Refining High-Free Fatty Acid Wheat Germ Oil

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ABSTRACT: Wheat germ oil was refined using conventional degumming, neutralization, bleaching, and continuous tray deodorization, and the effects of processing conditions on oil quality were determined. The crude wheat germ oil contained 1,428 ppm phosphorus, 15.7% free fatty acid (FFA), and 2,682 ppm total tocopherol, and had a peroxide value (PV) of 20 meq/kg. Degumming did not appreciably reduce the phosphorus content, whereas neutralization was effective in removing phospholipid. Total tocopherol content did not significantly change during degumming, neutralization, and bleaching. A factorial experimental design of three deodorization temperatures and three residence times (oil flow rates) was used to determine quality changes during deodorization. High temperatures and long residence times in deodorization produced oils with less FFA, PV, and red color. Deodorization at temperatures up to 250°C for up to 9 min did not significantly reduce tocopherol content, but, at 290°C for 30-min residence time, the tocopherol content was significantly reduced. Good-quality wheat germ oil was produced after modifying standard oil refining procedures.

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KEY WORDS: Bleaching, degumming, deodorization, neutralizing, refining, tocopherols, wheat germ oil.

Wheat germ oil is an excellent source of polyunsaturated fatty acids and vitamin E. It is one of the richest natural sources of α-tocopherol, the type of tocopherol with the greatest vitamin E activity. Although synthetic α-tocopherol is available, natural sources of the vitamin are often preferred. Wheat germ oil has been attributed to reduced plasma and liver cholesterol in animals, improved physical endurance, and delayed aging (1). The oil also has been reported to improve human physical fitness, an effect attributed to the long-chain *n*-alkanols (particularly octacosanol) (2). Wheat germ oil has been used as a fertility agent, an antioxidant, and an additive in natural food and health and cosmetic products.

The wheat germ constitutes only about 2% of the whole wheat grain and contains about 8–14% oil (3). Wheat germ oil is obtained by either mechanical pressing or solvent extracting the germ, which is separated during milling the wheat to flour. Although solvent extraction usually recovers more of the wheat germ oil (90%) than pressing (50%) (4), pressing is usually preferred by consumers because wheat germ oil obtained this way is perceived as "natural."

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Although wheat germ oil is often used in its crude form, refining improves the quality and stability of the oil. The free fatty acid (FFA) content of the crude oil is usually very high and quite variable (5–25% is typical), depending upon the conditions of germ separation, germ storage, and oil extraction. FFA often contributes to bitter and soapy flavor in food. Wheat germ oil is usually dark-colored and may have strong odor and flavor depending on the oxidative condition of the oil. Therefore, it is desirable to remove FFA and components resulting from oxidation while retaining as much of the tocopherols as possible.

Degumming, neutralization, bleaching, and deodorization are typical refining steps of vegetable oil processing. The highly desirable tocopherols are reduced during soybean oil refining, and this removal reduces oxidative stability and nutritional value of the refined oil when compared with the crude oil (5). Tocopherols are more volatile than the neutral triacylglycerides and can be removed with the high temperature and high vacuum of deodorization. Short exposure of the oil to heating is desirable to reduce this loss. Using a laboratory continuous deodorizer, similar in design to the system reported by Smouse (6), we are able to accurately adjust and control the operating parameters and thus determine oil quality changes, especially changes in tocopherol concentrations. The objectives of this study were to examine changes in qualities of wheat germ oil during typical oil processing and to study the effects of deodorization conditions on tocopherol retention.

EXPERIMENTAL PROCEDURES

Oil quality analyses. Crude wheat germ oil was provided by a Midwest agricultural processing company. The oil was obtained by solvent extraction. Official Methods of American Oil Chemists' Society (AOCS) (7) were used to determine the FFA contents (AOCS Ca 5a-40), peroxide values (PV) (AOCS Cd 8-53), oxidative stabilities by the active oxygen method (AOM) (AOCS Cd 12-57), phosphorus contents (AOCS Ca 12-55), colors (AOCS Cc 13b-45), tocopherol contents (AOCS Ce 8-89), and *trans* fatty acid contents (AOCS Cd 14c-94). The fatty acid profile was determined by the method of Hammond (8). For oil samples with high FFA content, an acid catalyst (sulfuric acid, 3% in methanol) was used, and the reaction was conducted in a sealed vial at 80°C for 100 min. All analyses were performed in duplicate.

Oil refining. All oil refining trials were conducted in duplicate, and the oil qualities after each processing step were de-

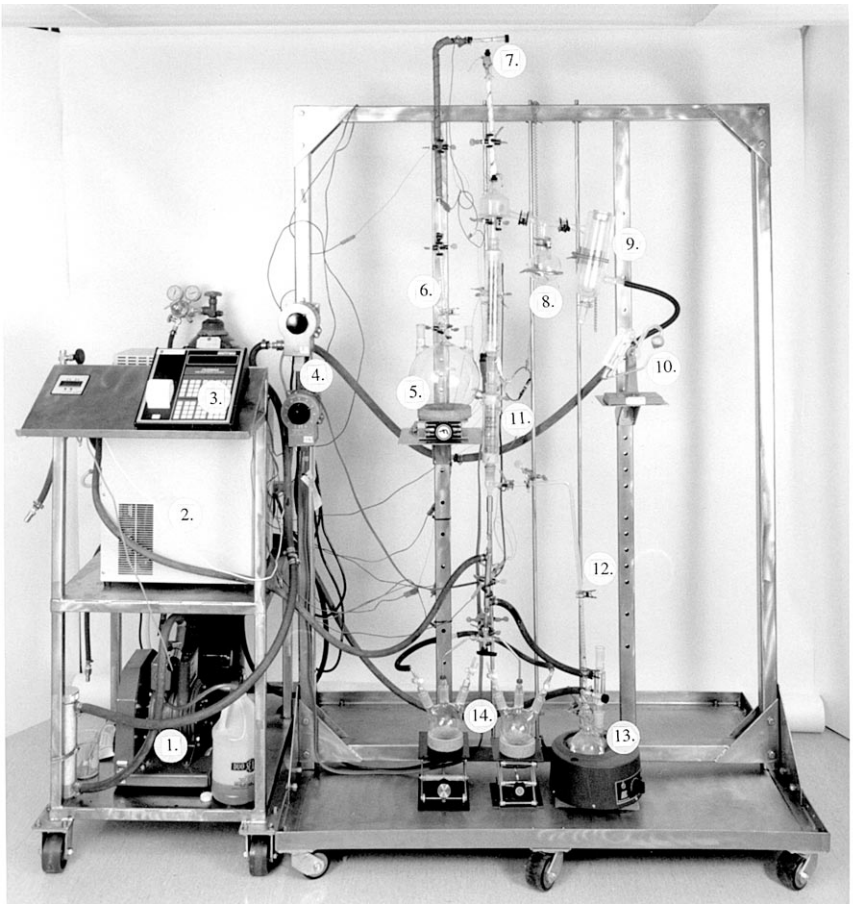

FIG. 1. Laboratory continuous deodorizer. Numbered parts: (1) vacuum pump; (2) cooling system; (3) temperature monitoring system; (4) temperature controller; (5) feed flask; (6) oil flow meter; (7) oil flow regulator; (8) liquid trap; (9) dry ice trap; (10) vacuum meter; (11) older shaw columns; (12) steam flow meter; (13) steam generator; (14) receiving flasks.

termined. The refining system consisted of 4-L glass vessels for degumming and neutralization, which were equipped with water-jacketed heating and variable-speed stirring. The bleaching vessel was equipped with vacuum and nitrogen purge systems. The deodorizer (Fig. 1) was a continuous, sieve plate Oldershaw column, countercurrent, steam deodorization apparatus and incorporated design features of Smouse (6).

(i) Degumming. Water degumming with acid pretreatment was performed with 3 L of oil. Phosphoric acid (85% concentration) was added at the 0.15% level to the heated oil (60°C), and the mixture was vigorously stirred (250 rpm) for 10 min. Distilled water (4%) was then added, and the oil was mixed at a high shear rate (250 rpm) for 5 min. The mixing speed was gradually reduced over a 1-h period, according to the rate of gum formation and aggregation. The mixture was centrifuged (8,627 × *g* for 15 min) to remove the hydrated phospholipids and other water-soluble impurities.

(ii) Neutralizing. Sodium hydroxide solution, 26°B concentration (19.7%), was used to neutralize the FFA. The amount of alkali needed was calculated based on the stoichiometric quantity plus 0.12% excess (9). The alkali was added to 3 L of degummed oil at ambient temperature and mixed vigorously (180 rpm) for 10 min. The oil was then gently stirred and heated until the temperature reached 65°C. The mixture was slowly stirred for an additional 10 min to break any emulsion that might have formed during neutralization. The mixture was then centrifuged (8,627 × *g* for 15 min) to remove the soap.

(iii) Bleaching. The neutralized oil (3 L) was treated with 1.5% acidic silica (Sorbsil R92; Crosfield, Joliet, IL) to absorb residual soap and phospholipids. Acid-activated bleaching earth was then added at the 2.5% level, and the bleaching was conducted under vacuum at 100–110°C for 10 min. The silica and bleaching earth were removed by filtering the oil under vacuum after cooling to 60–70°C.

(iv) Deodorizing. A factorial design was used to examine the effects of two factors—oil temperature and oil residence time (which was controlled by oil flow rate)—on the quality of deodorized oil. Temperatures of 290, 250, and 200°C and residence times of 4, 9, and 30 min, corresponding to flow rates of 25, 10, and 3 mL/min, were used. The steam flow rate was held constant at 0.25 mL water/min. The system vacuum was maintained at 5–2 mm Hg (6.7–2.7 millibar). Samples of about 300 mL of oil were used for each treatment.

RESULTS AND DISCUSSION

Effect of refining conditions on oil quality. The crude oil had very high phospholipid (1428 ppm P) and FFA (15.7%) contents compared with other crude vegetable oils in commerce. With such a high FFA content, physical refining (steam deacidification) would be appropriate provided the phosphorus content could be reduced to the very low level required (10,11). Corn germ and rice bran oils with very high FFA contents have been successfully refined by physical refining methods (12,13). In order to produce an oil with low phosphorus content suitable for physical refining, different degumming methods were tested: (i) water degumming, degumming with acid pretreatment; (ii) modified degumming in which about 400 ppm soap was created to absorb the phospholipid; and (iii) 7% acidic silica absorption treatment. None of the treatments were very effective. For example, the phosphorus contents were 1066, 1033, and 810 ppm for the acid pretreated degumming, modified degumming, and with absorption treatment, respectively.

The difficulty in degumming wheat germ oil was the very slow and incomplete hydration of the phospholipid. It is probable that a large amount of unhydratable phospholipid was present in the oil, caused by the action of phospholipase D during wheat milling. Wheat is tempered with water to raise the moisture content of the grain to 20% prior to milling; grinding wheat at this moisture content is very conducive to enzyme activity. The once-degummed oil was clear but still had a very high phosphorus content. Therefore, the phospholipid in this oil was likely the unhydratable type. Eventually, the oil was twice-degummed with acid pretreatment, but the degummed oil still contained 1,082 ppm phosphorus. Oil with high phosphorus content cannot be satisfactorily physically refined because it is prone to darkening in color. Therefore, alkaline neutralization had to be used to remove FFA instead of steam distillation.

The addition of the calculated amount of the alkali did not satisfactorily reduce the FFA content, and FFA contents of 1.6–2.8% were obtained. The once-neutralized oil was neutralized again using the stoichiometric amount plus 0.12% excess, and the resulting oil had an acceptable FFA content (0.09%). The high phospholipid content reduced the efficiency of the first neutralization treatment. A higher percentage of excess, such as 0.5–2% which has been used to neutralize rice bran oil with high FFA (14), might be used to counteract the high phospholipid content. Neutralizing twice significantly reduced the phosphorus content (99 ppm). The refining loss was about 37%, which was about 2.4 times the FFA content. The refining loss in rice bran oil can be as high as 10 times of the FFA when using the standard cup method (14).

In order to remove the soap and residual phospholipid to maximize the action of the bleaching earth, synthetic silica (Sorbsil R92), which adsorbs these oil components, was used. A relatively large amount of bleaching earth (2.5%) was required to purify the very dark-colored oil. For refining wheat germ oil, an optimal amount of silica or bleaching earth should be established for oils with different qualities to achieve the desired bleach and to minimize the amount of the bleaching earth required. The added cost of silica, which is three to five times more expensive than bleaching earth, is usually justified by the reduction in need for bleaching earth and the associated entrained oil loss.

Oil quality changes during refining. The quality changes after degumming, neutralizing, and bleaching wheat germ oil are presented in Table 1. The qualities of two commercial wheat germ oils were also measured and compared with our oils. The phosphorus content was significantly reduced after each step of processing; however, degumming did not reduce phosphorus as effectively as did neutralizing. The alkali significantly helped to hydrate the phospholipids. In addition, the soap formed may act as a good adsorbent for the phospholipids and other undesired oil components. The FFA content increased during degumming, possibly due to the residual phosphoric acid used to precondition the oil.

The bleached oil contained more FFA than did the neutralized oil, because both the synthetic silica and bleaching earth used in the bleaching step were acidic in nature, and the residual soap can be converted back to FFA. Furthermore, the acidic bleaching earth could have catalyzed the hydrolysis of triacyl-

TABLE 1
Effects of Processing on the Quality of Wheat Germ Oil[a]

	Phosphorus (ppm)	FFA (%)	PV (meq/kg)	Color (red, 1″)	AOM (h)	Tocopherol content (ppm) α	β	Total
Crude	1428 a	15.7 b	20.36 a	15.0 a	7.7 b	1817 a	864 a	2682 a
Degummed	1082 b	17.1 a	20.41 a	13.9 a	>60 a	1754 a	875 a	2628 a
Neutralized	99 c	0.09 c	20.60 a	8.3 b	1.2 c	1609 a	757 b	2366 a
Bleached	22 c	0.17 c	0.48 b	1.8 c	7.1 b	1718 a	751 b	2469 a
Cold-processed[b]	786	1.69	16.52	4.0	7.5	1810	720	2530
Cold-pressed[b]	74	0.30	64.53	6.1	3.2	3518	321	3839
$LSD_{0.05}$	103	0.26	4.37	1.3	1.1	400	36	402

[a]Means with different letters in the same column are significantly different at the 5% level.
[b]Obtained from Arista Industries, Inc. (Darien, CT). FFA, free fatty acid; PV, peroxide value; AOM, active oxygen method.

glycerols. PV was not significantly affected by degumming and neutralizing, but was significantly reduced after bleaching. The crude oil was very dark-colored, but neutralizing and bleaching significantly reduced the red color.

It is interesting to compare the oxidative stabilities of the oils after different processing steps. The degummed oil (>1000 ppm of residual phosphorus) was amazingly stable [more than 60-h active oxygen method (AOM)], whereas the neutralized oil was unexpectedly unstable (1.2-h AOM). It has been long recognized (15–18) that phospholipids have antioxidant activity, and phospholipids may have acted synergistically with the tocopherols. Along with the phosphoric acid, which was added to convert unhydratable phospholipid to hydratable form by chelating metal cations, the residual phospholipid may have significantly contributed to the stability of the degummed oil. Neutralizing removed both residual phospholipid and phosphoric acid, greatly reducing stability. The stability of the bleached oil was significantly increased compared with the neutralized oil due to the reduction in peroxides, which catalyze autooxidation.

The total tocopherol content did not significantly change during degumming, neutralizing, and bleaching, although β-tocopherol was reduced about 14% after neutralizing. The tocopherols are reduced during soybean oil processing, but the most significant reduction is during deodorization (5).

Tables 2 and 3 show the effects of deodorization conditions on FFA, PV, color, oxidative stability, and tocopherol content of wheat germ oil. Statistical analysis showed that there were no interactions ($P > 0.05$) between deodorization temperature and residence time for all quality parameters, except for tocopherol contents. When there is no interaction, it is sufficient to evaluate only the main effects of the two factors. With increased temperature and increased residence time, more FFA was distilled, more peroxide was destroyed, and more heat bleaching was accomplished. But the oxidative stability of the deodorized oil did not significantly differ among different deodorization treatments. Nevertheless, the deodorized oil was slightly more stable than was the bleached oil. It has been reported that deodorized soybean, corn, cottonseed, and olive oils have slower oxidation rates than do the corresponding bleached oils (5,19). By using linear regression, we estimate that with each 10°C increase in temperature, there are reductions of 0.02% in FFA, 0.083 meq/kg in PV, and 0.3 Lovibond units of red color. With each 10-min increase in residence time, the corresponding reductions are 0.02%, 0.18 meq/kg, and 0.7 in red color.

Deodorization conditions greatly impacted tocopherol contents of wheat germ oil. The effect of residence time affected tocopherol retention differently at different temperatures. Tocopherol contents of the oils deodorized at 200 and 250°C were not significantly different at different residence times. When the oil was deodorized at 290°C, deodorization time significantly affected tocopherol reduction. The reductions in α, β, and total tocopherol contents after 9 min of residence time were 25, 32, and 28% of that at 4 min of residence time. These reductions at 30 min of residence time were 60, 68, and 63% of that at 4 min of residence time. These observations indicate that at lower temperatures, the loss of tocopherol is not as sensitive to residence time as at higher temperature. Therefore, lower temperature and longer residence time

TABLE 2
Effects of Deodorization Conditions on Quality of Wheat Germ Oil[a]

Quality	Flow rate (mL/min)	Residence time (min)	Temperature (°C) 290	250	200	Mean
FFA	25	4	0.09	0.20	0.29	0.19 a
(%)	10	9	0.04	0.10	0.20	0.11 b
	3	30	0.04	0.08	0.20	0.11 b
Mean			0.05 c	0.13 b	0.23 a	$LSD_{0.05} = 0.07$
PV	25	4	0.54	0.88	1.26	0.89 a
(meq/kg)	10	9	0.14	0.71	1.14	0.66 b
	3	30	0.15	0.31	0.68	0.38 c
Mean			0.27 c	0.63 b	1.02 a	$LSD_{0.05} = 0.21$
Color	25	4	10.0	11.2	11.3	10.8 a
(red, 5.25″)	10	9	8.2	11.3	11.8	10.4 a
	3	30	6.9	9.1	10.6	8.9 b
Mean			8.3 c	10.5 b	11.2 a	$LSD_{0.05} = 0.8$
AOM	25	4	7.2	8.0	8.3	7.9 a
(h)	10	9	7.6	7.8	8.1	7.8 a
	3	30	8.4	7.5	7.5	7.8 a
Mean			7.7 a	7.8 a	8.0 a	$LSD_{0.05} = 1.4$

[a]Means with different roman letters in the same column or row are significantly different at the 5% level. See Table 1 for abbreviations.

TABLE 3
Effects of Deodorization Conditions on Tocopherol (toco) Retention in Wheat Germ Oil[a]

Quality	Flow rate (mL/min)	Residence time (min)	Temperature (°C) 290	250	200	Mean
α-Toco	25	4	2186	1946	1955	2029 a
(ppm)	10	9	1629	1903	1855	1795 b
	3	30	867	1862	1860	1463 c
Mean			1560 b	1912 a	1890 a	$LSD_{0.05}$ = 198
β-Toco	25	4	894	797	770	820 a
(ppm)	10	9	604	759	755	706 b
	3	30	286	749	762	569 c
Mean			595 b	772 a	762 a	$LSD_{0.05}$ = 103
Total toco	25	4	3080	2743	2725	2849 a
(ppm)	10	9	2233	2662	2610	2501 b
	3	30	1153	2610	2622	2032 c
Mean			2155 b	2684 a	2652 a	$LSD_{0.05}$ = 298

[a]Means with different roman letters in the same column or row are significantly different at the 5% level.

are effective means in reducing FFA, PV, and color, while retaining maximal tocopherol content.

The formation of *trans* fatty acid during each processing step and during various deodorization treatments was also examined. Degumming, neutralizing, and bleaching did not induce significant *trans* formation. Deodorizing at extreme conditions (290°C column temperature and 30-min residence time) produced about 0.7% total *trans* fatty acids. Even though the oil was treated at very high temperature, the total heat exposure time used in this study was shorter than is typical in conventional tray-type deodorizers; therefore the degree of *trans* isomerization was relatively low. The formation of *trans* isomers is dependent not only on temperature and residence time in the column, but also on the degree of unsaturation of the oil. Deodorized soybean oil typically contains 1–2% *trans* fatty acid due to its high C18:3 content (20).

The fatty acid compositions of our wheat germ oils were also determined and compared with literature values (Table 4). The compositions varied considerably, possibly due to the differences in wheat variety and growing region,

TABLE 4
Fatty Acid Compositions of Wheat Germ Oils

Source	Oil sample	Fatty acid composition (%) 16:0	18:0	18:1	18:2	18:3	20:0	20:1
Literature	Lab-extracted 1[a]	17.4	0.9	12.3	58.0	11.4	—	—
	Lab-extracted 2[a]	17.5	0.5	13.8	59.3	8.8	—	—
	Commercial 1[a]	12.3	2.0	19.3	61.2	5.2	—	—
	Commercial 2[a]	13.7	1.5	21.8	57.9	5.1	—	—
	Commercial[b]	14–16	1–6	8–30	44–65	4–10	—	—
This report	Cold-processed[c]	16.4	0.7	16.4	58.0	6.8	0.2	1.6
	Cold-pressed[c]	11.2	1.6	26.1	58.6	1.8	0.3	0.4
	Crude	15.8	0.7	17.5	57.9	6.1	0.2	1.7
	Degummed	15.5	0.7	17.3	58.4	6.3	0.2	1.6
	Neutralized	16.3	0.8	18.1	56.6	6.1	0.2	1.9
	Bleached	16.3	0.9	18.2	56.5	6.0	0.2	1.9
	Deodorized 1[d]	16.7	0.8	17.8	56.6	6.1	0.2	1.8
	Deodorized 2[e]	16.3	0.9	18.2	56.7	5.8	0.2	1.9

[a]Reference 2.
[b]Reference 3.
[c]Obtained from Arista Industries, Inc. (Darien, CT).
[d]Deodorized at oil flow rate of 10 mL/min or at residence time of 9 min, column temperature of 250°C.
[e]Deodorized at oil flow rate of 3 mL/min or at residence time of 30 min, column temperature of 290°C.

and possibly contamination with other oils. The presence of long-chain fatty acids has been mentioned in some literature (2), but there was no information on the quantitative analysis. In this study, we have quantified C20:0 and C20:1 fatty acids, which accounted for about 2% of the total fatty acid. It is worth noting that the neutralized oil had a higher percentage of palmitic, stearic, and oleic acids, and a lower percentage of linoleic and linolenic acids, when compared with the crude and degummed oils. The FFA that was removed after neutralization must have been rich in polyunsaturated fatty acids, indicating that the triacylglycerols were selectively hydrolyzed during germ preparation and oil extraction. This result contradicts the finding of Morrison (21), in which the composition of the FFA was found to be similar to that of the total fatty acid in the simple glyceride, and the author attributed this observation to the random hydrolysis of the triglycerides by lipases. Another possible reason for the decrease in the unsaturated fatty acids, especially linoleate, in the neutralized oil is the removal of the phospholipids that usually contain more polyunsaturated fatty acids compared with the neutral oil. However, this may only contribute a small part to the fatty acid profile change.

Good-quality wheat germ oil, with relatively high levels of tocopherols, can be produced by conventional oil refining techniques; however, the high levels of phosphorus and FFA are problematic. The preferred method of refining wheat germ oil involves degumming with acid pretreatment, at elevated temperature and high shear, for longer times than typical of other vegetable oils to maximize phospholipid hydration. Multiple stages of degumming may be necessary to remove as much phospholipid as possible to reduce refining loss. Neutralizing the FFA at higher than usual amounts of excess alkali to completely neutralize the FFA and to maximize phospholipid hydration and adsorption may be required. Bleaching with high levels of bleaching earth to remove the pigment and peroxides may be required. Deodorizing at the appropriate combination of temperature and oil flow rate to maximize tocopherol retention and to minimize FFA, PV, and color development is desired. Different deodorization system designs and operating conditions can have profound effects on tocopherol retention.

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