the vials were placed in a drying oven (100°C) for 30 min to evaporate any residual solvent, and transferred to a desiccator to cool for 30 min. The weight of the remaining lipids was used to calculate SLCs by expressing as a percentage of the original head rice sample (5 g). In order to determine significant differences among extraction temperatures, durations, and extraction systems for each rice variety, a Student's t-test $p<0.05$ using one-way analysis of variance (JMP IN 5.0., Cary, N.C.) was conducted.

RESULTS AND DISCUSSION

The extraction duration of 50-min generally produced SLCs greater than the 30-min extraction duration for 'Cypress' and 'Cocodrie' rice at all temperatures (Table 1). However, at the highest extraction temperature, 150°C, the Soxtec produced significantly lower SLCs for both 'Cypress' and 'Cocodrie' (0.46% and 0.43%, respectively) at the longest extraction duration of 50-min compared to the 30-min duration.

Comparing total extraction duration with the ASE system for 'Cocodrie' indicated that the total extraction length of 30 min (one 30-min cycle) generally resulted in similar or greater SLCs than the 50 min total extraction duration. This would imply that a complete surface-lipid extraction could be accomplished with the extraction duration of 30 min when using ASE. Comparing static cycles (one 30-min versus two 15-min cycles) on the ASE to determine if one cycle is as efficient as two consecutive cycles, results indicated that one 30-min cycle gave comparable or greater SLCs than two 15-min cycles (Table 2) except for 'Cocodrie' at 150°C. This would suggest that the total extraction duration might be more influential in increasing or decreasing the extraction efficiency than the number of static cycles. The extraction temperature of 150°C on the ASE produced significantly lower SLCs than the other extraction temperatures at all extraction durations, except for 'Cocodrie' at two 15-min cycles. These results concur with a study conducted on pressurized liquid extraction of medicinal plant extracts in which the extraction efficiency increased with increasing temperature up to a specific point, 120°C, and then declined when extracted at a higher temperature, 140°C (Ong et al., 2000). Generally, a higher temperature had more impact on lowering SLCs than the number of static cycles did when extracting surface lipids by ASE for both 'Cypress' and 'Cocodrie'. It could be reasoned then that one 30-min extraction cycle at temperatures 135°C or lower produces maximum SLCs when using the ASE system.

Table 3 shows the average SLCs of 'Cypress' and 'Cocodrie' compared between the two extraction systems for the 50-min extraction length, which is also illustrated more dramatically in Fig. 1, and then compared between the Soxtec at 50-min extraction and the ASE at 30 min (one 30-min cycle). The 50 min extraction duration was chosen since it was a better comparison between the two systems due to the 25-min boiling and 25-min rinsing with the Soxtec system that would be analogous to two 25-min extraction cycles when using the ASE system. Overall, it was observed that SLCs when extracted by ASE were equivalent to or greater than those obtained by the Soxtec system for both 'Cypress' and 'Cocodrie' (Fig. 1). This is similar to findings of other investigators who noted that when measuring polychlorinated biphenyl from various spiked organic matrices, the ASE had comparable to or slightly higher extraction efficiencies than those obtained by Soxhlet (Abraha and Raghavan, 2000). Wang et al. (1999) found polycyclic aromatic-hydrocarbon recoveries from several biological samples by the ASE method were comparable to or better than those obtained by Soxhlet extraction. In addition, using the ASE system reduced the extraction time by 20 min per sample since it produced SLCs with a 30-min extraction as great as the Soxtec system which produced acceptable SLCs with a 50-min extraction. Although 'Cocodrie' was not included in the 30-min ASE vs. the 50-min Soxtec comparison, the SLCs produced were similar or greater when using ASE at the 30-min extraction duration (Table 2) compared to SLCs produced using the Soxtec at the 50-min extraction duration (Table 1). Also notable was the reduction in solvent consumption between the two systems. For example, ASE used approximately 25 mL solvent/sample and the Soxtec system 70 mL/sample.

In our current study, overall the ASE provided surface-lipid determinations that were as reliable as are those obtained by the Soxtec system. This would suggest that the ASE is as thorough in extracting surface lipids as are commonly used conventional methods for DOM

determinations with the former offering advantages of shorter extractions, full automation, reduction in the amount of organic solvents required for extraction, and less handling required by the operator.

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LITERATURE CITED

Abrha, Y. and D. Raghavan. 2000. Polychlorinated biphenyl (PCB) recovery from spiked organic matrix using accelerated solvent extraction (ASE) and Soxhlet extraction. J. Hazard Mater. 80:147-157.

Chen, H. and T.J Siebemorgen. 1997. Effect of rice kernel thickness on degree of milling and associated optical measurements. Cereal Chem. 74:821-825.

Hogan, J.T. and J.H. Deobald. 1961. Note on a method of determining the degree of milling of whole milled rice. Cereal Chem. 38:291-293.

Marshall, W.E. 1992. Effect of degree of milling of brown rice and particle size of milled rice on starch gelatinization. Cereal Chem. 69:632-636.

Ong, E-S., S-O. Woo, and Y.-L. Yong. 2000. Pressurized liquid extraction of berberine and aristolochic acids in medicinal plants. J. Chromat. A. 313:57-64.

Perdon, A.A., T.J. Siebenmorgen, A. Mauromoustakos, V.K. Griffin, and E.R. Johnson. 2001. Degree of milling effects on rice pasting properties. Cereal Chem. 78:205-209.

Piggot, J.R., W.R. Morrison, and J. Clyne. 1991. Changes in lipids and in sensory attributes on storage of rice milled to different degrees. Int. J. Food Sci. Technol. 26:615-628.

Sun, H. and T.J. Siebenmorgen. 1993. Milling characteristics of various rough rice kernel thickness fractions. Cereal Chem. 70:727-733.

Wang, G., A.S. Lee, M. Lewis, B. Kamath, and R.K. Archer. 1999. Accelerated Solvent Extraction and Gas Chromatography/Mass Spectrometry for Determination of Polycyclic Aromatic Hydrocarbons in Smoked Food Samples. J. Agric. Food Chem. 47:1062-1066.

Watson, C. A., E. Dikeman, and R.A. Stermer. 1975. A note on surface lipid content and scanning electron microscopy of milled rice as related to degree of milling. Cereal Chem. 52:742-747.

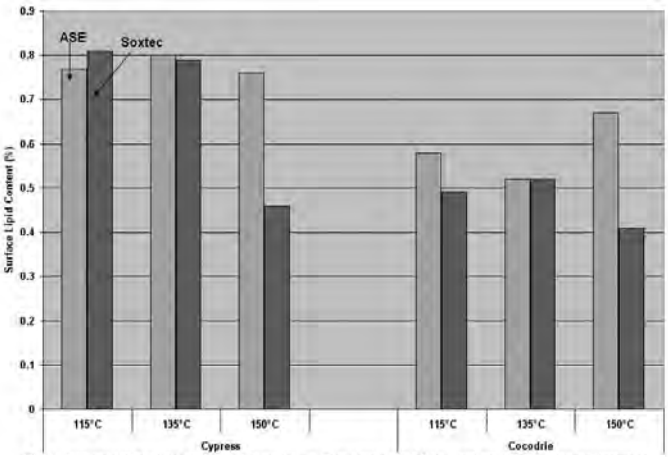

Fig. 1. Average surface-lipid contents of 'Cypress' and 'Cocodrie' rice at 50-min total extraction compared between ASE and Soxtec.

Table 1. Average surface-lipid contents (% of original head rice mass) of 'Cypress' and 'Cocodrie' rice using the Soxtec extraction system.

Temperature (°C)	Cypress		Cocodrie	
	Extraction durations			
	30 min[z]	50 min	30 min	50 min
115[y]	0.54Bb	0.81Aa	0.45Bb	0.49Ab
135	0.62Bab	0.76Aa	0.46Bb	0.52Aa
150	0.77Aa	0.46Bb	0.49Aa	0.43Bc

[z] Values between extraction durations within each temperature for each rice cultivar with different capital letters are significantly different ($p \leq 0.05$) by student t-test.

[y] Values within each rice cultivar for each extraction duration with different lowercase letters are significantly different ($p \leq 0.05$) by student's t-test.

Table 2. Average surface-lipid contents (% of original head rice mass) of 'Cypress' and 'Cocodrie' rice using accelerated solvent extraction.

Temperature (°C)	Cypress[z]			Cocodrie		
	Cycles[y]					
	1 30-min	2 15-min	2 25-min	1 30-min	2 15-min	2 25-min
115	0.70Aa	0.78Aa	0.80Aa	0.77Aa	0.63ABa	0.59Ba
135	0.81ABa	0.73Ba	0.84Aa	0.79Aa	0.61ABa	0.53Bb
150	0.45Bb	0.38Bb	0.76Ab	0.54Bb	0.61Aa	0.52Bb

[z] Values within each temperature for extraction cycles of each rice cultivar with different capital letters are significantly different ($p<0.05$) by students t-test.

[y] Values within each extraction cycle for each rice cultivar with different lowercase letters are significantly different ($p<0.05$) by student's t-test.