Pretreatment of Corn Oil for Physical Refining

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ABSTRACT: Crude corn oil that contained 380 ppm of phosphorus and 5% of free fatty acids was degummed, bleached, and winterized for physical refining. The pretreatment and the steam-refining conditions were studied in pilot plant scale (2 kg/batch). The efficiency of wet degumming and of the total degumming processes, at different temperatures, was evaluated. TriSyl silica was tested as an auxiliary agent in the reduction of the phosphorus content before bleaching. The experimental conditions of the physical refining were: temperature at 240 or 250°C; 8 to 18 mbar vacuum, and distillation time varying from 1 to 3 h. Degumming at 10 or 30°C resulted in the removal of more phosphorus than at 70°C. Water degumming was more efficient than the processes of total degumming or acid degumming. Corn oil, degummed at 10 or 30°C, after bleaching passed the cold test, irrespective of the degumming agent used. Degumming and winterization took place simultaneously at these temperatures. The pretreatment was able to reduce the phosphorus content to less than 5 ppm. The amount of bleaching earth was reduced by carrying out dry degumming or by using silica before bleaching. Corn oil acidity, after physical refining, varied from 0.49 to 1.87%, depending on the residence time. Contrary to alkali refining, physical refining did not promote color removal due to the fixation of pigments present in the crude corn oil.
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KEY WORDS: Bleaching, corn oil, degumming, physical refining, pretreatment, silica.

Most edible oils are produced through alkali refining, a well-known versatile process that can be applied to any crude oil. However, the physical refining process could be economically advantageous for oils with low levels of gums and high levels of free fatty acids (1–4). The main disadvantages presented by chemical refining, as compared to physical refining, are the losses of neutral oil that occur due to saponification and entrainment by the soapstock. Alkali refining requires centrifuges for neutralization and washing, implying higher investments and effluent treatment costs (5–8).

Physical refining also has its limitations because it can be applied to neither cottonseed oil nor fish oil (3). Sometimes, the lower oil losses attained by physical refining are not enough to compensate for the larger amount of bleaching earth required by this process (4,8,9). The acidity is not reduced to the same levels as achieved by chemical refining (1), and the quality of the final product is always determined by the quality of the crude oil (4,10).

Degumming prior to physical refining is a crucial preliminary step (10) because the stability of the oil is affected by traces of metals and phosphorus (9). According to Wesdorp (11), to obtain good-quality oils through physical refining, the phosphorus content of the oil must be less than 5 ppm before stripping. However, according to Cleenewerch and Dijkstra (10), the phosphorus content of the oil can be 10 ppm, but its iron content must be lower than 0.2 ppm because iron may cause darkening of the oil during distillation and reduce its oxidative stability.

Several patents cover the degumming process for physical refining, such as Unilever's superdegumming, Safinco's total degumming, Alfa Laval's special degumming, and Lurgi's Enzymax enzymic degumming (11–14).

Silica has been used in the refining process of oil for the adsorption of soaps and phospholipids, as in modified caustic refining, but it can also be used in physical refining as a bleaching auxiliary agent to remove residual phospholipids. In modified physical refining, soaps are generated by adding small amounts of caustic soda to the degummed oil to facilitate adsorptive removal of phospholipids (15,16).

In the pretreatment for physical refining of sunflowerseed oil, after degumming, silica treatment was used instead of bleaching (17). For physical refining of palm oil, after dry degumming with phosphoric acid, silica was used simultaneously with bleaching earth (16).

Both chemical and physical refining processes can be used for crude corn oil. It is generally accepted to use the alkali refining process, suppressing the degumming, with a concentration of caustic soda varying from 12 to 18° Bé, followed by washing, drying, bleaching with 0.09% of earth and deodorizing. Winterization can be carried out at a temperature between 5 to 10°C, up to 24 h. Deodorization occurs at 240–260°C under a pressure of 3 to 6 mm Hg. In physical refining, degumming can be carried out with 2 to 5% water or with citric or phosphoric acid and water. Bleaching requires a pretreatment with phosphoric or citric acid and 1 to 3% of activated earth (18).

In physical refining of corn oil by the Simon Rosendows System Ltd. process, 0.15% of phosphoric acid at 60–80°C for 20 min and a minimum amount of water are used for degumming, followed by centrifugation at high speed. After drying

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and bleaching, distillation occurs at 260°C and a pressure of 3 to 5 torr. The acidity decreases from 5.24 to 0.03%, and the Lovibond color from 36Y-4.2R to 34Y-2.5R (5.25" cell) for the bleached and the steam-refined oils, respectively. The phosphorus contents of the crude oil are reduced from 330 ppm to 50 and 12 ppm after degumming and bleaching, respectively (5).

Physical refining of corn oil with 23.6% free fatty acids can be carried out through the following process: wet degumming, followed by dry degumming with 0.3 to 0.5% of phosphoric acid and bleaching with 2% earth at 110°C, for 20–25 min. The distillation temperature varies from 220 to 250°C with 5% direct steam. The acidity can be reduced to levels of 0.3 to 1%, and postrefining becomes necessary (7).

Strecker *et al.* (18) reported the theoretical refining losses of crude corn oil with 1.5% free fatty acids, and concluded that chemical refining was advantageous. According to Leibovitz and Ruckenstein (7), alkali refining of a crude corn oil with a free fatty acid content between 8.4 and 14% results in a yield between 75 and 85%, while for the physical refining process a yield between 80 and 89% can be achieved.

In Brazil, crude corn oil undergoes direct neutralization, and degumming is suppressed. This process, when applied on an industrial scale to corn oil with a free fatty acid content of 4%, results in 14% losses during centrifugation, which may justify the use of the physical refining process.

Because there is little information about degumming and refining of corn oil, this work had the purpose of studying the pretreatment conditions for physical refining of crude corn oil produced in Brazil.

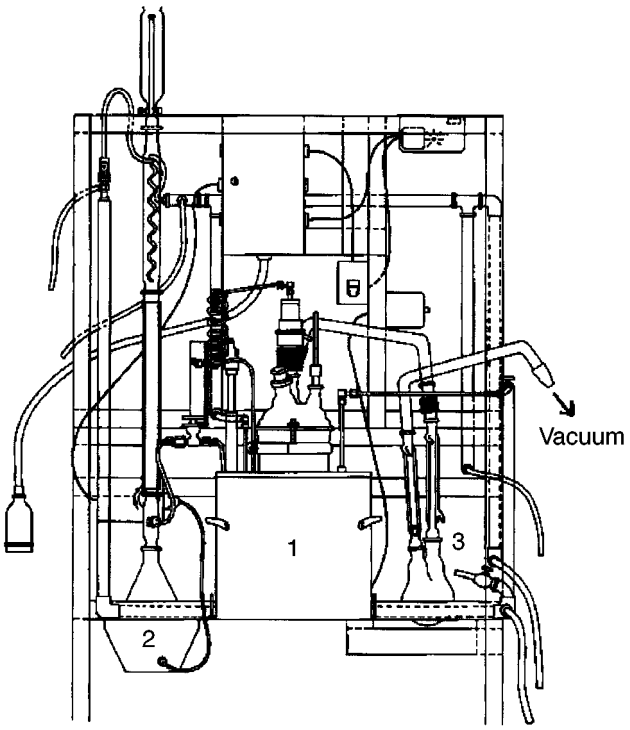

FIG. 1. Deodorization/distillation unit. (1) Thermal oil bath; (2) steam generator; (3) condenser.

EXPERIMENTAL PROCEDURES

Materials. Commercially available crude corn oil was supplied by Refinadora de Óleos Brasil Ltda. (São Caetano do Sul, SP, Brazil). Analytical-grade citric acid and commercially available lactic and phosphoric acids (c.a. 85%) were used for degumming. Other materials included TriSyl Silica 3000 (W.R. Grace Co., Baltimore, MD), silicic acid p.a., and bleaching earths Tonsil Optimum (Química Sumex, Mexico City, Mexico) and Pure Flo (Oil Dri, Chicago, IL).

Equipment. Glass reactors, with a capacity of 2 L/batch, were used for degumming, neutralization, bleaching, and winterization of the oil. The reactors were equipped with compressed air stirrers, thermostatic baths, vacuum pumps, filters, temperature programmers, and a refrigerated centrifuge (Heraeus Sepatech Suprafuge, Worms, Germany).

The deodorization/distillation unit, a 2-L glass vase, was immersed in a thermal oil bath with an electrical resistance. The steam generator system consisted of a heating mantle and a round flask connected to the distiller and the condenser. The steam, before entering the vase, circulated in metal tubing through the thermal oil. The distillate was collected in a round flask and connected to two heat exchangers for cooling, which in turn were connected to the vacuum pump (Fig. 1).

Analytical methods. Free fatty acid, phosphorus and soap content, and the cold test (6 h at 0°C) were carried out according to the American Oil Chemists' Society (19). Lovibond color was read in a Lovibond Tintometer model E in a 5.25" cell and was expressed in units of yellow (Y), red (R), and blue (B).

Physical refining. The conditions used in the tests (from A to I) are described below with the following steps: pretreatment (wet degumming, dry degumming, or silica treatment and bleaching), winterization, and deodorization/distillation.

Test A: The total degumming process (14,20) was applied to the crude corn oil with the addition of 0.05% phosphoric acid (85%), allowing 15 min of contact at 30°C; sodium hydroxide (calculated quantity based on the added acid) was added and 5 min of contact was allowed, then 2% water was added, and agitation was continued for 10 min. Centrifugation was carried out at 7,000 rpm for 10 min. The degummed oil was dried at 60°C for 30 min under 1 mbar vacuum, followed by bleaching with 1.5% Tonsil Optimum (w/w) at 90°C under vacuum for 30 min, and filtration under vacuum. The bleached oil was then winterized. The temperature was reduced from 30 to 0°C for 15 h, and the final temperature was maintained for 10 h to allow crystal formation and maturation. After filtration, deodorization/distillation was carried out at 240°C, with steam stripping, under 8 mbar vacuum for 3 h.

Test B: The total degumming process was applied to crude corn oil as in test A, but under 10°C. The degummed oil was dried and bleached with 2% Tonsil Optimum at 100°C. After bleaching, the oil was winterized as in test A, but the final temperature was maintained for 20 h. Steam refining was carried out at 250°C, under 7 mbar vacuum, for 1 h.

Test C: Crude corn oil was degummed with 2% water (w/w) at 70°C for 30 min. After drying, the oil was submitted to dry degumming with 0.1% citric acid (64%) at 30°C for 30 min, before bleaching at 100°C with 1.5% of Pure Flo B-80. Winterization was carried out from 30 to 5°C for 10 h, and the final temperature was maintained for 10 h.

Test D: The total degumming process was applied to crude corn oil with 0.46% citric acid (58.5%) during 60 min, with the addition of sodium hydroxide, allowing 5 min of contact, and of 2% water for 15 min at 30°C. Silica (0.2%) was then used for 15 min at 60°C, before the addition of 1.5% of Pure Flo at 100°C. Steam refining was carried out at 250°C for 1 h under 7 mbar vacuum.

Test E: Crude corn oil was degummed with 2% water at 30°C for 30 min. Sodium hydroxide was then added to the oil to form 500 ppm of soaps, followed by the addition of 0.2% silica at 60°C, which was maintained in contact for 15 min before bleaching with 1.5% of Pure Flo at 100°C. Steam refining was carried out at 250°C for 3 h under 10 mbar vacuum.

Test F: Crude corn oil was degummed with 0.05% phosphoric acid (85%) at 30°C for 15 min, followed by the addition of 2% water during 15 min. Pretreatment with silica and bleaching were similar to those carried out in test E, except that 1.5% of Tonsil Optimum was used. Steam refining was carried out at 250°C for 1.5 h under 18 mbar vacuum.

Test G: Crude corn oil was degummed with 0.1% citric acid (64%) at 30°C for 15 min, with the addition of 2% water and 15 min of contact. Pretreatment was similar to that carried out in test E, except that vacuum was employed with silica. Steam refining was carried out at 250°C for 2 h under 15 mbar vacuum.

Test H: Crude corn oil was degummed by the same procedure described in test A. The oil was then dried and bleached by the same procedures described in test B. Steam refining was carried out at 250°C for 3 h under 15 mbar vacuum.

Test I: Crude corn oil was degummed with lactic acid (85%) by the same procedure described in test G. The oil was dried with the addition of 0.2% silicic acid and 1.5% of Tonsil Optimum at 100°C. Steam refining was carried out at 240°C for 2 h under 11 mbar of vacuum.

Chemical refining. Crude corn oil was neutralized at 40°C during 15 min, with suppression of degumming. After centrifugation, the neutralized oil was washed, dried, and bleached with 1% of Tonsil Optimum FF at 100°C. Winterization was carried out by reducing the temperature from 30 to 5°C during 12.5 h and maintaining at the final temperature for 10 h. The winterized oil was deodorized at 250°C under 10 mbar vacuum for 2 h (experiment is codified as Test J).

RESULTS AND DISCUSSION

Degumming of crude corn oil with water at 30°C (Test E) and 70°C (Test C) resulted in the reduction of its phosphorus content to 14.5 and 23.3 ppm, respectively; this shows a higher efficiency when the process was carried out at the lower temperature (Table 1).

Usually, the degumming temperature varies from 60 to 90°C. However, Esteves *et al.* (21) observed a higher reduction of the phosphorus content at 30°C in water degumming of rice bran oil at temperatures varying from 30 to 70°C. For canola oil, Diosady (22) used temperatures varying from 20 to 80°C and observed a higher efficiency in phosphorus removal at 40°C.

Although lactic acid has not been reported as a degumming agent, when degumming was carried out with citric acid (Test G) and lactic acid (Test I), both at 30°C, a similar reduction in the phosphorus content of the oils to 26.8 and 26.9

TABLE 1
Phosphorus Content (ppm), Free Fatty Acid Content (% w/w), and Lovibond Color of Corn Oil Refined in the Pilot Plant

		Test									
Corn oil		A	B	C	D	E	F	G	H	I	J
Crude	P (ppm)[a]	381.03	381.03	374.71	367.38	381.03	367.38	367.38	374.89	374.89	374.89
	Lovibond color[b]	70-15	70-15	72-14	70-14	70-15	73-14	73-14	70-15	70-12-0.1	70-15
	% FFA[c]	5.10	5.10	—	—	5.10	—	—	—	5.03	4.94
Degummed	P (ppm)[a]	—	—	23.27	27.04	14.47	—	26.81	—	26.93	14.58[d]
	Lovibond color[b]	60-18	50-24	70-13	60-20	50-18	50-21	60-21	40-21-0.1	70-9	70-8-0.1[d]
	% FFA[c]	—	5.21	—	—	5.15	4.94	5.14	5.10	5.00	0.51[d]
Bleached	P (ppm)[a]	9.02	7.87	4.94	4.82	4.52	8.10	8.81	5.89	8.38	7.75
	Lovibond color[b]	40-11	30-8	30-6	30-8	40-9	40-8	50-9	30-6.6	60-8	60-3
	% FFA[c]	4.87	5.06	5.26	4.91	5.09	4.89	4.97	4.91	4.88	0.46
Winterized	P (ppm)[a]	7.33	8.04	4.79	—	—	—	—	—	—	8.54
	Lovibond color[b]	30-10	40-8	40-7	—	—	—	—	—	—	50-4.2
	% FFA[c]	4.87	4.77	5.32	—	—	—	—	—	—	0.53
Steam-refined	Lovibond color[b]	70-11-2	—	—	50-6	60-8-1	60-5	60-6-0.2	40-5	50-5.5	30-2.3
	% FFA[c]	0.67	—	—	1.87	0.75	0.79	0.73	0.49	0.74	0.26

[a]Phosphorus content (ppm).
[b]Yellow–red–blue (5.25" cell).
[c]Free fatty acid content (expressed as % oleic acid).
[d]Neutralized corn oil.

ppm, respectively, was observed. Total degumming of crude corn oil with citric acid at 30°C reduced the phosphorus content to 27.0 ppm (Test D). The phosphorus content of the oil degummed with phosphoric acid was not determined because residual traces of the acid could be present in the oil.

Comparing the results obtained, the degumming of crude corn oil with water at 30°C showed the highest efficiency (Test E). Phosphorus removal for the total degumming process with citric acid, water degumming at 70°C, and acid degumming with lactic or citric acids at 30°C was similar.

The ideal pH for degumming of high-acidity corn oil should be close to neutrality because the acids added during the process would interfere with gum hydration, possibly increasing the solubility in the oil.

Alkali refining (Test J) reduced the Lovibond color from 70Y-15R to 70Y-8R-0.1B (Table 1). Similar reduction of the red component was observed when degumming was carried out with lactic acid.

Because the degummed oils contained phosphorus at 14.47 to 27.04 ppm, dry degumming and pretreatment with silica were compared as alternative steps before bleaching to remove residual phosphatides.

After total degumming with phosphoric acid and bleaching, the phosphorus contents were reduced to 9.02, 7.87, and 5.89 ppm for tests A, B and H, respectively (Table 1). The Lovibond colors obtained for the bleached oil in test A (1.5% bleaching earth) and in tests B and H (2% bleaching earth) were 40Y-11R, 30Y-8R, and 30Y-6.6R, respectively.

The oil degummed with phosphoric acid (Test F), after silica treatment and bleaching with 1.5% of Tonsil Optimum, had a phosphorus content of 8.10 ppm and the Lovibond color of 40Y-8R. The silica treatment reduced the amount of bleaching earth when compared to the total degumming process with phosphoric acid.

After the total degumming process with citric acid (Test D), silica was used before bleaching with 1.5% of earth, which reduced the phosphorus content from 27.04 to 4.82 ppm and the Lovibond color to 30Y-8R.

Dry degumming with citric acid of the oil that was previously degummed with water at 70°C (Test C), followed by bleaching with 1.5% of earth, reduced the phosphorus content from 23.27 to 4.94 ppm and the Lovibond color to 30Y-6R.

When oils degummed with water (Test E) or citric acid (Test G) at 30°C were treated with silica in the presence of 500 ppm of soaps, followed by bleaching with 1.5% of earth, a reduction was observed in the phosphorus content from 14.47 to 4.52 ppm and from 26.81 to 8.81 ppm, respectively.

The utilization of silica reduced the amount of bleaching earth from 2 to 1.5% and presented a higher efficiency in phosphorus removal under vacuum without the addition of soaps.

Silicic acid was added simultaneously with 1.5% of earth to the oil degummed with lactic acid (Test I) and reduced the phosphorus content from 26.93 to 8.38 ppm.

Dry degumming as well as silica treatment, followed by bleaching, reduced the phosphorus content of the bleached oils below 5 ppm.

The neutralized oil was bleached with 1.0% of earth, resulting in a reduction of the phosphorus content from 14.58 to 7.75 ppm. The Lovibond color decreased from 70Y-8R-0.1B to 60Y-3R, which was the best color removal observed in all bleaching tests carried out in this work.

All bleached oils passed the cold test (0°C for 6 h) when degumming was conducted at 10 or 30°C, irrespective of the degumming agent used. Simultaneous removal of gums and waxes occurred at these temperatures.

The bleached oils obtained in tests A, B, C, and J were winterized, and the filtration rate and the amount of obtained wax were evaluated. During winterization of the bleached oil in Test B, which was degummed at 10°C, no waxes were retained in the filter. As a result, the filtration rate was very high (194 g/min). Thus, during degumming at 10ºC, the waxes were carried by and removed with the gums.

The winterized oil in Test A was filtered at a rate of 75 g/min, and a small wax residue was obtained. Therefore, when degumming was carried out at 30°C, only part of the wax was removed by the gums, but the winterized oil passed the cold test. To avoid winterization of corn oil, degumming should be carried out at temperatures below 30°C.

When compared to the other tests, the filtration rate of the winterized oil in Test C (previously degummed at 70°C) was excessively slow, 28 g/min. After filtration, the winterized oil did not pass the cold test. For Test J (alkali refining), the filtration rate of the winterized oil was 140 g/min, in spite of the large amount of wax obtained. The winterized oil passed the cold test. There was no wax removal before winterization in tests C and J. Filtration rate differences could be explained by the presence of free fatty acids, phosphatides, and partial glycerides that do not allow crystal formation. In spite of the lower phosphorus content of the bleached oil obtained in Test C, when compared to the oil obtained in Test J, neutralization removed most of the free fatty acids and probably a fraction of its partial glycerides. Thus, the winterization process of the neutralized oil was subjected to less interference than the oil in Test C, which presented a high content of free fatty acids.

Steam refining at 240°C for 3 h at 8 mbar vacuum reduced the acidity from 4.87 to 0.67% (Test A). At the same temperature, however, at 11 mbar vacuum and a residence time of 2 h (Test I), it was possible to reduce the acidity from 4.88 to 0.74% (Table 1).

Steam stripping at 250°C for 1 h at 7 mbar vacuum resulted in a reduction of the free fatty acid content from 4.91 to 1.87% (Test D). At the same temperature, distillation for 3 h at 10 mbar vacuum, and for 1.5 h at 18 mbar vacuum decreased the acidity from 5.09 to 0.75% (Test E) and from 4.89 to 0.79% (Test F), respectively. Steam refining at 250°C, 15 mbar vacuum, for 2 h (Test G) and 3 h (Test H), reduced the free fatty acid content from 4.97 to 0.73% and 4.91 to 0.49%, respectively.

Deacidification showed a higher dependency on residence time than on temperature and vacuum conditions used. However, after 1.5 h of distillation, the results were quite similar.

In alkali refining, after 2 h of deodorization at 250°C and 10 mbar vacuum, the acidity was reduced from 0.53 to 0.26%

(Test J). Therefore, it was not possible to reduce the acidity of the steam-refined oils to less than 0.05%, possibly due to the small area of contact between the oil and steam, reflux of the free fatty acids, or hydrolysis of the triglycerides.

In relation to the results obtained for the Lovibond color of the steam-refined oils, a small reduction of the red units was observed in this process. In some oils a blue component appeared.

Forster and Harper (5), studying physical refining of corn oil, observed a small variation of the Lovibond color between bleached and distilled oils, 36Y-4.2R to 34Y-2.5R (5.25" cell), respectively. The final color was acceptable but different from the color of the bleached oil obtained in this work.

For the oil winterized in Test A, with 7.33 ppm of phosphorus, the Lovibond color varied from 30Y-11R to 70Y-11R-2B, after distillation, indicating the possible occurrence of color fixation and alteration due to the presence of pigments that were not removed during bleaching.

The alteration of the final color of the steam-refined oil was probably not due to the residual phosphorus content in the bleached oil because a similar result was obtained when the phosphorus content was below 5 ppm. In the chemical refining process, the phosphorus content of the winterized oil (8.54 ppm) did not compromise the color of the deodorized oil. The Lovibond colors of the winterized and deodorized oils were 50Y-4.2R and 30Y-2.3R, respectively.

Darkening of the oil during physical refining was related to its phosphorus and iron contents (10) and could be due to the oxidation of colorless compounds or the existing color pigments becoming fixed by high temperatures (6). The color of crude corn oil is due to the formation of Maillard reaction compounds because the thermal treatment of the germ before extraction is carried out at high temperature.

Unlike alkali refining, bleaching of the degummed corn oil was not efficient to remove these compounds, which do not undergo decomposition during the physical refining. Possibly to obtain corn oil with an acceptable color through physical refining, thermal treatment of the germ should be controlled to avoid burning and darkening of the crude oil and consequent color fixation.

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