Refining Normal and Genetically Enhanced Soybean Oils Obtained by Various Extraction Methods

Tong Wang* and Lawrence A. Johnson

Department of Food Science and Human Nutrition and Center for Crops Utilization Research, Iowa State University, Ames, Iowa

ABSTRACT: Four different extraction methods, extrusion-expelling, conventional flaking-solvent extraction, expander-solvent extraction, and screw pressing, were used to separate oil and meal of a commodity soybean. The quality and refining characteristics of oils obtained by these methods were evaluated, and the effects of extraction method on oil quality were determined. The screw-pressed oil was more oxidized and hydrolyzed than the oils from the other extraction methods. The extruded-expelled oil had oxidative status similar to the solvent-extracted oils, although it contained the lowest amount of tocopherols. Five genetically enhanced soybeans were also processed by extrusion-expelling and solvent extraction methods, and differences in refining of these oils were examined. Overall, extruded-expelled oils were significantly different from the solvent-extracted oils in that they contained less tocopherols and were more oxidized than the solvent-extracted oils during refining. The differences between oils from the two extraction methods were magnified owing to the inclusion in the experiment of oils with modified compositions. The more unsaturated oils underwent significantly more oxidative degradation during refining than did the more saturated ones.

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KEY WORDS: Extrusion-expelling, genetically enhanced soybeans, identity-preserved soybeans, oil quality, refining, screw pressing, solvent extraction, soybean oil.

Extrusion-expelling (E-E) has been increasingly used to mechanically separate soybean oil and meal (1,2). The growing interest in E-E processing is due to its small scale [8–20 metric tons (MT)/d], low capital investment ($100,000–250,000), and suitability for handling various types but small quantities of soybeans, including identity-preserved organic, nongenetically modified, and genetically enhanced soybeans. Currently, most E-E processed oil is transported to large-scale refineries, where it is combined with solvent (hexane)-extracted (SE) oil and refined conventionally as is the case for the SE oil. It is often assumed that E-E oil has poorer or less consistent quality than SE oil, and it is oftentimes sold at lower-than-board prices. A recent comprehensive study comparing the qualities of crude E-E oils with SE oils showed that E-E oils had unique qualities compared to SE oils (3). For example, E-E oils were easily degummed by mere natural settling, and they had lower free fatty acid contents, which translated into lower refining loss. To further quantitatively illustrate how E-E oils are different from oils extracted by various other methods, a systematic and comparative refining study was designed to evaluate the refining characteristics of soybean oils obtained from four mechanical and solvent extraction methods. The methods include E-E, SE, continuous screw pressing (SP), and expander-solvent extraction (E-SE). SP involves extensive cooking to facilitate oil extraction by screw press, and E-SE is a variation of flaking and solvent extracting in which the flakes are expanded into porous collets; mass density is thereby increased, more cell walls are ruptured, and processing capacity and efficiency are increased (4).

Since E-E technology has the greatest potential for processing identity-preserved soybeans, a comparison of extracting and refining these specialty oils will also provide important information about how different seeds and oils may perform during processing. Therefore, the objectives of this research were to determine if E-E oil can be processed similarly to oils extracted by SE, SP, and E-SE, and to examine how the soybeans with modified compositions behave during E-E processing and conventional refining compared to those processed by SE.

EXPERIMENTAL PROCEDURES

Soybean processing. Commodity soybeans (CS) were obtained from West Central Cooperative (Ralston, IA) and were used for comparing refining characteristics of oil obtained by four extraction methods. Three other soybean lines with modified fatty acid compositions were obtained from Optimum Quality Grains (Des Moines, IA): a high-oleic acid (HO) line, A233HO, containing 79.2% oleic acid; a low saturated fatty acid (LS) line, P92B72, containing 8.4% total saturated fatty acids; and a low linolenic acid (LLL) line, P9322, containing 3.1% linolenic acid. A lipoxygenase-free (LOX) line, IA2027, provided by the Committee for Agricultural Development, Iowa State University (Ames, IA), was also used. An experimental soybean line high in cysteine was obtained from Richard Wilson, U.S. Department of Agriculture, Agricultural Research Service, North Carolina State University (Raleigh, NC). All seed lots were conditioned to 6 to 7% moisture content before processing. All oil extractions were performed in duplicates.

(i) E-E processing. Five types of soybeans (20 bu each), as described above, were processed at a commercial E-E plant (Iowa Soy Specialties, Vinton, IA) by using an Insta-Pro extruder (Model 2500) and screw press (Model 1500). This

*To whom correspondence should be addressed at Department of Food Science and Human Nutrition, 2312 Food Sciences Bldg., Iowa State University, Ames, IA 50011. E-mail: tongwang@iastate.edu

equipment operated at a capacity of about 1 MT/h. The seeds were cracked with a roller mill and dehulled by aspirating, 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 inches (0.94 cm). The temperature in the last segment of the extruder barrel was 130–143°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 were 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.

(ii) Conventional flaking-SE processing. The beans were cracked using a roller mill and aspirated to remove the hulls. The meats were flaked using a smooth roller mill to a flake thickness of 2.54–3.05 mm. A pilot-plant-scale solvent extractor simulator (French Oil Mill Machinery Co., Piqua, OH) was used to extract one bushel of dehulled and flaked soybeans. Five stages of hexane extraction were used (10 min solvent circulation and 5 min draining for each stage) at 60°C. The majority of hexane was then evaporated in an evaporator at 65°C (oil temperature), and any residual solvent was removed with a rotary evaporator.

(iii) E-SE processing. The beans were cracked, dehulled, and flaked as described above. An Anderson International expander (Cleveland, OH), 1/4 linear scale, with a 51-cm i.d. barrel was used to expand the flakes of the commodity soybeans. The barrel temperature was 110°C and the feed rate was 113 kg/h. SE was performed as described above.

(iv) Continuous SP. SP was conducted in the facilities of West Central Cooperative (Ralston, IA). The commodity soybeans were heated at elevated temperature in the range of about 113–177°C and maintained at this elevated temperature for about 60 min (5). The heated beans were then pressed in a Dupps 12-6 Oil Seed Pressor (the Dupps Company, Germantown, OH), which was specially engineered for West Central Cooperative.

Conventional oil refining. The crude oils were degummed with 3% water at 60°C with controlled agitation speeds (1.0 min at 250 rpm for maximum water dispersion and gradually reducing the speed to 60 rpm in 10 min, followed by a 60 rpm agitating-holding period of 50 min). The hydrated gum was separated from the oil by centrifuging. Water-degummed oil was then neutralized according to the procedure described in the standard methods of the American Oil Chemists' Society (AOCS) Ca 9C-52 (6). A 12°Bé (8% by weight) NaOH solution was used at 0.2 g/100 g oil excess to ensure complete neutralization. The reaction was carried out at ambient temperature with vigorous stirring for 3 min. Then the emulsion was immediately heated in a hot water bath to about 65°C under gentle stirring. Breaking of the emulsion was clearly visible, and flocculant rapidly settled to the bottom of the container. Soap was removed by centrifuging. Warm deionized water (15% of oil weight) was then used to wash the neutralized oil. Bleaching was conducted at 95°C under vacuum (23 mm Hg) in a rotary evaporator. Engelhart F-160 (Engelhart, Jackson, MS) bleaching earth was used at 1% oil weight. The rotary evaporator flask 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 bleached oil was deodorized using a continuous tray-type laboratory deodorizer (7) at a column temperature of 250°C and residence time of 10 min.

Oil quality analysis. Duplicate oil samples from each treatment were analyzed according to standard methods of the AOCS (6): peroxide value (PV), AOCS Cd 8-53; anisidine value (AV), AOCS Cd 18-90; free fatty acid (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.

To estimate soybean proximate composition (moisture, protein, oil, and fiber contents), seeds were analyzed by near-infrared (NIR) spectroscopy by using a calibrated Foss-Infratec 1229 Whole Grain Analyzer (Foss North America, Inc., Eden Prairie, MN) (8). A gas chromatographic method (9) was used to determine fatty acid composition of the oils. In estimating the amount of meal fines in the E-E oil, the freshly pressed and filtered (through a shaker screen) oil was centrifuged at $1000 \times g$ for 5 min. The settled fines were collected, and the percentage of fines was calculated. The residual oil content and protein denaturation of the defatted E-E meals were determined by using standard AOCS methods (6): oil content, AOCS Ba 3-38, and protein dispersibiity index (PDI), AOCS Ba 10-65.

Statistical analysis. The general linear model of SAS program (10) was used for the analysis of variance. To examine the effect of extraction type on oil quality, a two-factor factorial design was used, with extraction type (four levels) as one factor and refining step (five levels) as the other. A three-factor factorial design was used to examine the effect of extraction type (two levels), oil type (five levels), and processing step (five levels) on oil quality characteristics. The least significant differences ($LSD_{0.05}$) were calculated to compare treatment means.

RESULTS AND DISCUSSION

Effect of extraction methods on oil quality. Four extraction methods—E-E, SE, E-SE, and SP—were used to separate oil and meal from one lot of commodity soybean. The refining characteristics of the oils obtained are presented in Figure 1. SP oil from the same lot of soybeans that was used for the other extractions was requested from West Central Cooperative, but degummed oil, rather than crude oil was mistakenly provided. The data for this sample at the crude stage were treated as missing values in the data analysis. Statistical analysis results of the main effects of extraction methods and refining steps and their interactions are shown in Table 1. For the quality parameters that did not have significant interaction at the 5% level, the main effect of extraction is presented in Table 2.

Extraction method significantly affected oil quality, as did the refining step (Table 2). SP produced oil with significantly higher

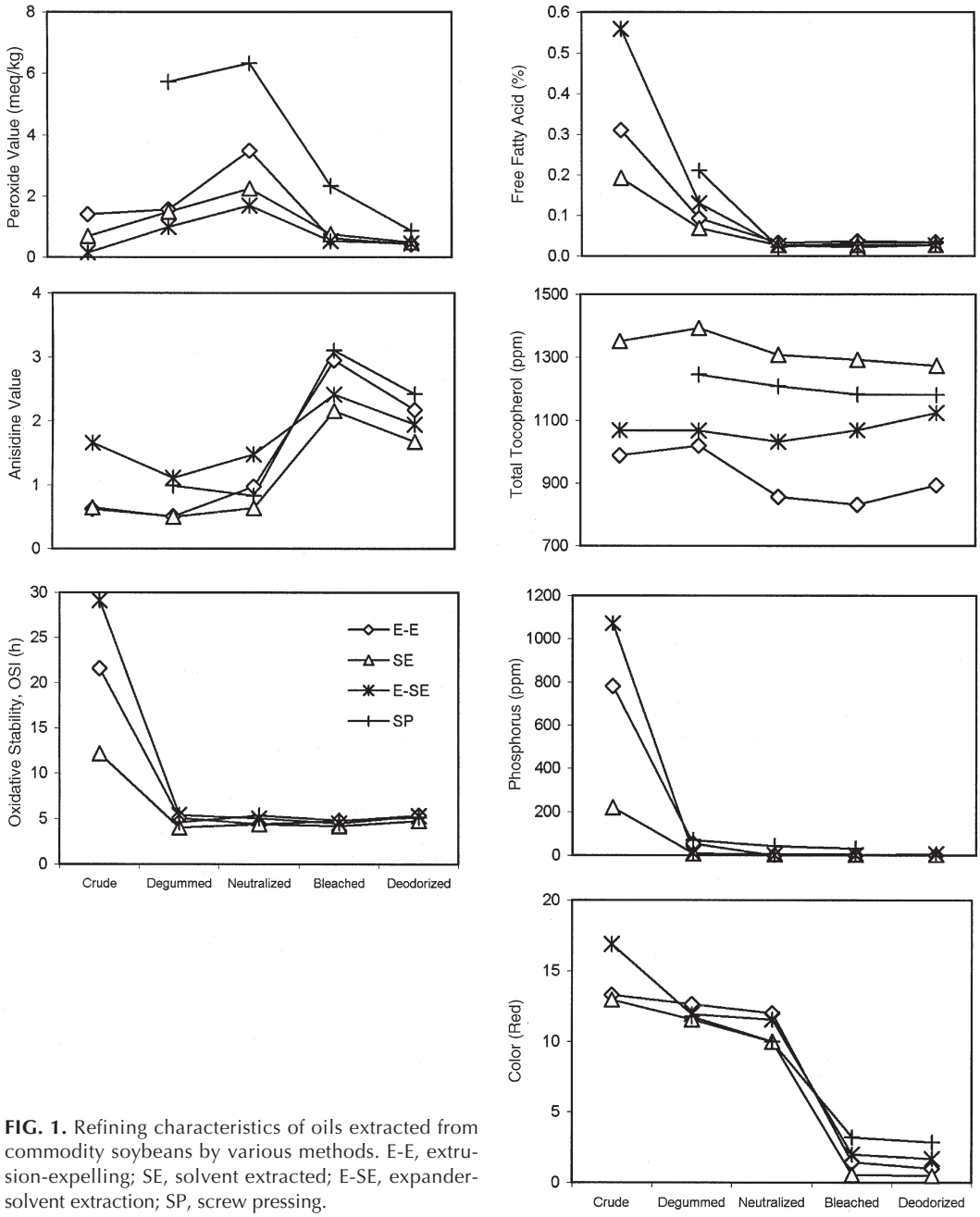

FIG. 1. Refining characteristics of oils extracted from commodity soybeans by various methods. E-E, extrusion-expelling; SE, solvent extracted; E-SE, expander-solvent extraction; SP, screw pressing.

PV (3.82 meq/kg) than the others (0.77–1.50 meq/kg). SP also resulted in oil that contained the highest amount of secondary oxidation products (AV = 2.21), but SE oil had the lowest amount of these compounds (AV = 1.12). The high temperature and long cooking period to which the seeds were exposed prior to oil pressing in SP may have contributed to this oxidative degradation. E-E oil was not significantly more oxidized than the SE oils. SE was much more effective in extracting tocopher-

TABLE 1
***P* Values and Least Significant Differences ($LSD_{0.05}$) of Quality Parameters of Oils Extracted by Various Methods and Conventionally Refined[a]**

	PV	AV	FFA	Color	Total tocopherols	Phosphorus	OSI
Extraction	0.0001	0.0023	0.0001	0.0001	0.0001	0.0001	0.0001
Refining step	0.0002	0.0001	0.0001	0.0001	0.0004	0.0001	0.0001
Extraction × refining step	0.1982	0.1350	0.0001	0.0001	0.1309	0.0001	0.0001
$LSD_{0.05}$ for extraction	1.002	0.460	0.019	0.3	44.3	9.7	0.5

[a]PV, peroxide value; AV, anisidine value; FFA, free fatty acid; OSI, oxidative stability index.

TABLE 2
Main Effect of Extraction Method on Oil Quality[a]

	E-E	SE	E-SE	SP
PV (meq/kg)	1.50 b	1.14 b	0.77 b	3.82 a
AV	1.45 b,c	1.12 c	1.72 a,b	2.21 a
Tocopherol (ppm)	918 d	1324 a	1072 c	1205 b

[a]Values in the same row with different letters are significantly different at 5% level. E-E, extrusion-expelling; SE, solvent extracted; E-SE, expander-solvent extraction; SP, screw pressing; for other abbreviations see Table 1.

ols (1,324 ppm) from the seed than the other methods. E-E extracted the least amount of tocopherols (918 ppm).

The quality changes during conventional refining of oils extracted by different methods are illustrated in Figure 1. Mechanically pressed oils tended to form more peroxides during refining than did the SE oils. This may be due to their higher initial peroxide contents. The peroxides function as catalysts for oil oxidation. Although SP oil had higher tocopherol content than did E-SE and E-E oils, more peroxides were found in SP oil during refining than in the other oils. E-E oil had slightly higher PV or AV values than did SE oils at some refining steps.

FFA content is a measure of hydrolytic degradation of oil during processing. Degummed SP oil had much higher FFA content than did the other oils, which may be due to the high temperature and long heating pretreatment of the seed, causing hydrolysis of the oil. E-SE oil had higher FFA than did SE and E-E oils, and the reason may be the longer processing time in a crushed state under mild conditions and possible exposure of oil to active lipases. The degumming step significantly decreased the apparent FFA content due to the removal of the water-soluble compounds, which may have contributed to the acidity of the oil.

As expected, SE resulted in a more complete tocopherol extraction than did the other methods. It was surprising to observe that SP oil had much higher tocopherol content than E-SE oil. It was shown that oven heating increased tocopherol recovery from corn fiber (11). Although the expander treatment is also a heating process, it may have caused stronger interactions between tocopherol and other seed components; therefore, the subsequent SE could not extract as much tocopherols as from the unexpanded seeds.

Phospholipid contents of the crude oils were not as expected, particularly for the SE oil that had very low phosphorus concentration. If moisture was not completely removed from the oil, the phospholipid could hydrate and settle and result in partially degummed oil. The oxidative stability of the oils correlated well with the phospholipid content at the crude stage. Phospholipids have been shown to be effective antioxidants (12).

All oils had similar colors, except for E-SE crude oil, which had darker color, and fully refined SP oil, which had a darker color than the others due to the severe heat treatment to which the seeds were exposed.

General observations for processing soybeans with modified fatty acid compositions. Soybeans with modified compositions were processed with conventional SE and E-E methods. The fatty acid compositions of the five oils extracted and refined in this study are shown in Table 3. The seed compositions and other parameters related to E-E processing are presented in Table 4. Another two soybeans, low stachyose (LST) from Optimum Quality Grains and high cysteine (HC) seeds from North Carolina State University, were also processed, but their oils were not refined in this study.

All seeds had similar protein, oil, and fiber contents, except for LLL, which had higher oil content (20.2%) than the others (ranging from 17.2 to 18.5%). The processing parameters or the settings on the extruder and screw press were the same for all seeds, but the extruder temperature ranged from 130 to 143°C. Different types of seeds processed differently by E-E, as reflected by the state of the extrudate when exiting the orifice, the cake formation from the screw press, the amount of meal fines in the oil, and the degree of protein denaturation of the meal. HO, LLL, and LST seeds processed easily and as expected; that is, semisolid extrudates were formed, large and smooth press cakes were produced, and small amounts of meal fines (6–9% of the oil by weight) were found in the oils. When processing the other types of seeds, either the extrudate was foamy or the press cake did not form correctly, causing significant amounts of fines in the oils (11–16%). The difference in processing was also reflected by the meal quality data—the degree of protein denaturation and the residual oil content in the meal. For example, HO protein was denatured to a much less degree (PDI = 44.2) than was the LLL protein (PDI = 14.8). It is unclear what caused this difference. Other composition factors or cellular structure features of the seeds may have contributed to the differences. It is also worth noting that HC press cake had much darker surface color than that of other types of soybeans, and different aromas were released when different types of seeds were processed.

During solvent extraction of these modified seeds, little difference was observed among different seeds, with the exception of the HC protein meal, which crumbled more than the others during desolventizing-toasting.

Comparison of E-E and SE oils during conventional refining. The extraction method had a significant effect on refining quality of the oils, as shown in Table 5. Comparisons of how the E-E and SE oils differed are illustrated in Figures 2 and 3.

There were significantly higher amounts of peroxides and secondary oxidation products produced in E-E oils during refining compared to the SE oils. This may be due to the slightly higher amount of peroxides present in the crude E-E oils, which may have catalyzed further oxidation of these oils. E-E oils also contained lower amounts of tocopherol than the SE

TABLE 3
Fatty Acid Compositions of Modified Soybeans[a]

	16:0	18:0	18:1	18:2	18:3
CS	10.82	4.89	25.21	51.61	7.47
LOX	10.15	4.60	33.14	45.42	6.68
HO	6.72	3.80	79.22	7.15	3.12
LS	4.61	3.82	22.43	62.02	7.12
LLL	10.74	4.55	25.03	56.60	3.07

[a]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.

TABLE 4
Comparison of Seed Composition, E-E Processing, and Product Quality[a]

	Seed composition (%)[b]			Temperature	Fines in oil	Meal	
	Protein	Oil	Fiber	(°C)	(%)	PDI	Oil (%)[d]
CS[a]	34.7	18.1	5.3	142	14.5	24.6	7.5
LOX	39.8	17.2	5.0	142	15.9	28.3	6.3
HO	37.8	18.5	5.5	133	6.4	44.2	7.8
LS	36.7	17.8	4.7	139	11.3	19.3	6.0
LLL	35.5	20.2	5.0	134	8.8	14.8	6.7
LST[c]	38.0	16.8	5.2	130	8.7	24.3	6.3
HC[c]	38.4	17.1	5.1	143	13.7	26.3	6.2

[a]LST, low stachyose; HC, high cysteine; PDI, protein dispersibility index. See Tables 2 and 3 for other abbreviations.
[b]Composition measured by near-infrared and calculated based on 13% moisture content.
[c]Oils obtained from these seeds were not used in refining evaluation.
[d]Dry weight basis.

oils, making them more susceptible to oxidation. The oxidative stabilities, as measured by OSI, were similar for the two types of oils. FFA contents of the E-E and SE oils were similar, with the exception of HO oil, which was hydrolyzed to a larger degree during SE processing than during E-E processing. SE has relatively longer process time and milder conditions, and oil may be hydrolyzed more by active lipase. The significantly higher amount of FFA in HO oils, compared to the levels in the other oils, may be caused by the inherent seed composition, lipase activities, or growing and storage conditions. Phospholipid contents of E-E and SE oils were lower than expected, possibly due to natural hydrating and settling during storage. Both oils water-degummed effectively. The oil color difference among types of seeds was greater in E-E oils than in SE oils. SE is a gentler process than E-E, and the natural pigments and other components should be less damaged. However, our data show that several E-E oils had lighter colors than their SE counterparts.

In the study of quality of oils extracted by four methods, as described above, E-E oil was not as different from the SE oils as in this study of oils of different compositions and extracted by different methods. The highly unstable oils with higher unsaturated fatty acid content magnified the quality difference of E-E and SE oils.

Refining characteristics of oils with various fatty acid compositions. The type of oil also had a significant effect on quality of refined oil (Table 5). The interactions among various factors were significant in most cases, except for tocopherol amount and color. The comparison of how the oils differed is illustrated in Figures 2 and 3.

LS and LLL oils were much more susceptible to oxidation than the other oils, and HO oil was the most stable oil during processing, as indicated by its PV and AV values. This is particularly evident for E-E oils. The fatty acid compositions and tocopherol contents of the oils may have been the cause. LS and LLL oils had considerably higher percentages of linoleate than others, and this fatty acyl group is very susceptible to oxidation. LLL oil actually had a lower percentage of linolenate which is the most easily oxidized fatty acyl group, but its low total tocopherol content could have contributed to its instability. Although LS oil had the highest amount of total tocopherol, its unfavorable linoleate content may have played a major role in oil oxidation. The oxidative stabilities were similar for all oils under the standard testing conditions, except for HO oil, which showed significantly higher stability than the other oils. The crude E-E HO oil did not show a typical oxidation curve over a very long period of time (>80 h), therefore the OSI could not be calculated.

The FFA contents were similar for all oils except HO, as discussed earlier. Tocopherol contents were different in different types of oils, and the orders of concentration were the same for both E-E and SE oils.

TABLE 5
***P* Values and Least Significant Difference Values ($LSD_{0.05}$) of Quality Parameters of Oils of Various Extraction Methods, Compositions, and Refining Steps[a]**

	PV	AV	FFA	Color	Total tocopherols	Phosphorus	OSI
Extraction	0.0001	0.0001	0.0155	0.0015	0.0001	0.0001	0.0411
Composition	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
Step	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
Extraction × composition	0.0008	0.0001	0.0001	0.0001	0.0001	0.0001	0.0021
Composition × step	0.0001	0.0001	0.0001	0.0001	0.3210	0.0001	0.0001
Extraction × step	0.0001	0.0001	0.0001	0.0001	0.0354	0.0001	0.0700
Extraction × composition × step	0.0018	0.0001	0.0001	0.0739	0.7165	0.0001	0.0028
$LSD_{0.05}$ for extraction	0.1928	0.1818	0.0085	0.1415	20.9	13.5	0.8686
$LSD_{0.05}$ for composition or step	0.3049	0.2874	0.0135	0.2237	33.0	21.4	1.3785

[a]See Table 1 for abbreviations.

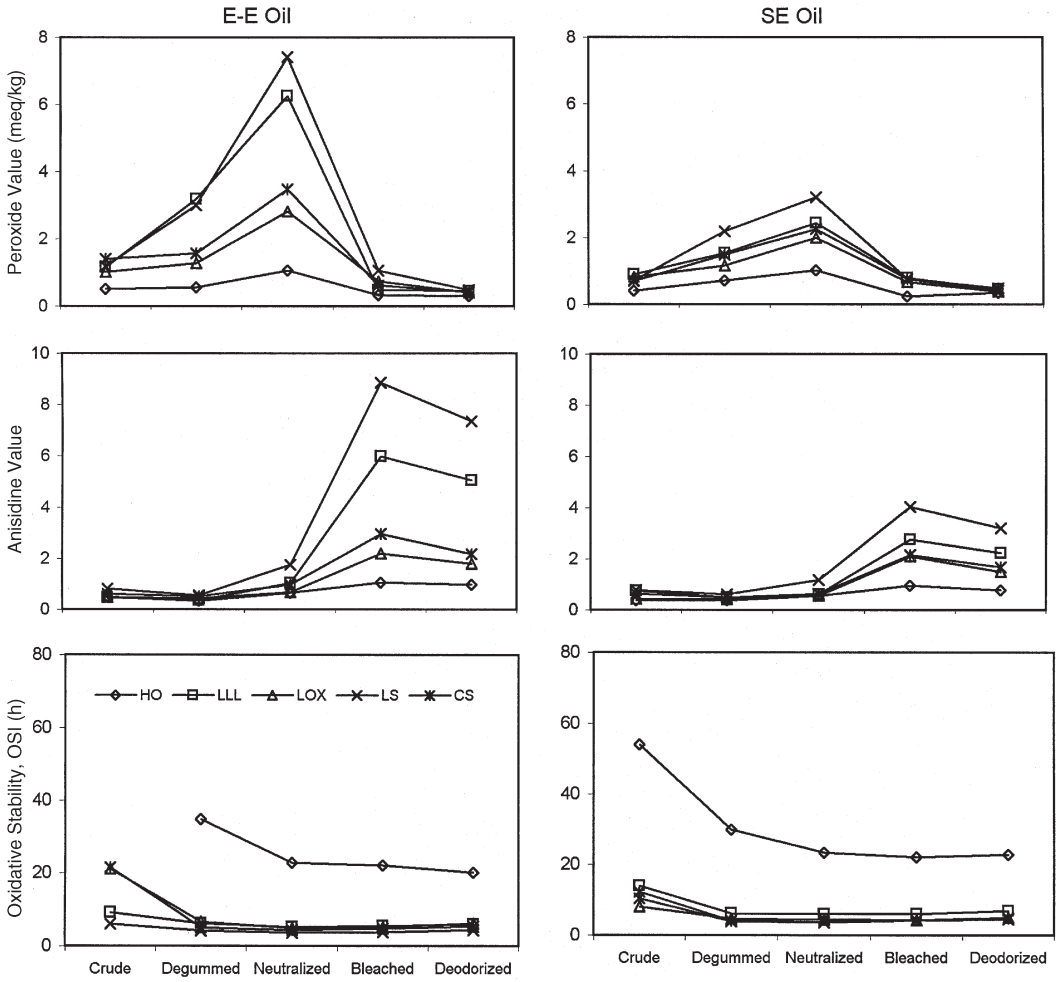

FIG. 2. Oxidative stabilities during refining of oils with various fatty acid compositions and extracted by E-E and SE methods. HO, high oleic acid soybean oil; LLL, low linolenic acid soybean oil; LOX, lipoxygenase-free soybean oil; LS, low saturated fatty acid soybean oil; CS, commodity soybean oil. For other abbreviations see Figure 1.

Phospholipid contents of the crude oils were similar, except for E-E CS and LOX oils, possibly due to difference in moisture content and gum settling.

The colors of the various oils were different. The CS oil had the darkest color, while HO oil had the lightest color. LLL oil had lighter color than LS and LOX oils. These color differences may be due to the differences in their genetic background and pigment concentration. The color reduction during bleaching did not seem to be affected by type of oil.

Overall, soybean oil quality and refining characteristics were affected by oil extraction method. SP oil was more oxidatively damaged during processing than oils from other extraction methods. Fatty acid composition significantly affected oil quality. When processing commodity type soybeans, E-E oil had similar quality as SE oil, and it was satisfactorily refined by conventional methods. But the oils with more unsaturated fatty acid content and extracted by E-E had more oxidative degradation than the same oils extracted by SE.

E-E oils could alternatively be refined by a nonchemical, minimal-refining method, in which the oxidative degradation can be significantly reduced (13) due to its gentler process procedure. Such mechanically extracted and minimally processed oils may find a niche market in the organic or natural product sector of the food industry.

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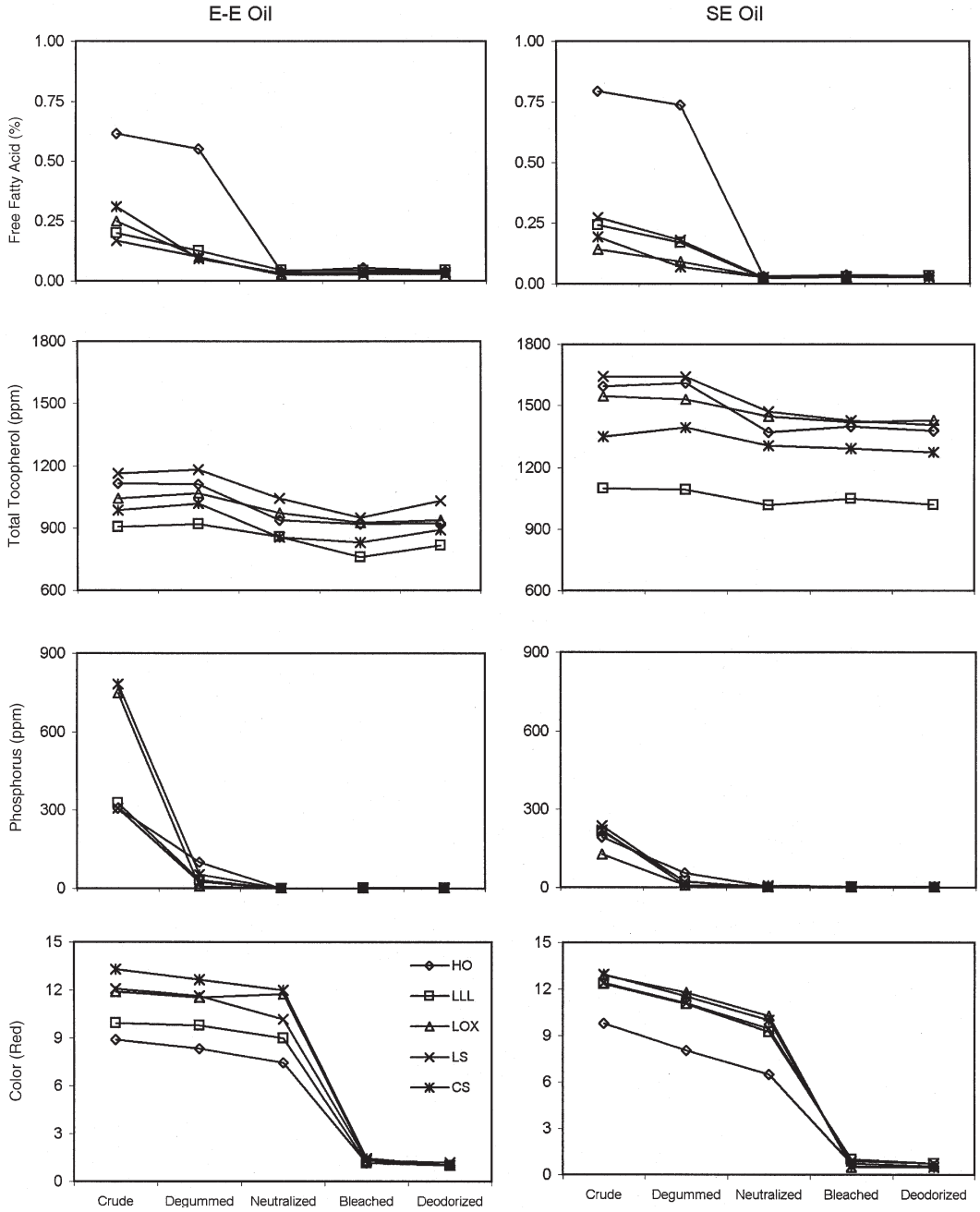

FIG. 3. Changes in quality characteristics during refining of oils of various compositions and extracted by E-E and SE methods. See Figures 1 and 2 for abbreviations.

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