Natural Refining of Extruded-Expelled Soybean Oils Having Various Fatty Acid Compositions

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ABSTRACT: Simple, low-capital-investment oil refining techniques, which may also meet the needs of natural or organic food industries, were explored to process extruded-expelled (E-E) soybean oils with various fatty acid compositions. Most settled E-E oils are naturally low in phosphatides (<100 ppm phosphorus) and were easily water degummed to low phosphorus levels (<55 ppm). Free fatty acids were reduced to 0.04% by adsorption with 3% Magnesol®. Magnesol reduced residual phosphorus contents to negligible levels. This material also adsorbed primary and secondary oil oxidation products. Our adsorption refining procedure was much milder than conventional refining, as indicated by little formation of primary and secondary lipid oxidation products and less loss of tocopherol. The remaining challenge to effective natural refining is the removal of off-flavor components. Our adsorption treatment reduced the natural flavor of soybean oil but flavor was still present, probably too strong for many consumers. Polyunsaturated oils oxidized more easily than did the other types of oils; therefore, precautions should be taken when refining such oils. High-oleic soybean oil, on the other hand, had excellent oxidative stability and better flavor characteristics after degumming and adsorption with Magnesol compared with other oils.

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KEY WORDS: Adsorption, extrusion-expelling, low-cost refining, mechanical extraction, modified fatty acid composition, natural refining, refining, soybean oil.

A growing number of mini soybean-crushing mills [5–20 metric tons per day (mt/d) processing capacity] have been built as farmer-owned cooperatives or on farms to process locally produced soybeans or other oilseeds (74 plants in North America as of spring 1999). These mini soybean plants employ recently developed extrusion-expelling (E-E) technology now marketed by Insta-Pro International, Triple "F," Inc. (Des Moines, IA). E-E uses a dry autogenous extruder in which heat is generated by friction prior to screw pressing (1,2). It is convenient and economical to have local suppliers of soybean meal for livestock feeders. Also, there is growing interest in natural, organic, and identity-preserved nongenetically modified (GM) or genetically enhanced (GE) soybean products (modified by either transgenic means or traditional breeding). Various types of GE soybeans are being developed to improve nutritional and functional properties of oil, such as beans containing a higher percentage of oleate or lower percentage of linolenate or saturates compared to commodity product, or beans containing no lipoxygenase. Oils from these seeds may command higher prices than commodity oil. These market opportunities cannot be easily served by today's large solvent extraction plants (typically 3,000 mt/d), but the small E-E plants are eager to adopt such strategies to capture greater returns, provided little capital investment is required and the process technology is relatively simple.

Solvent-extracted oil is considered to be chemically treated because of the exposure to hexanes during extraction and the use of subsequent extensive refining with chemicals under harsh conditions, including neutralization with alkali and high-temperature deodorization (which causes isomerization). E-E, on the other hand, is completely mechanical, and the settled crude oil contains low levels of phosphatides and free fatty acids (FFA) (3-5) and has a nutty roasted flavor. The oil, as well as the meal, has potential to be further refined or processed by natural or physical means to produce an array of value-added soybean products.

The objectives of the present research were to assess options for natural and low-capital-investment methods to refine E-E oils with normal and GE fatty acid compositions. We believe that some GE soybeans may be more suitable for natural refining and produce different oil flavors than the commodity soybeans.

EXPERIMENTAL PROCEDURES

Soybean sources. Three soybean lines with GE fatty acid compositions were obtained from Optimum Quality Grains (Des Moines, IA): high-oleic acid (HO) line, A233HO, containing 79.2% oleic acid; a low saturated fatty acid (LS) line, P92B72, containing 8.4% saturated fatty acids; and a low linolenic acid (LLL) line, P9322, containing 3.1% linolenic acid. A lipoxygenase-free (LOX) line, IA2027, was provided by the Committee for Agricultural Development, Iowa State University (Ames, IA). Commodity soybeans (CS) were obtained from West Central Cooperative (Ralston, IA) and used as a control. The fatty acid compositions of the oils are shown in Table 1. All seed lots were conditioned to 6–7% moisture content before processing.

E-E processing. Five types of soybeans (20 bu each) were processed at a commercial E-E plant (Iowa Soy Specialties,

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TABLE 1
Fatty Acid Compositions of Modified Soybean Oils[a]

	16:0	18:0	18:1	18:2	18:3
CS	10.8	4.9	25.2	51.6	7.5
LOX	10.2	4.6	33.1	45.4	6.7
HO	6.7	3.8	79.2	7.1	3.1
LS	4.6	3.8	22.4	62.0	7.1
LLL	10.7	4.6	25.0	56.6	3.1

[a] Abbreviations: CS, commodity soybean oil; LOX, lipoxygenase-free soybean oil; HO, high oleic acid soybean oil; LS, low saturated fatty acid soybean oil; and LLL, low linolenic acid soybean oil.

Vinton, IA) by using an Insta-Pro extruder (Model 2500) and screw press (Model 1500) under conditions previously described (5). This equipment operated at a capacity of about 1 mt/h. The seeds were cracked and dehulled, and the meats were extruded and expelled. The shear-lock configuration of the extruder was 11-11-6-6. Double-flight screws were used, and the restriction die (nose cone) setting was 3/8 in. (0.94 cm). The temperature in the last segment of the extruder barrel was 132–143°C, and the total residence time was about 20–25 s. The meals and oils were collected in an identity-preserved fashion after the residuals from the previous seed lot had been flushed and operating parameters restabilized (usually about 10 min between seed lots). Duplicate oil samples (22 L each) were collected directly off the screw press at two different times during the 30-min processing period.

Natural oil refining. The crude oils were allowed to settle naturally in a cold room (5°C) for 2 d to remove meal fines and any spontaneously formed gums. On average, about 11% of the original weight was removed as sludge from the clear oil. The settled oils were stored in a freezer (–20°C) until refined. About 1,600 g of crude settled oil was water degummed with 3% deionized water at 60°C with vigorous stirring at the beginning and with gentle stirring when hydrated gums were visible and flocculent mass formed. During the 1-h period, the gums quickly and completely settled to the bottom of the beaker to produce clear oil. The degummed oil was divided into two portions: one portion was subjected to our adsorption refining process and the other to conventional alkali refining, bleaching, and deodorization.

For FFA removal, Magnesol (Dallas Group of America, Jeffersonville, IN), an adsorbent whose major component is magnesium silicate, was used at the 3% level (a level found to be effective in lowering FFA content to 0.03% in previous experiments). The treatment was carried out in a rotary evaporator rotating at 150 rpm, with a water bath temperature of about 95°C, and *ca.* 30-mm Hg vacuum, for a period of 20 min. The oil was then filtered to remove the Magnesol. Similarly, 3% silica (Sigma Chemical, St. Louis, MO), a level at which significant reduction in peroxide value (PV) and anisidine value (AV) was achieved, was used to further remove oxidation products.

"Soft" deodorization (lower temperatures than normal) was performed on each oil sample by using a glass laboratory continuous tray-type deodorizer, as described by Wang and Johnson (6), with 200°C column temperature and 10-min residence time. The oil flow rate was 10 mL/min, the steam flow rate was 0.25 mL water/min, and the system vacuum was 1–5 mm Hg.

Conventional oil refining. Water-degummed oil was neutralized according to the procedure described in the standard methods of American Oil Chemists' Society (AOCS) Ca 9C-52 (7). NaOH (8°Bé) was used at 0.2 g excess/100 g oil. The reaction was carried out at ambient temperature with vigorous stirring for 3 min. The emulsion was then immediately heated in a hot water bath to about 65°C under gentle stirring. Breaking of the emulsion was clearly visible, and flocculent mass rapidly settled to the bottom of the container. A laboratory centrifuge was used to remove the soap and minimize oil loss. Warm deionized water (15% of oil weight) was then used to wash the neutralized oil.

Bleaching was conducted at 95°C under vacuum in a rotary evaporator. Engelhart F-160 (Engelhart, Jackson, MS) bleaching earth (1% oil weight) was used. The rotary evaporator with oil and bleaching earth rotated at 150 rpm for 15 min, and the oil was cooled to about 60°C before breaking the vacuum. The bleaching earth was then removed by filtering.

The alkali-neutralized and bleached oil was deodorized using the laboratory deodorizer described above but using normal conditions: column temperature, 250°C, and residence time, 10 min.

Oil quality analysis. Duplicate oil samples for each treatment were analyzed according to standard methods of the AOCS (7): PV, AOCS Cd 8-53; AV, AOCS Cd 18-90; FFA, AOCS Ca Sa-40; phospholipid content, AOCS Ca 12-55; total tocopherol content, AOCS Ce 8-89; oxidative stability index (OSI), AOCS Cd 12b-92; and color, AOCS Cc 13b-45.

Statistical analysis. The General Linear Model of SAS program was used for analysis of variance (8). A factorial experimental design was used to examine the effects of oil type (five types) and processing step (five steps) on oil quality. The least significant differences (LSD) at $P = 0.05$ were calculated to compare treatment differences.

RESULTS AND DISCUSSION

In conventional oil refining, water and conditioners (e.g., phosphoric acid) are used to remove gums, NaOH is used to remove FFA, activated clays are used to remove pigments and to break down primary oxidation products, and deodorization is used to remove residual hexane, natural volatiles (flavors and odors), and lipid oxidation products formed during processing. The high capital cost and energy requirement of continuous centrifuges and deodorizers are limiting factors in setting up refineries to process relatively small quantities of E-E oil. In the present study, removal of FFA and residual phosphatides was achieved by adsorption and filtration. The effectiveness of physical adsorption of other impurities, especially oxidation products in E-E oil, was also examined in an attempt to eliminate energy-intensive and cost-prohibitive deodorization.

Degumming. Our previous work (5) showed that much of the phosphatides present in crude E-E oils spontaneously hydrated (without water addition) and settled more readily than did the phosphatides in solvent-extracted (SE) crude oil. E-E oils may have higher proportions of hydratable phospholipids than does SE oil. Simple settling of crude E-E oil produced clear oil with less than 100 ppm phosphorus compared with a mean of 267 ppm phosphorus in SE oil (5). In the present study, fresh crude E-E oil (unsettled but screened to remove meal fines) contained a mean of 828 ppm phosphorus, while settled oils contained a mean of 400 ppm phosphorus (because of the relatively short period of settling and low settling temperature). All oils water degummed very easily, with phosphorus being reduced from a mean of 400 ppm to below 55 ppm, except for HO oil, which was reduced to 100 ppm residual phosphorus. The higher level of the unhydratable phospholipid in HO oil may relate to its poorer initial seed quality, as indicated by the high FFA content (HO crude oil had 0.6% FFA vs. 0.2–0.3% FFA in the other oils).

FFA removal by absorption. In order to produce natural oil, no chemical exposure or reaction, such as neutralization with alkali, should be used. Surface adsorption (a physical treatment) may be a viable alternative to alkali treatment. In many instances, losses due to neutral oil being entrained with the adsorbent can be more than compensated by lower capital investment and operating costs for centrifuges in small refineries and by the increased value of marketing specialty and organic oils.

Many adsorbents (various neutral and acidic silica products, alumina, DE 52 ion exchanger, soyhull carbon, sodium metasilicate) were screened, and Magnesol and zeolite (Sigma, St. Louis, MO) were identified as the most effective materials to adsorb FFA. These materials were used at various concentrations (0.5, 1.5, 3.0%) to remove FFA in oils having various levels of phospholipid. To test how the phospholipid affects FFA adsorption, oils with various phosphorus contents (14–45 ppm) were obtained from settled crude E-E oil (133 ppm phosphorus) by treating with two different levels (0.5 and 1%) of Sorbsil R92 silica (Crosfield Co., Joliet, IL). Sorbsil R92 has been marketed to remove phospholipid and soap. Figure 1 shows that FFA, as well as phosphorus, was removed by Magnesol at the three levels tested. Magnesol more completely removed phosphorus than did zeolite (data not shown). FFA adsorption was not significantly affected by the presence of phospholipid in the range tested (14–133 ppm phosphorus).

Sodium silicate has been used as an adsorbent in natural refining of specialty oils (9). The synthetic nature, slight solubility in water (especially at high temperatures), and strong alkalinity of sodium silicate when dissolved made Magnesol the adsorbent of choice in our research. Magnesium silicate is the primary component of Magnesol. It occurs naturally and is practically insoluble in water.

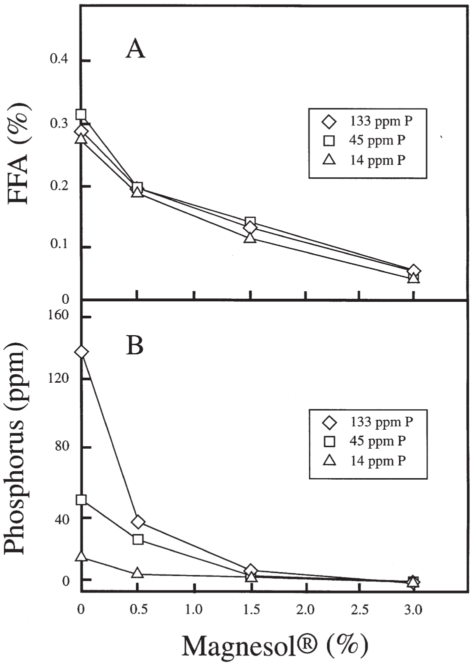

FIG. 1. Effects of Magnesol treatment on free fatty acids (FFA) and phosphorus removal of soybean oil: (A) free fatty acid content and (B) phosphorus (P) content. Initial phosphorus contents given in insets.

When oils contain high levels of phospholipid (>150 ppm), either water degumming or a phospholipid adsorption treatment may be needed prior to FFA adsorption. Large refineries normally use centrifuges. If a relatively small quantity of oil is processed and time is not a constraint, as is the case in most E-E plants, simple gum settling is feasible. Alternatively, adsorption and filtration treatments can be performed to reduce the phospholipid content of E-E oil. E-E oil that has been well settled usually contains very low levels of gums, and adsorption treatment may be more economically feasible than water degumming. Sorbsil R92 silica is an effective adsorbent for phospholipid and can be used when phosphorus levels exceed 150 ppm.

To simplify the refining process, it would be more effective and economical if the R92 silica and Magnesol could be added in sequence and only one filtration performed at the end of the two treatments. To test the interaction between R92 silica and Magnesol, the absorbents were applied in two different manners. In one experiment, R92 silica was added for phospholipid absorption and the oil was filtered, and then Magnesol was added to reduce FFA. In another experiment, Magnesol was added to the silica-treated oil without filtering the silica absorbent. Separate adsorption/filtration treatments proved to be more effective for removing phospholipid and FFA than a single filtration with sequentially combined adsorbents. Separate R92 silica (0.5%) and Magnesol (0.5%) treatments reduced the original levels (0.3% FFA and 133 ppm phosphorus) to 0.2% FFA and 21 ppm phosphorus, whereas the combined adsorbent treatment had no effect on FFA content and reduced the phosphorus content to only 70 ppm.

By increasing the Magnesol level to 3% without silica treatment, we were able to achieve low FFA content (0.05%) in E-E oil. If a stepwise countercurrent batch system were used, the adsorption process might be much more efficient and less Magnesol could be used. On the other hand, it may not be absolutely necessary to reduce the FFA to below 0.05%, which is the common maximum specification for today's commercially refined soybean oil, and higher FFA levels may be acceptable in some products.

Bleaching. Bleaching is almost always used in conventional refining to break down the hydroperoxides, which are primary oil oxidation products and catalysts for further oil oxidation, so that they can be easily removed during deodorization, as well as to remove pigments responsible for color. Hydroperoxide breakdown products are secondary oxidation products, which are measured by AV. They are primarily responsible for the oxidized off-flavor of the oil and are usually removed by deodorization. If deodorization is not used in natural refining, the generation of the secondary oxidation products must be minimized. Therefore, bleaching becomes an undesirable step, and we did not conduct a bleaching operation in our natural refining process. We speculate that a golden-colored oil may be quite marketable, and color may provide a means of product differentiation in the marketplace.

Adsorption of off-flavor compounds. The removal of peroxides (without breaking down) and flavor compounds is a challenge in the development of a low-capital-investment refining process. Our data indicate that Magnesol not only is a good adsorbent for FFA but also is a reasonably effective adsorbent for primary and secondary oxidation products as shown by reduced PV and AV (Fig. 2). Bleaching earth, on the other hand, catalyzed the breakdown of peroxides and formation of secondary oxidation products, as indicated by the reduced PV and increased AV. Bleached oil usually has a very undesirable taste due to these secondary oxidation products.

Although Magnesol and silica considerably reduced PV and AV, the oil still had a slight off-flavor that may be objectionable to some consumers. HO oil had much less off-flavor than did the other oils and might be satisfactorily refined by adsorption treatment without deodorization. Mild (soft) deodorization removed the off-flavor without significantly reducing AV, indicating the volatile and potent nature of the off-flavor compounds.

Comparison of refining characteristics of oil with various fatty acid compositions. Figures 3 and 4 show the characteristics of natural and conventionally refined E-E oils with modified fatty acid compositions. Tables 2 and 3 present the statistical significance of the oil-quality characteristics during natural and conventional refining. Oil type significantly affected the properties of the refined oils. The interactions between oil type and refining step were significant for all properties, except for total tocopherol during natural refining (Table 2). The significant interaction indicated that at least one oil behaved differently from the others during refining.

Because there was no interaction between oil type and refining step for total tocopherol content during natural refining, the main effects of the two factors were examined. Figure 5 shows the main effect of oil type on the amount of total tocopherol and percentage of α-tocopherol. Different types of oils had significantly different amounts of total tocopherol. Moreover, the percentages of α-tocopherol were very different among the five types of oil, with HO oil containing much less α-tocopherol (which has the most potent vitamin E activity). Figure 6 illustrates the main effect of processing step on

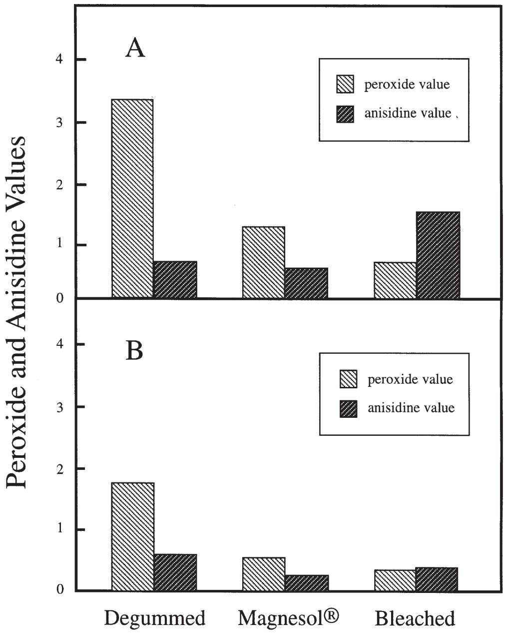

FIG. 2. Effects of adsorption treatment on oxidative status of two types of soybean oil: (A) commodity soybean oil and (B) high-oleic soybean oil.

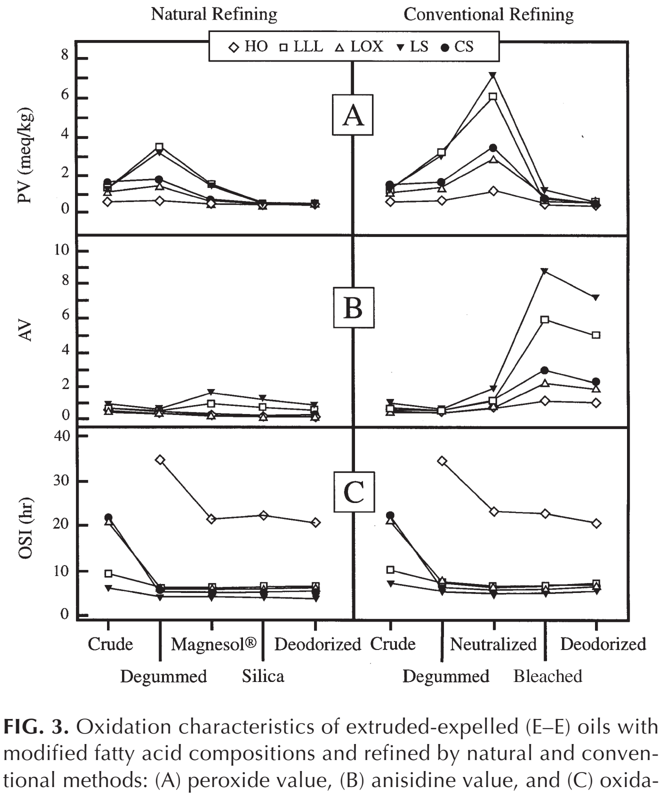

FIG. 3. Oxidation characteristics of extruded-expelled (E–E) oils with modified fatty acid compositions and refined by natural and conventional methods: (A) peroxide value, (B) anisidine value, and (C) oxidative stability index. HO = high-oleic, LS = low saturated fatty acid, LLL = low linolenic, LOX = lipoxygenase-free, CS = commodity soybeans, AV = anisidine value, PV = peroxide value, OSI = oxidative stability index.

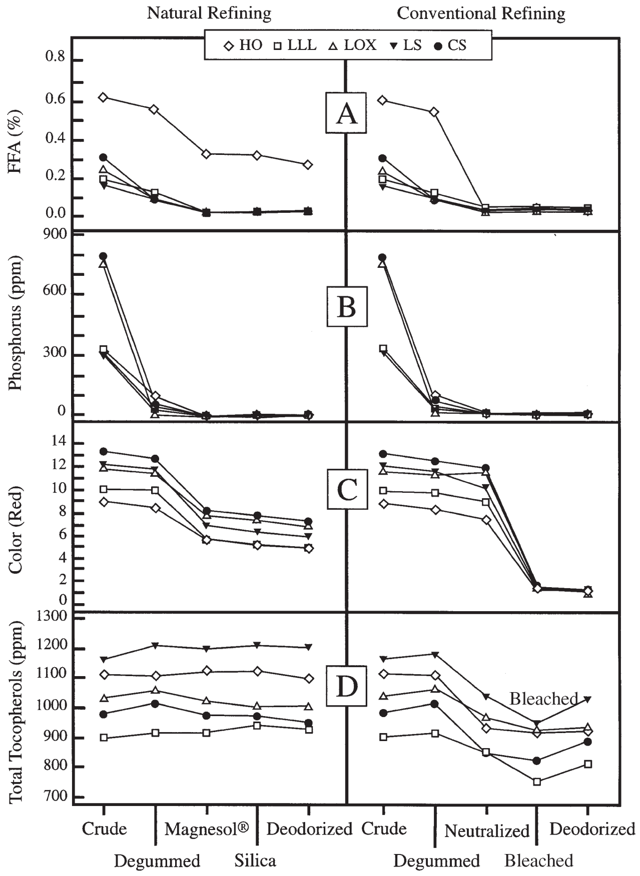

FIG. 4. Quality characteristics of E–E oils with modified fatty acid compositions and refined by natural and conventional methods: (A) free fatty acid value, (B) phosphorus content, (C) color (red), and (D) total tocopherol content. Abbreviations same as Figures 1 and 3.

total tocopherol content. Magnesol treatment did not significantly affect tocopherol contents, whereas silica treatment significantly reduced total tocopherol concentration. Soft deodorization at 200°C over a 10-min residence time did not significantly change total tocopherol content, nor were these conditions expected to cause isomerization.

LS and LLL oils were much more susceptible to oxidation during processing than were the other oils, and HO oil was the most stable oil during processing as indicated by PV and AV. LS and LLL oils had considerably higher percentages of linoleate than did the others (Table 1), which made them very susceptible to oxidation. LLL oil had less linolenate, but its very low total tocopherol content could have contributed to its instability. Although LS oil had the highest amount of total tocopherol, its unfavorable linoleate content likely contributed to low stability to oxidation. Magnesol effectively adsorbed secondary oxidation products from HO, LOX, and CS oils, but it did not reduce AV of the LS and LLL oils.

The FFA removal was similar for all oils except for HO oil, which we attribute to its much higher initial FFA content. The reason for the high FFA level in HO oil was not clear but it could be due to higher activities of hydrolytic enzymes in the seed or poor storage conditions before the sample arrived. The 3% Magnesol treatment was only effective for oils with less than about 0.3% FFA, and much higher quantities of adsorbent would be needed for oils higher in FFA. In conventional refining, the amount of alkali needed for neutralization was calculated based on FFA content, so that all FFA was removed.

The oxidative stabilities of the various refined oils were similar for all oils, except for HO oils, which showed significantly better stability. The crude HO oil did not show a typical oxidation curve over a long time period (>80 h), therefore, the OSI could not be calculated. Among the four other oils, LS oil was the least stable during both natural and conventional refining. LLL oil was comparable to the other oils, except at the crude stages where oxidative stability was as poor as that of LS oil.

The colors of the various crude oils were different. The CS oil had the darkest color, and the HO oil had the lightest color. The LLL oil had lighter color than LS and LOX oils. These color differences may be due to differences in their genetic background and pigment concentrations. Color reduction at various steps did not seem to be affected by type of oil, except for the LOX and HO oils during natural refining and for the LOX oil during conventional refining.

Comparison of natural and conventional refining processes. Conventional refining generated much more peroxide than did natural refining, especially during FFA removal. This reaction was carried out in a beaker and water bath under ambient conditions, which may have contributed to overestimating the extent of oxidation compared with oxidation during the continuous conventional process. When bleaching, the hydroperoxides decomposed to secondary products, as shown by significantly increased AV. Conventional deodorization at 250°C for a 10-min residence time did not totally re-

TABLE 2
P Values (>*F*) and LSD$_{0.05}$ Values of Quality Parameters of Naturally Refined E-E Oils of Various Types[a]

	PV	AV	FFA	P	Color	Toco	OSI	α-Toco
P values								
Oil type	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
Processing step	0.0001	0.0012	0.0001	0.0001	0.0001	0.0177	0.0001	0.0022
Type × step	0.0001	0.0001	0.0001	0.0001	0.0024	0.8138	0.0001	0.5787
LSD values								
Oil type	0.1234	0.1211	0.0186	23.432	0.3356	30.623	0.8632	2.2384
Processing step	0.1238	0.1211	0.0185	23.494	0.3356	30.623	0.8579	2.2384

[a] PV, peroxide value; AV, anisidine value; FFA, free fatty acids; α-toco, α-tocopherol; LSD, least significant difference.

TABLE 3
***P* Values (>*F*) and $LSD_{0.05}$ Values of Quality Parameters of Conventionally Refined E-E Oils of Various Types**[a]

	PV	AV	FFA	P	Color	Toco	OSI	α-Toco
P values								
Oil type	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
Processing step	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
Type × step	0.0002	0.0001	0.0001	0.0001	0.0001	0.0307	0.0001	0.0628
LSD values								
Oil type	0.6136	0.5477	0.0138	23.344	0.2341	27.338	0.4644	1.8436
Processing step	0.6136	0.5477	0.0138	23.406	0.2341	27.338	0.4615	1.8387

[a]See Table 2 for abbreviations.

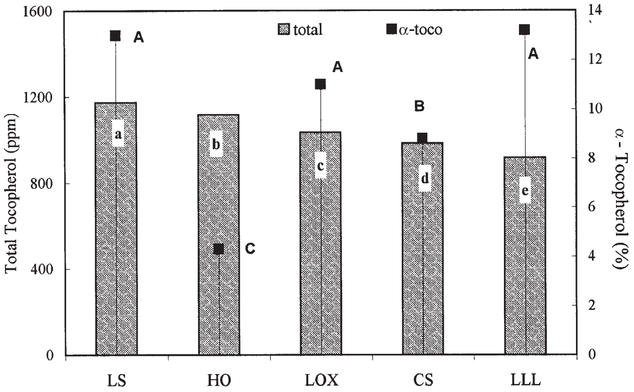

FIG. 5. Main effects of oil type on total tocopherol and α-tocopherol contents of naturally refined E–E oils. Bars or lines with different letters are significantly different at 5%. See Figure 3 for abbreviations.

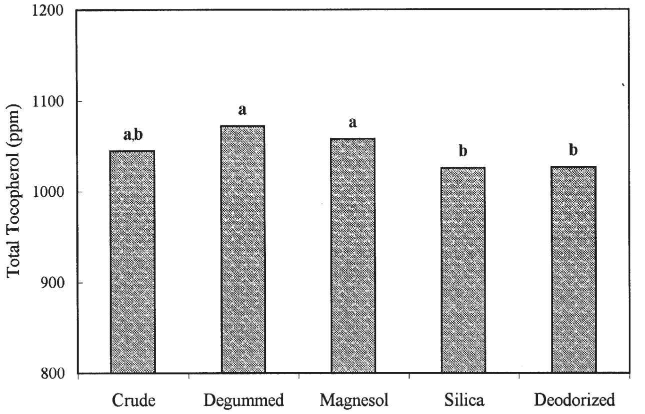

FIG. 6. Main effects of processing steps on total tocopherol of naturally refined E–E oils. Bars with different letters are significantly different at 5%. See Figure 3 for abbreviation.

move these compounds. On the other hand, there were few hydroperoxides to break down during natural refining, and the oils generally had lower AV after soft deodorization than did the conventionally refined oil (Fig. 3B). Natural refining is a much milder process, causing less oxidation of the oil.

FFA reduction, phospholipid removal, and oxidative stability were similar for the two refining methods, except for HO oil in FFA adsorption. The colors of the naturally refined oils were much more red than were the conventionally refined oils due to eliminating the bleaching step (Fig. 4C). The carotenoids contributing to the red color are micronutrients and maybe functionally important, such as acting as an antioxidant.

Total tocopherol content changed differently for the two types of refining (Fig. 4D). The steps in natural refining did not appreciably affect total tocopherol contents (although statistically significant), whereas the neutralization step in conventional refining appreciably reduced total tocopherol content. Other micronutrients were reportedly lost during this harsh treatment (10).

The newly developed natural refining technique shows promise in producing good-quality oils that might be marketed as natural or organic oils.

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