Extraction of Rice Bran Oil Using Supercritical Carbon Dioxide and Propane

Darrell Sparks[a], Rafael Hernandez[a,]*, Mark Zappi[b], Dean Blackwell[c], and Trey Fleming[c]

[a]Mississippi State University, Dave C. Swalm School of Chemical Engineering, Mississippi State, Mississippi 39762,
[b]University of Louisiana–Lafayette, College of Engineering, Lafayette, Louisiana 70504, and [c]AgraPure, Jackson, Mississippi 39288

ABSTRACT: Extraction of rice bran lipids was performed using supercritical carbon dioxide (SC-CO_2) and liquid propane. To provide a basis for extraction efficiency, accelerated solvent extraction with hexane was performed at 100°C and 10.34 MPa. Extraction pressure was varied for propane and SC-CO_2 extractions. Also, the role of temperature in SC-CO_2 extraction efficiency was investigated at 45, 65, and 85°C. For the SC-CO_2 experiments, extraction efficiencies were proportional to pressure and inversely proportional to temperature, and the maximal yield of oil achieved using SC-CO_2 was 0.222 ± 0.013 kg of oil extracted per kg of rice bran for conditions of 45°C and 35 MPa. The maximal yield achieved with propane was 0.224 ± 0.016 kg of oil per kg of rice bran at 0.76 MPa and ambient temperature. The maximum extraction efficiencies of both SC-CO_2 and propane were found to be significantly different from the hexane extraction baseline yield, which was 0.261 ± 0.005 kg oil extracted per kg of rice bran. A simulated economic analysis was performed on the possibility of using SC-CO_2 and propane extraction technologies to remove oil from rice bran generated in Mississippi. Although the economic analysis was based on the maximal extraction efficiency for each technology, neither process resulted in a positive rate of return on investment.

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KEY WORDS: Bran, lipid extraction, propane, rice bran, rice bran oil, solvent extraction, supercritical, supercritical carbon dioxide, supercritical fluid extraction, vegetable oil extraction.

Globally, over 500 million metric tons of rice is produced every year (1). When rice is milled to produce white rice, the outer layers of the rice kernel are removed. These layers include the hull, the germ, and the bran, which includes the testa, pericarp, nucellus, and aleurone layer (2). Typically, the rice bran comprises about 8% of rice paddy, and thus, approximately 40 million metric tons of rice bran is generated annually worldwide (3). The state of Mississippi generates approximately 9091 kg (20,000 lb) of rice bran per year. A large fraction of this rice bran is disposed of in landfills because it becomes rancid. Another fraction is sold as animal feed at approximately \$65/ton (\$72/metric ton). Depending on rice type and applied stabilization technique, rice bran contains on average 10–23% oil (4). For example, parboiling is one of the oldest and most commonly practiced stabilization methods, and parboiled rice bran has higher lipid levels than unstabilized rice bran (4,5). Rice bran oil consists largely of saponifiable compounds (90–96%), especially TG (86–89%) (6).

Oil is usually separated from rice bran by using solvent extraction, and hexane is the extraction solvent most commonly used. Hexane is relatively inexpensive (\$1.15/gal = \$0.304/L) and excellent for extraction of nonpolar lipids (7). However, it has a high volatility and is considered toxic to animals and humans at relatively low concentrations. Hexane vapors need to be monitored during industrial oil extraction operations because uncontrolled vapors could lead to explosions. Additionally, if the oil and defatted meal are to be used for animal feed, expensive and time-consuming processes, such as distillation, have to be used to remove the hexane residue completely (8).

Alternative extraction solvents include supercritical carbon dioxide (SC-CO_2) and compressed gases. Carbon dioxide is an attractive solvent because it is nonflammable, nontoxic, nonexplosive, and inexpensive (9). A major advantage of CO_2 is its relatively low critical temperature of 31°C (10). Extraction of oils at this temperature minimizes the thermal degradation of proteins, antioxidants, and other nutritionally valuable components. The critical pressure of CO_2 is 7.38 MPa (10). In supercritical fluid extraction, both the temperature and pressure can be controlled to modify solvent physical properties such as density, diffusivity, and viscosity (8). Control of these physical properties could result in the improvement of overall extraction efficiency and/or the selectivity and yield of specific compounds.

Several studies have compared the extraction of rice bran oil with SC-CO_2 and with hexane. Kuk and Dowd (11) obtained a maximal oil yield of 20.4% using SC-CO_2 at 62 MPa and 100°C and a yield of 20.5% oil with hexane at 69°C and 0.101 MPa. In another study, SC-CO_2 (at 30 MPa and 35°C) and hexane oil yields were 17.98 and 20.21%, respectively (8). In that study, SC-CO_2 extraction of rice bran using ethanol as a co-solvent was also investigated. The addition of 5 wt% ethanol to the solvent stream resulted in a yield of 18.23% under extraction conditions of 30 MPa and 35°C (8). Hence, the addition of a polar modifier was shown to increase oil yield by extracting more compounds than carbon dioxide alone. In general, rice bran oil extraction efficiency using SC-CO_2 compares favorably with hexane. The main disadvantage of SC-CO_2 is the relatively high operating pressures, which could result in high capital, operating, and maintenance costs. Also, SC-CO_2

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*To whom correspondence should be addressed at Mississippi State University, Dave C. Swalm School of Chemical Engineering, Swalm Bldg., Mississippi State, MS 39762. E-mail: rhernandez@che.msstate.edu

can selectively extract lipid components based on solubility differences. Prior studies showed that the solubilities of FA and glycerides in SC-CO_2 can be changed based on the density and temperature of the SC-CO_2 (12,13).

Another alternative to hexane for the extraction of oils is the use of compressed gases, such as propane and butane. Propane has several advantages over both hexane and CO_2. First, propane is relatively inexpensive and does not leave a toxic residue. Second, the pressures involved in oil extraction using propane are at least an order of magnitude (hundreds of psi compared with thousands of psi) lower than those in SC-CO_2 extraction. Several commercial processes have used propane as an extraction solvent. One example is the Solexol process. This process has been used to extract menhaden and soybean oils effectively, decolorize tallow, and separate lubricating oils from crude petroleum residues (14–16). Similar to supercritical fluid extraction, in the Solexol process, the pressure and temperature are controlled to separate desired products. Previous research on propane fractionation of menhaden oil showed that saturated lipids could be preferentially extracted from unsaturated lipids; however, the operating conditions implemented were not provided in that study (17).

This study compares rice bran oil extraction efficiencies obtained by using hexane, SC-CO_2, and propane. Efficiencies were based on oil yield and oil composition. Additionally, a preliminary economic analysis was performed to compare the feasibility of using propane or SC-CO_2 instead of hexane to generate rice bran oil. The rice bran protein remaining in the bran after extraction has a very high nutritional value and is used as a hypoallergenic food ingredient (18). Therefore, the economic analysis also includes potential profits generated from the rice bran protein.

EXPERIMENTAL PROCEDURES

Parboiled stabilized rice bran with approximately 8% moisture was used for all extraction tests. The rice bran was provided by a rice mill located in Greenville, Mississippi. The moisture was determined using IR heating (Model MB45; Ohaus, Pine Brook, NJ). To establish a baseline for oil yield, rice bran was extracted with hexane at 100°C and 10.34 MPa using an accelerated solvent extractor (ASE) manufactured by Dionex (Model ASE 200; Salt Lake City, UT). This extraction was performed in triplicate. Approximately 40 g of rice bran was placed in stainless steel vials and mixed with hydromatrix (sieved diatomaceous earth). The hydromatrix adsorbs the moisture from the bran. This bulk adsorbent is manufactured by Varian, Inc. (Palo Alto, CA). Then, the stainless steel vials were placed in the automated sampler, which facilitated the introduction of each vial into the oven and connection to the solvent line. The vials were filled with solvent and then heated over a period of 5 min to 100°C. The vials were kept at 100°C and 10.34 MPa for a 5-min static interval. At the end of the static period, fresh solvent was used to flush the vials, and then each vial was purged and depressurized. A Dionex flow controller with a capability for four solvents controlled the solvent flow into each vial. Then the hexane extract was collected and analyzed to determine yield and FA profile.

The SC-CO_2 extractions were performed with a Thar SFE-100 unit (Thar Technologies, Pittsburgh, PA). Extractions in triplicate were conducted at 45, 65, and 85°C and 20, 30, and 35 MPa. For each extraction, 40 g of rice bran was placed in the 100-mL sample vessel. The inner heating element of the sample cell was used to heat the rice bran to the desired extraction temperature. Then SC-CO_2 was allowed to flow through the sample vessel at the prescribed temperature and pressure for a period of 30 min at a flow rate of 25 g/min. During the experiment, the CO_2 and extracted oil passed through a cyclone, where the CO_2 was separated from the oil, allowing the oil to collect in a product vessel. Next, 20 mL of hexane was used to wash the product vessel to ensure collection of any residual oil. Then hexane was distilled from the rice bran oil, and oil yields were determined using a gravimetric method. Finally, the rice bran raffinate was collected and analyzed for protein, fat, fiber, and carbohydrate.

A low-carbon steel propane extractor was provided by AgraPure, Inc. (Jackson, MS). All the extractions were conducted in triplicate at ambient temperature in an explosion-proof hood. The controlled parameters in the experiments were the propane charged into the extractor per unit mass of rice bran and pressure (0.62, 0.69, and 0.76 MPa). For each extraction, 250 g of rice bran was placed in the sample vessel. Then, the two halves of the vessel were bolted together tightly, and the initial weight of the propane extractor with rice bran was recorded. Liquid propane was charged into the extraction vessel to the desired pressure, and the head space of the extraction vessel was purged to 0.41 MPa.

After the final weight of the extraction vessel with rice bran and propane was recorded, the extraction vessel was agitated by turning the vessel clockwise and counterclockwise five times in each direction. Then the bottom valve of the extractor was opened so that liquid propane with extracted oil could flow into a tared 2000 mL beaker. Since propane boils at approximately –42°C at atmospheric pressure, the propane began immediately to separate from the rice bran oil. Once the pressure in the vessel reached atmospheric pressure, the rice bran oil was allowed to sit for 3 h to allow the propane to boil off. The rice bran oil yield was determined using a gravimetric method, and the rice bran raffinate was collected and analyzed for proteins, fats, fibers, and carbohydrates.

The rice bran oil and rice bran raffinate collected from each of the examined extraction techniques were analyzed to determine composition. To quantify FA, the rice bran oil was derivatized into FAME and analyzed using an Agilent gas chromatograph (Model 6890; Palo Alto, CA) with a FID. The separation was achieved with a fused-silica capillary column composed of stabilized poly(90% biscyanopropyl/10% cyanopropylphenyl siloxane) (SP-2380; Supelco, Bellefonte, PA). The dimensions of the column were 100 m × 0.25 mm × 0.2 μm. A calibration curve was prepared by injecting known concentrations of an external standard mixture composed of 37 FAME (Supelco). 1,3-Dichlorobenzene was used as an internal standard. The method consisted of injecting 1 µL of sample into the gas chromatograph with a split ratio of 100:1. The temperature program began at 110°C and ended at 240°C over a nonlinear temperature gradient of 99 min. Lipid classes were determined using HPLC equipped with an ELSD. This analysis was conducted by Avanti Polar Lipids (Alabaster, AL). The amount of protein, ash, moisture, fiber, fat, and carbohydrate in the rice bran raffinate was determined by the Mississippi State Chemical Laboratory (Mississippi State, MS). In particular, protein was determined using a LECO FP-528 that utilized the Dumas method of combustion as opposed to the traditional Kjeldahl digestion method to determine the amount of protein.

RESULTS AND DISCUSSION

A baseline yield of 0.261 ± 0.005 kg of oil extracted per kg of rice bran was achieved using accelerated solvent extraction (ASE) with hexane. As mentioned previously, rice bran typically contains 10–23% oil. Therefore, the rice bran used in this study has a higher oil content than average, which is probably due to the parboiling of the rice bran.

The yields resulting from all the propane extraction experiments are displayed in Figure 1. In general, as the extraction pressure increases and as the amount of propane used increases, more oil is extracted per kg of rice bran. At about 0.2 kg of oil extracted per kg of rice bran, the graph begins to level off. When the yields are grouped based on the amount of propane used, the highest yield achieved was 0.224 ± 0.016 kg oil extracted per kg of rice bran at conditions of 0.76 MPa and 1.58 kg of propane.

The results obtained for the SC-CO_2 extractions also are presented in Figure 1. A maximal yield of 0.222 ± 0.013 kg of oil extracted per kg of rice bran was obtained at 35 MPa and 45°C. Extraction efficiencies were directly proportional to pressure and inversely proportional to temperature. Depending on the solute, solubility of lipids has been shown to increase with increasing temperature (12,13). However, SC-CO_2 is a compressible fluid at the evaluated pressures (<35 MPa) At

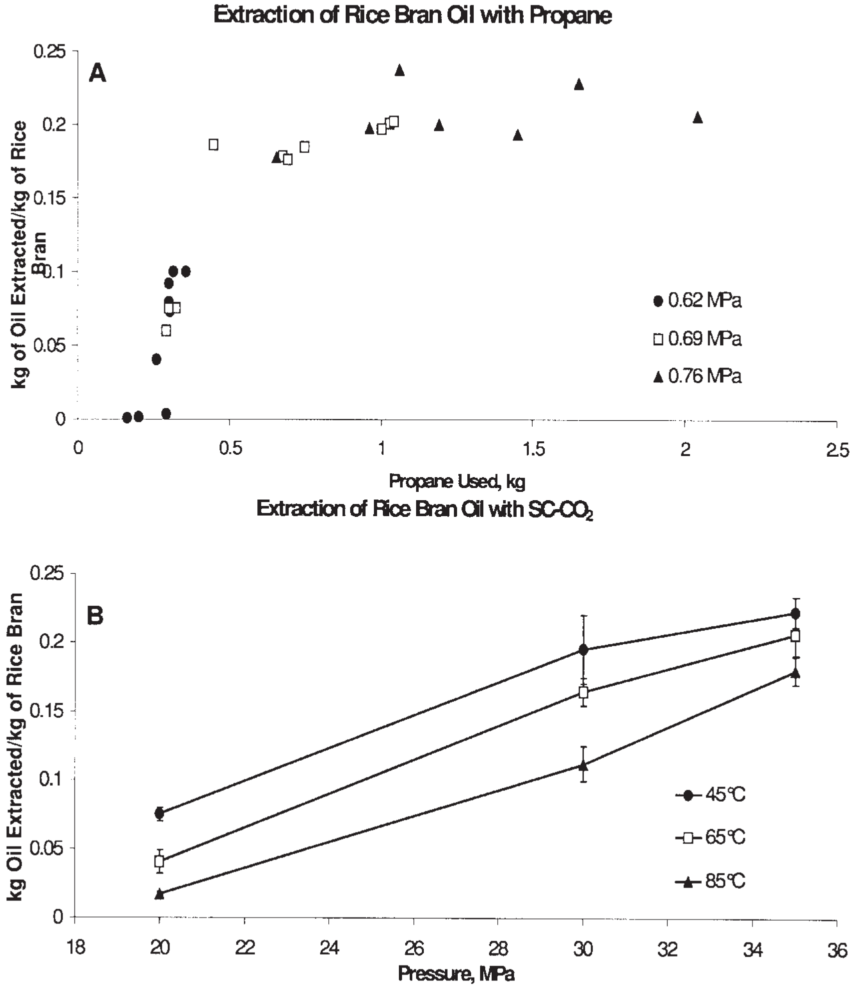

FIG. 1. (A) Propane extraction yields based on the amount of propane used at 0.62, 0.69, and 0.76 MPa and (B) supercritical CO_2 (SC-CO_2) extraction yields based upon pressure at 45, 65, and 85°C. The error bars indicate mean ± 1 SD (*n* = 3).

these conditions, the solubility is controlled by the SC-CO_2 density, which is maximized at relatively low temperature and high pressure.

The extraction results were analyzed using SAS 9.1 (SAS, Cary, NC) to determine whether any significant differences existed between the maximal extraction yields for the three solvents used. All statistical analyses were performed at a 0.05 level of significance. The results showed that the extraction efficiencies obtained with propane and SC-CO_2 significantly differed from hexane. Although propane provided a slightly higher yield than SC-CO_2 within the parameters of this study, the propane and SC-CO_2 yields were not significantly different. At higher pressures and temperature, SC-CO_2 extraction could possibly result in a higher oil yield compared with propane's 0.224 ± 0.016 kg of oil per kg of rice bran at 0.76 MPa.

As previously mentioned, it is possible to achieve some selectivity for particular compounds by extracting with SC-CO_2 and with propane at different temperatures and pressures. To evaluate this selectivity effect, the lipid class profiles were determined for the SC-CO_2 and propane extracts. These profiles are shown in Figure 2. In each case, only three lipid classes were detected: TG, FFA, and cholesterol. The lipid profile of oil extracted with SC-CO_2 at 45°C and 35 MPa is almost identical to that of oil extracted with propane at 0.76 MPa and 1.58 kg of propane. However, SC-CO_2 extraction at 85°C and 20 MPa yielded fewer TG and more FFA than SC-CO_2 extraction at 45°C and 35 MPa.

The apparent observed selectivity toward FFA at 85°C and 20 MPa could be explained by the increase in solubility with increasing temperature that some lipids, such as oleic acid, can experience (12). The apparent FFA selectivity also could have been caused by the conversion of TG to FFA *via* oxidation. A small concentration of oxygen is introduced into the SC-CO_2 test chamber during rice bran loading. Since oxygen is completely miscible with SC-CO_2, the mass transfer limitations for the reaction of oxygen with TG are eliminated and the reaction rate is accelerated at the relatively high temperature (85°C) of the experiment. Another reaction that can be accelerated because of the high temperature is the hydrolysis of TG. The moisture contained in rice bran (8%) could participate in this reaction.

Oil extracted by 1.58 kg of propane at 0.76 MPa showed the same distribution of lipids as oil extracted at 0.62 MPa with 0.16 kg of propane, so within the parameters of this study, propane does not provide any lipid class selectivity or lipid transformation. The oil extractions are performed using liquid propane. Potential oxidation reactions are limited by the transfer of oxygen from the gas phase into the liquid phase and the relatively low temperature (20°C). In the case of hydrolysis, reactions could be limited by poor mixing and low temperature.

FA analysis was performed on the hexane extract and the maximum and minimum yield extracts of propane and SC-CO_2. Figure 3 compares the FA profiles of the propane and SC-CO_2 extracts to the hexane extract. The FA profile of oil extracted with 1.58 kg of propane at 0.76 MPa is similar to the profile of oil extracted with 0.16 kg of propane at 0.62 MPa. Both of these profiles are also similar to oil extracted with hexane at 100°C and 10.34 MPa. This similarity is expected when the Hansen solubility parameters of the three solvents are compared, as shown in

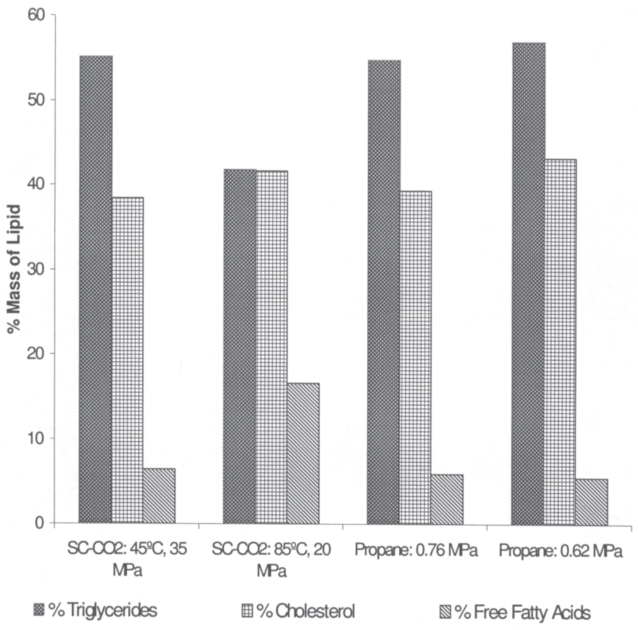

FIG. 2. Lipid class profiles of propane and SC-CO_2 extracts. For abbreviation see Figure 1.

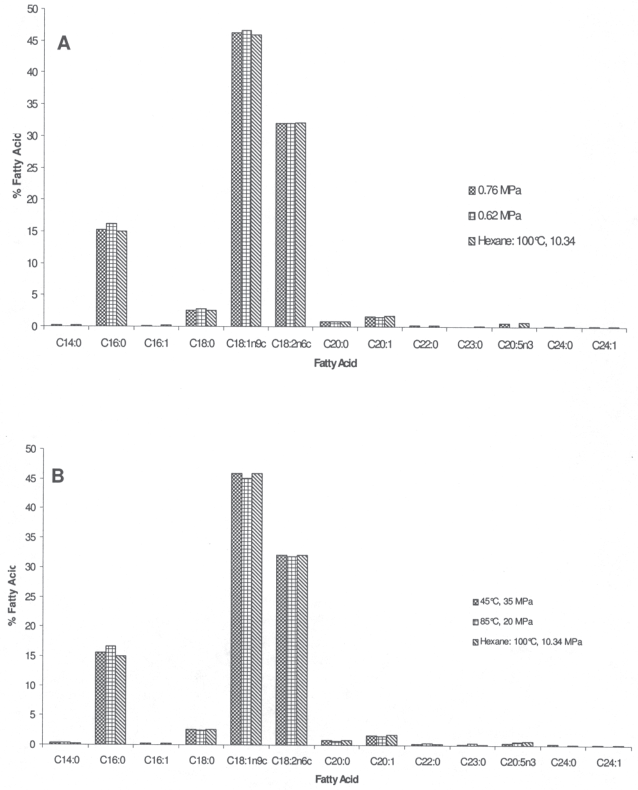

FIG. 3. FA analysis of (A) propane extracts and (B) SC-CO_2 extracts. For abbreviation see Figure 1.

TABLE 1
Hansen Solubility Parameters and Molar Volumes of Solvents

Solvent	Dispersion contribution[a] (MPa$^{0.5}$)	Polar contribution[a] (MPa$^{0.5}$)	Hydrogen bonding contribution[a] (MPa$^{0.5}$)	Molar volume[b] (cm^3/mol)
Hexane	14.90	0	0	143.5
Propane	13.10	0	0	88.2
SC-CO_2	12.14	6.9	4.1	47.9

[a] Based on data from Reference 19.
[b] Based on data from Reference 20. SC-CO_2, supercritical carbon dioxide.

Table 1. The Hansen solubility parameters take into account dispersion forces, permanent dipole–permanent dipole forces, and hydrogen bonding forces. Both hexane and propane are nonpolar molecules that do not participate in hydrogen bonding, so they only have a dispersion force contribution term. Because rice bran is composed primarily of nonpolar lipids, the primary solute–solvent interaction is due to dispersion forces. Since hexane and propane have similar Hansen dispersion force values, the same FA are extracted for each solvent. However, SC-CO_2 has a quadrupole moment and can participate in hydrogen bonding. Hence, for SC-CO_2, there are contributions from dispersion, polar, and hydrogen-bonding interactions. Therefore, it would be expected that SC-CO_2 would show a different lipid extraction profile from propane and hexane. However, the molar volume of each solvent must also be considered. As shown in Table 1, the molar volume of SC-CO_2 is much less than hexane and propane, and molecules with a lower molar volume provide better solvation than molecules with similar Hansen parameters and greater molar volumes. Therefore, the difference in the Hansen parameters of SC-CO_2 compared with propane and hexane is compensated by having a lower molar volume. Over 90% of the FA are composed of oleic acid (C18:1, 46%), linoleic acid (C18:2, 32%), and palmitic acid (C16:0, 15%). Although some selectivity was achieved with SC-CO_2 in the area of lipid classes, no real selectivity was achieved in the area of individual FA. The distribution of FA is the same as with propane, with over 90% of the FA being oleic acid (46%), linoleic acid (32%), and palmitic acid (15%).

Another source of value from the rice bran is the protein fraction. Once the oil has been removed from the rice bran, the relative amount of protein in the rice bran will increase. Table 2 reveals the effect of extraction on the amount of protein remaining in the rice bran raffinate. In raw rice bran the protein content is approximately 15%, but, as expected, removing the oil increases the protein fraction to approximately 20%. Once defatted, the rice bran can be sold as a low-fat, protein-rich animal feed, or the protein fraction can be separated from the rice bran and sold as a value-added product.

Extraction economics. An economic analysis was performed for hexane, propane, and SC-CO_2 extraction using CAPCOST, a capital cost estimation software (21). Each case was based on the maximal yield achieved for each type of extraction. The following parameters were taken into account: raw material cost of rice bran and solvent, capital investment of equipment, cost of utilities, cost of labor, and revenue generated from rice bran oil and rice bran protein. The following assumptions were used for all cases: tax rate of 35%, interest rate of 5%, no land cost, modified accelerated cost recovery system (MACRS) depreciation over 10 yr, period of construction of 2 yr with 60% of the capital being invested during the first year, a salvage value of $1, and 90% solvent recycle. The extraction unit would operate for 8322 h per year. Three shifts with one laborer per shift would operate the unit at a wage of $29.62/h. The market price of rice bran oil was taken to be $3.08/kg, and the price of rice protein was assumed to be $4.928/kg (7).

In order to perform the economic analysis, several quantities had to be either calculated from the extraction results or estimated. An estimated quantity was the cost of rice bran protein fractionation, which was based on the pilot scale protein concentration work by Connor *et al.* (22). In that study, 18.18 kg of rice bran was mixed with 90.91 kg water and approximately 2 kg of 3 N NaOH. For purposes of economic evaluation, deionized water ($1.00/1000 kg) was used. The price of solid sodium hydroxide was taken to be $427.64/1000 kg. The solvent efficiency, which is the amount of solvent required per mass of rice bran to reach the maximal yield, was calculated from the extraction results. Table 3 provides the prices of the raw materials of all the experiments and gives the solvent efficiency for each solvent. It should be noted that solvent efficiency increases as the amount of solvent required per mass of rice bran decreases. Liquified CO_2 operating under supercritical conditions showed the best solvent efficiency and was the cheapest solvent considered for this study. Although hexane and propane were the same price, the solvent efficiency of propane is lower.

TABLE 2
Rice Bran Raffinate Composition[a]

	Raw rice bran	SF-CO_2: 45°C, 35 MPa	SF-CO_2: 85°C, 20 MPa	Propane: 0.76 MPa	Propane: 0.62 MPa
			% Mass of sample		
Protein	14.6	20.3	15.6	19.4	15.0
Ash	19.0	25.5	20.3	22.2	18.8
Moisture	9.4	6.7	5.3	11.2	8.9
Fiber	43.3	52.0	46.6	50.6	44.7
Fat	24.8	3.4	23.6	3.9	23.0

[a] For abbreviation see Table 1.

TABLE 3
Material Cost and Solvent Efficiency

Material	Price ($/kg)	Solvent efficiency (kg of solvent/kg of rice bran)
Rice bran	0.0715	—
Hexane	0.484	2.8
Liquified CO_2	0.352	0.082
Compressed propane	0.484	6.69

The SC-CO_2 and propane economic studies are very similar because assumptions about propane extraction capital costs were estimated from SC-CO_2 costs. The SC-CO_2 study is based on the treatment of 438.9 kg (1152 L) of rice bran per day, which would yield 97.65 kg (28.35 gal) of rice bran oil per day. The equipment used for this extraction would utilize two extraction vessels, each 12 L in size. The unit would treat 48 L of rice bran per hour. A capital investment of $225,000 would be required for the SC-CO_2 unit. The propane study is also based upon the treatment of 438.9 kg of rice bran at a rate of 48 L of rice bran per hour. However, since the maximum yield of propane extraction (0.224 ± 0.016 kg of oil per kg of rice bran) is slightly higher than SC-CO_2 extraction (0.222 ± 0.013 kg of oil per kg of rice bran), 98.31 kg (28.54 gallons) of rice bran oil would be extracted per day with propane. Since information could not be obtained on the capital investment required for the propane extraction unit, the equipment cost was assumed to be $190,000 (or about 85%) of the SC-CO_2 capital investment. The capital investment required for propane extraction should be lower than that required for SC-CO_2 extraction because the unit would have to withstand lower operating pressures.

The economic assessment of hexane extraction of rice bran was based on the treatment of 408 kg of rice bran per day to generate 106.48 kg (30.96 gal) of rice bran oil per day. The amount of rice bran processed each day with hexane was less than that of propane and SC-CO_2 because the capacity of the hexane extractor was smaller. The yield of rice bran oil was based on ASE, but the economics were based on a traditional solvent extraction unit that operates near atmospheric pressure. The capital investment required for the extraction equipment was $220,000.

Table 4 summarizes the results of the economic analysis and shows that none of the extraction methods is profitable. Although propane extraction had a lower capital investment and a slightly higher revenue than SC-CO_2, the manufacturing cost of propane extraction was higher. This can be attributed to the solvent efficiency that was mentioned earlier. Because more propane per kg of rice bran is required than in the case of SC-CO_2, the raw material cost of propane is higher. The experimental results showed that both propane and SC-CO_2 significantly differ from hexane in their ability to extract oil from rice bran. Although none of the evaluated technologies was economically feasible, further research is recommended using an industrial scale process. By increasing capacity, "economies of scale" can have an effect by lowering the cost per unit of rice bran.

TABLE 4
Economic Analysis Results of Extraction Processes

Economic parameter	Hexane extraction	Propane extraction	SC-CO_2 extraction[a]
Capital investment ($)	220,000	190,000	225,000
Manufacturing cost ($/(yr–kg rice bran))	5.18	4.99	4.67
Raw material cost, ($/(yr–kg rice bran))	0.22	0.40	0.08
Operating labor ($/h)	29.62	29.62	29.62
Revenue ($/(yr–kg rice bran))	1.51	1.41	1.40
DROROI[a], %	—	—	—
Disc. payback period (yr)	—	—	—

[a]DROROI, discounted rate of return on investment; for other abbreviation see Table 1.

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