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The Flavor Problem of Soybean Oil. XI. Phytic Acid as an Inactivating Agent for Trace Metals[1]

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WITHIN the past few years the addition of inactivating agents to inhibit the possible chemical effect of trace metals in soybean oil has become an accepted processing technique. Soybean oil has a high tocopherol content and is very sensitive to metallic contamination, thus making it an excellent oil in which to study trace-metal inactivation. The removal of pro-oxidant metal traces reduces the rate of oxidation and allows for the extended functioning of the antioxidant. This paper reports on our studies with phytic acid as a metal-inactivator for soybean and other edible oils.

Phytic Acid

Interest in phytic acid previously has centered largely in nutritional and biochemical investigations, and it was in such studies that the metal-binding capacity of phytic acid was first observed. The ability of phytic acid to remove catalytic amounts of iron and copper from gelatin has been patented by Grettie (9), also by Pedersen (18) for the stabilization of mercaptans. Both rice bran (13) and oat "flour" (16), which are the finely ground bran coat, have received some attention as antioxidants for fats and foods. It is likely that phytic acid was an important constituent of these preparations. Other examples of phosphorus containing products which were active as antioxidants were reported by Hilditch, *et al.* (8, 12).

Phytic acid, the hexaphosphoric acid ester of inositol, occurs as the mixed calcium, magnesium, or potassium salts in all seeds, and these salts are collectively referred to as phytin. In soybeans, phytin is reported (6) to make up about 75% of the phosphorus content of the seed. An excellent literature review on phytin and phytic acid has been prepared (20) covering references up to 1950.

The present commercial source of phytin is corn steep liquor, from which phytic acid is precipitated by the addition of lime. Crude phytic acid can be prepared easily in the laboratory by dissolving the commercial phytin in a minimum amount of 6% hydrochloric acid. The solution is then diluted to reduce the concentration of hydrochloric acid to less than 1%, filtered, and demineralized by use of a high capacity ion exchange resin. Later the hydrochloric acid is removed by one or two vacuum evaporations. The darkening which occurs during stripping of the hydrochloric acid is removed by slurrying with carbon and filtering. In this simplified procedure, nonvolatile acids present in the original phytin will also tend to concentrate with the phytic acid. To obtain pure phytic acid repeated precipitations with barium or iron must subsequently be carried out.

Methods for the determination of phytic acid leave much to be desired. All methods depend upon an iron titration or some modification of the original method of Heubner and Stadler (11). In addition to the iron titration, the determination of total phosphorus, free orthophosphate, and an acidity titration to the methyl orange end-point are helpful in characterizing phytic acid solutions. The first replaceable hydrogen shows a distinct end-point, and apparently quantitative results are obtainable. The titration of the second replaceable hydrogen (phenolphthalein end-point) is not equivalent to the first, and the titration end-point is very poor. Electrometric titration curves show only a small dip at this point. It has been found that the phosphoric acid content of various samples of phytic acid have varied from 2.0 to 17.0%. A further complication in the scheme of analysis arises because the phosphoric acid content of phytic acid solutions increases as the sample ages and darkens. Apparently the phytic acid molecule retrogrades, splitting off phosphoric acid. In a phytic acid sample stripped of hydrochloric acid, the phosphoric acid increased from 5.88 to 6.80% within 4 months. Phytic acid does not give the molybdenum blue test,

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and it is assumed that all orthophosphate present in the phytic solutions is present as free phosphoric acid.

Reports in the literature on the stability of phytic acid are quite contradictory (1, 2, 10, 22). Samples of phytic acid containing relatively large amounts of hydrochloric acid have been observed to remain perfectly clear over a period of two years. These same samples when subjected to vacuum stripping and concentrated to a thick syrup, followed by dilution with distilled water, showed considerable darkening, even after only one such operation. Phytic acid solutions cannot be dehydrated by a toluene distillation method because of the darkening and charring. Anderson (1) reported that phytic acid could be boiled for hours in dilute hydrochloric or sulfuric acid and remain relatively stable. Fleury (7) reported a maximum stability toward hydrolysis at an acid strength of 5 N.

Metal Inactivation

In exploring the trace metal inactivating properties of any new compound, we have employed a screening test in which 0.01% of such compound is added to soybean oil just prior to deodorization. The effectiveness of the material is measured by peroxide development in the oil after 8 hours under A.O.M. conditions. An oil of low stability is usually used in this test so that the relative degree of effectiveness between compounds can be more readily ascertained. The index of efficiency of any compound in preventing oxidation is the ratio of the peroxide value of the control material to that of the test material. Compounds showing high activity may have an index between 3 and 10, but, in the presence of added metals, the index may go as high as 30 or 40.

TABLE I

Effect of Phytic Acid on the Oxidation of Soybean Oil

Concentration of inactivator		Oil No.	Fe added	Cu added	Peroxide value A.O.M. 8 hours—100°C.
per cent			*p.p.m.*	*p.p.m.*	
Phytic	0.1	A	....	.....	7
Phytic	.01		....	.....	13
Phytic	.001		....	.....	58
Control			....	.....	64
Citric	.01		....	.....	47
Phytic	0.1	B	....	0.1	6
Phytic	.01		....	0.1	3
Phytic	.01		....	.01	3
Control			....	.....	40
Phytic	.01	C	....	.....	2
Phytic	.01		0.3	.....	2
Control			0.3	.....	55
Control			....	.....	11
Phytic	.001	D	0.1	.03	61
Phytic	.005		0.1	.03	26
Phytic	.025		0.1	.03	10
Control			....	.....	81
Phytic	0.005	E	....	.....	13
Phytic	0.05		....	.....	4
Phytic	0.5		....	.....	7
Control			....	.....	19

Phytic acid appears not only promising as a deactivator, but it is as effective as any of those which we have tested. The effect of concentration and the activity against added copper and iron is shown in Table I. Soybean oils from several different sources have been tested, and all have shown comparable results. The effectiveness of phytic acid in retarding the oxidative peroxide development in the presence of added metals is adequately demonstrated by the data presented in this and the following tables. The effective range of phytic acid was found to be above .005% and below 1.0%. Concentrations of 0.5% were found to be inferior to those of 0.1% concentration in both the taste and oxidative tests; however, even at the higher concentration, the atypical cucurbitaceous flavor developed in oils treated with phosphoric acid was not observed.

TABLE II

Phosphorus Balance

Phosphorus	Deodorization	
	Control 0.01% phytic	With added iron 0.01% phytic + 0.3 p.p.m. Fe
Phosphorus added:		
In oil percentage	0.0002%	0.0002%
In oil	1.82 mg.	1.82 mg.
As phytic	23.05 mg.	23.05 mg.
As orthophosphate	2.45 mg.	2.45 mg.
Total	27.32 mg.	27.32 mg.
Phosphorus found:		
In oil percentage	0.0009%	0.00067%
In oil	8.54 mg.	6.33 mg.
In deodorizer residue	14.20 mg.	18.50 mg.
Total	22.74 mg.	24.83 mg.
Phosphorus unaccounted for	4.58 mg.	2.49 mg.
Phosphorus unaccounted percentage	16.75 %	9.78 %
Added phosphorus recovered in oil	26.35	17.68

Phytic acid solution analysis	%
Total phosphorus	9.85
Orthophosphate	0.96
Orthophosphate as H_3PO_4	3.03
Phytic acid (by difference)	31.52
Phytic acid found by iron titration	34.77
Phytic acid (found by methyl orange titration, corrected for phosphoric acid)	30.91

The stability of phytic acid to deodorization conditions is a moot question. Analysis of several oils treated with a minimum amount of phytic acid have failed to show any increase in phosphorus contents. These same samples however have shown a marked improvement in their oxidative stabilities. Other samples treated with different lots of phytic acid have shown an increase in their phosphorus contents.

We have been unable to make a satisfactory phosphorus balance, but, in laboratory deodorization experiments employing a phytic acid solution containing 3.03% phosphoric acid, 50-60% of the added phytic acid can be recovered as a water-soluble material from the residues remaining in the deodorization flasks. From 20 to 25% of the phosphorus was found in the oil, and 15 to 20% of the phosphorus was unaccounted for. It appears that phytic acid is almost completely oil-insoluble, and any increase in the phosphorus content of the oil is a result of either the addition of phosphoric acid from the phytic acid or from the breakdown of phytic acid. On this assumption the breakdown of phytic acid during deodorization could be estimated at about 10%. The addition of phosphoric acid prior to deodorization has shown essentially 100% recovery of the phosphorus in the oil. Table II shows the results of two deodorizations using phytic acid.

Phytic acid was not effective as an inactivator when added directly to the oil without going through a deodorization. Whether the lack of activity is a result of the insolubility of the acid or because its activity depends on some breakdown product cannot be established at this time. Oxidative stability tests have shown that samples deodorized with phytic acid exhibit activity toward subsequent metal contamination occurring after deodorization. This is in contrast to citric acid and a number of other heat labile metal

scavengers which we have tested. This carry-through effect may be due to phosphoric acid as it is greater in tests employing phytic acid that had a higher phosphoric acid content.

A greater than normal reduction in color is commonly observed during the deodorization of oil samples containing phytic acid. Occasionally a small amount of charring is observed in the deodorizer flasks to which phytic acid has been added. This probably results from adding phytic acid as a concentrated solution, which immediately settles to the bottom of the flask and consequently is not uniformly dispersed in the oil. Although slight charring has been observed at times, no burned odor or flavor has ever been detected in the deodorized oil.

The improvement in the oxidative stability of various edible oils through the use of phytic acid is shown in Table III. The iron content of the corn oil was 0.24 p.p.m. and for the cottonseed oil was 0.22 p.p.m., values which would be considered about average for a good oil. The amount of iron in the peanut oil was very high: 1.45 p.p.m., and the iron content of the safflower oil No. 242 was very low, being only 0.042 p.p.m. Copper contents were about the same for all oils. Data are not available for the metal contents of the second sample of safflower oil.

TABLE III

Effect of Phytic Acid on the Oxidation of Edible Oils

Oil	Peroxide value A.O.M. 8 hours—100°C.	Index
Corn	26	7.4
Corn + 0.01% phytic	3.5	
Cottonseed	40	2.9
Cottonseed + 0.01% phytic	14	
Peanut	270	40.0
Peanut + 0.01% phytic	6.8	
Safflower No. 247	51	1.5
Safflower + 0.01% phytic	34	
Safflower No. 337	150	2.9
Safflower + 0.01% phytic	51	

Calcium soaps, like the heavy metal soaps, are oil-soluble, and their effects on the stability of soybean oil have never been adequately investigated. Analyses showed calcium to be a large constituent of the oil ash, but it is not known whether the calcium is a natural constituent of the oil or has been added as a contaminate during refining or water washing. Results presented in Table IV show the effects of added calcium oleate on the oxidative stability of soybean oil. The control of added calcium by phytic acid is also apparent from these data. Organoleptic evaluations made after four days' storage on oil samples containing increasing amounts of added calcium have not shown significant differences in flavor scores. The improvement shown in the oxidative stability would indicate that differences of flavor scores could be expected of these oils.

TABLE IV

Phytic Acid Treatment of Calcium-Contaminated Soybean Oil

Treatment	Peroxide value A.O.M. 8 hours—100°C.
Oil No. 338 control	31
Oil No. 338 + 0.01% phytic	4
Oil No. 338 + 15 p.p.m. Ca as oleate	44
Oil No. 338 + 15 p.p.m. Ca + 0.01% phytic	6.7
Oil No. 176 control	16
Oil No. 176 + 3 p.p.m. Ca as oleate	31
Oil No. 176 + 15 p.p.m. Ca as oleate	64
Oil No. 176 + 30 p.p.m. Ca as oleate	59

The organoleptic data on phytic acid-treated oils are comparable to the oxidative data in showing the superiority of the treated oils. Obviously, a close relationship exists between phosphoric and phytic acid. It is sufficient to point out here that amounts of only 1 p.p.m. of free phosphoric acid gives positive evidence of improved oxidative stability. This concentration of phosphoric acid is below the limits of phosphorus analysis in oils. The high sensitivity of phosphoric acid further complicates any explanation in attempting to separate the mode of action of phytic acid from that of phosphoric.

The organoleptic evaluation of soybean oil containing added iron and copper is shown in Table V. Highly significant results were obtained in favor of the samples containing phytic acid, both in the presence and absence of added metals. Organoleptically, the sample containing both added metal and phytic acid is equal to the control sample, and, in regard to oxidative stability, it is superior to the control sample. This run is typical of many organoleptic evaluations made for phytic acid-treated soybean oils and also shows the possible degree of control over any metallic contamination.

Pilot-plant investigations of phytic acid were made in our laboratory, employing 100-pound batches of oil. The results of these runs are shown in Table VI, where duplicate runs are shown for both the control and phytic acid-treated oils. The superiority of the treated samples is evident from the highly significant results obtained in these tests. The low scores obtained for the second control sample are a result of an air leak during deodorization. The instability of this sample is also shown by the higher peroxide values developed during storage.

TABLE V

Organoleptic Evaluation of Phytic Acid-Treated Soybean Oil

Phytic acid 0.01%	0.1 p.p.m. Fe 0.03 p.p.m. Cu	Phytic plus iron and copper	Control	Sig.[a] dif.
0 time				
8.6 (0.3)[b]	7.3 (0.2)	8.8 (0.2)	8.2 (0.3)	2**3
After 5 days—60°C.				
6.8 (1.8)		6.1 (1.8)		†
6.1 (1.8)			5.7 (1.8)	†
	3.2 (4.3)	5.5 (2.2)		**
6.2 (1.8)	3.3 (4.3)			**
		5.9 (2.2)	6.1 (1.8)	†
	3.7 (4.4)		7.0 (1.8)	**
Peroxide values A.O.M. conditions 8 hours				
2.4	43	2.2	15	

[a] † No significant difference; * Significant difference (5% level); ** Highly significant differences (1% level).
[b] Peroxide value at time of organoleptic evaluation shown in parenthesis.

Evaluations were also made of phytic acid-treated hydrogenated soybean oils deodorized in both the laboratory and in the pilot plant. Included for comparison were citric acid-treated oils, and the results of these tests are shown in Table VII. Phytic acid is again shown to be equal to citric acid in improving the stability of these oils.

TABLE VI

Organoleptic Evaluation of Phytic Acid-Treated Soybean Oil Pilot-Plant Prepared

Control	Phytic 0.01%	Control	Phytic 0.01%	Sig.[a] dif.
0 time				
6.9 (0.3)	6.9 (0.4)	4.8 (0.4)	7.8 (0.3)	
4 days—60°C.				
2.8 (1.1)	5.8 (1.4)			**
3.1 (1.1)			6.0 (1.3)	**
	6.0 (1.5)	3.0 (9.2)		**
4.0 (1.0)		3.7 (8.9)		†
	6.2 (1.4)		5.7 (1.3)	†
		3.5 (9.0)	6.5 (1.2)	**
Peroxide values A.O.M. conditions 8 hours				
75	1.5	71	1.8	

[a] See Table V for explanations.

Table VIII shows the result obtained in the evaluation of cottonseed oil to which both iron and phytic acid have been added. Although the effects of metallic contamination are much less in cottonseed oil than in soybean oil, these data show that definite improvements can also be made in the quality of cottonseed oil. From the data presented in this table and those shown in Table III, it can be noted that phytic acid has produced significant improvement in the stability of regular-processed edible oils.

Many investigators are hesitant to give acceptance to the theory of metallic inactivation because they can show instances where the addition of citric acid or other metal inactivators, such as phosphoric acid to distilled fatty esters, have greatly improved the stability of these materials which are supposedly free of trace metals (14, 17, 21). Distillation does not insure the absence of metals. Data on the metallic contents of distilled esters are lacking, probably because special methods are required to detect the presence or absence of many metals at the levels encountered here.

TABLE VII

Evaluation of Phytic Acid-Treated Hydrogenated Soybean Oil (laboratory deodorization)

Phytic acid 0.01%	Citric acid 0.01%	Control	Sig. dif.[a]
0 time			
6.8 (0.2)	5.9 (0.2)	3.4 (0.3)	1†2**3 1**3
Stored 4 days—60°C.			
5.0 (1.2)	4.3 (0.8)		†
	4.3 (0.9)	4.1 (2.0)	†
5.6 (1.9)		3.6 (2.2)	**
Peroxide values A.O.M. conditions 72 hours			
11	19	310	

[a] See Table V for explanations.

Lemon (14) in his investigation on the stabilizing effects of citric acid has reported 0.19 p.p.m. of iron in distilled methyl esters of peanut oil. This concentration of iron is far in excess of that needed to show the beneficial effects of phytic acid or citric acid in soybean oil. Woodle and Chandler (23), in their studies on petroleum distillates, concluded that the presence of metals in vacuum distillates is caused by actual volatilization of metal compounds indigenous to the crude oil.

The extreme sensitivity of phenolic-type antioxidants to trace metal contamination is not fully appreciated. An idea of this sensitivity can be gained from the data of Morris (15), where it is shown that 2 p.p.m. of iron lowered the stability of lard containing 0.01% lauryl gallate from 51 hours to 0.7 hour, and for 0.01% ethyl pyrogallol from 126 hours to 1 hour. Ten of the 11 antioxidants studied were phenolic, and all showed high sensitivity toward metal contamination. The synergistic effects of citric acid with four phenolic antioxidants in distilled methyl esters is reported by Stirton (21).

The extreme catalytic activity shown by trace metals and the sensitivity of both antioxidants and metal inactivators make it difficult to study the exact function of each in fat oxidation. However it is believed that as more information is accumulated, metal inactivation will explain the synergistic action of these "acid activators."

TABLE VIII

Organoleptic Evaluation of Phytic Acid-Treated Cottonseed Oil

Phytic acid 0.01%	Iron 0.3 p.p.m.	Phytic plus iron	Control	Sig.[a] dif.
0 time				
8.8 (0.4)	8.4 (0.5)	8.4 (0.4)	8.5 (0.5)	†
After 8 days—60°C.				
6.7 (2.4)	4.4 (12.9)			**
5.7 (2.3)			3.7 (2.4)	*
	3.5 (13.2)	5.6 (5.0)		**
5.2 (2.5)		5.5 (5.0)		†
	3.5 (13.2)		4.8 (3.0)	†
		4.8 (4.7)	3.5 (2.3)	†
Peroxide values A.O.M. conditions 8 hours				
14	49	18	40	

[a] See Table V for explanations.

No toxicity studies have been reported on phytic acid; however the Canadian government now permits the use of calcium phytate in certain food products (3). Grettie (9) has patented the use of phytic acid in the preparation of marshmallow whips from edible gelatin. Cohee (4, 5) has described its use in the clarification of vinegar and in preventing the discoloration of packed cherries. The concentrations proposed for use in edible oils are lower than that found in many foods. Phytic acid is a strong acid and in that respect would be toxic in high concentrations. Recent studies on the metabolism of radioactive phytin (19) have indicated that the labeled phosphorus will exchange with the phosphorus of the bones and tissues of the turkey poult. Whether phytin can be used for a net gain in phosphorus was not established.

Summary

Phytic acid, a natural constituent of cereal grains and oilseeds, is shown to be highly effective in the control of metallic contamination in edible oils. Phytic acid added to regularly processed edible oils at the 0.01% level has shown a marked improvement in the organoleptic and oxidative stability of these oils.

We could not establish whether the mode of action of phytic acid was the result of the direct formation of insoluble phytate salts, or the breakdown of phytic acid with the formation of phosphoric acid which would serve as the active agent. Phytic acid solutions

all contained some free phosphoric acid. Most phytic acid samples darkened with age and showed a progressive increase in the amount of free phosphoric acid. To be effective, it was necessary to add phytic acid prior to the deodorization process.

Evidence is presented to show that a large part of the synergistic effects of many activators can be explained by metallic inactivation. It is suggested that much of the antioxidant effect observed for rice bran, oat "flour," and cereal brans could be the result of metal inactivation by the phytic acid present in these materials.

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Steam Stripping of Lard[1]

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A STUDY was made to establish a means of evaluating the effect of steam stripping at high vacuum on removal of free fatty acids from lard. Normal industrial procedure is to supply steam through sparge pipes at the bottom of large vessels. The upward flow of steam bubbles provides agitation and stripping of volatile products. No data have heretofore been available to show the advantageous effect of adding mechanical agitation to this batch stripping procedure. Accordingly experiments were run, using rotating mixing turbines to disperse steam and to mix and agitate the batch thoroughly, also to determine optimum operating conditions of mixing for different rates of steam flow, pressure, and temperature. Stripping rates were increased as much as 50% with the use of mixing turbines. There was no difference in behavior between steam and nitrogen, hence steam acts as an inert gas in the stripping operation at the temperature involved.

Introduction

Oils and fats from natural sources contain small amounts of impurities in addition to the fatty glycerides. Included among these impurities are high molecular weight alcohols (sterols), hydrocarbons, free fatty acids, protein residues and other nitrogenous matter, phosphatides, and carotenoid pigments. These materials not only decompose into odoriferous substances but may act as catalysts, bacterial substrates, or inhibitors, either retarding or promoting reactions contributing to the spoilage of oils and fats (5).

Among the most abundant and difficult to remove of these substances is the free fatty acid, the presence of which in the oil is reported to cause an unpleasant taste, the so-called rancid taste. In order to eliminate this undesirable effect the concentration of free fatty acid in the lard must be reduced to less than 0.1% (1).

Countless methods have been employed in industrial processes to deacidify fats and oils including various means of neutralization by alkali and esterification, the removal of acids by extraction, steam distillation, and steam stripping. The steam stripping operation is the method most widely used in industry today for the deodorization of lard and hydrogenated vegetable oils. This stripping may be briefly described as the passing of steam through oil at a high temperature in a container in which a vacuum is maintained. This will effect the removal of not only free fatty acids but also all of the volatile impurities mentioned above. Although continuous steam stripping has been employed to a limited extent, the batchwise operation is by far the most widely used.

This investigation was undertaken to establish a means of evaluating the steam stripping effect under various conditions and to correlate the change in this effect with the variation in a given condition. The conditions varied were the amount of agitation by a mixing impeller, methods of introduction of the stripping steam, the absolute pressure in the stripping tank, the temperature of the liquid, and the rate of steam introduction.

A refined lard usually containing less than 1% free fatty acid was used, and a fresh batch was provided

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