Effects of Extraction, Solvents, Cold Pressing, and Period of Storage upon Oven Stability of Raw Peanut Oil

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ABSTRACT

Keeping time (oven stability, 60 C), oleate/linoleate ratios, peroxide values, and free fatty acid numbers were determined on oils from 5 peanut varieties grown at 2 Texas locations in 1972. Cold-pressing within polyethylene bags (bag-pressing) resulted in increased oven stabilities and better correlations between oleate/linoleate ratios and oven keeping times in comparison to cold-pressed oils. Average relative storage stabilities for the solvent extracted oils were: chloroform-methanol (3:1) $\gg$ cyclohexane $\cong$ ether $>$ acetone $\cong$ bag-pressed $>$ cold-pressed. Correlations of oleate/linoleate ratios and keeping times were increased using low peroxide ether in place of reagent grade ether. Extracted oils from samples including skins appeared to have longer keeping times than those minus skins. Oven keeping times and correlations with oleate/linoleate ratios decreased with extended peanut storage times (11 months), but the use of chloroform-methanol tended to compensate for this effect. For samples taken from storage after 3 and 6 months, the following average correlations were obtained: bag-pressed, 0.27; chloroform-methanol (without skins), 0.87; chloroform-methanol (with skins), 0.90; ether (reagent), 0.49; ether (low peroxide), 0.89; cyclohexane, 0.87; and acetone, 0.62. Generally poor correlations were found between oven stability and peroxide and free fatty acid numbers. Differences in the degree to which minor components are extracted from the cotyledons and skins, interaction between the oils, and residual traces of solvents or trace impurities in solvents were suggested as possible factors leading to differences in keeping times of solvent extracted oils.

INTRODUCTION

Recently, there has been increased interest in the composition of raw peanut oil (1-3) due to the possible relationship between the shelf life of peanut products and the fatty acid composition of raw peanuts. Several parameters, including peroxide and iodine numbers, free fatty acid content, oven stabilities of the expressed oils, and the oleate/linoleate (O/L) ratios of the oils, have been used to perdict the stability of peanut oils (1,4,5). More recently the use of O/L ratios and oven stabilities for predicting shelf lives has been extended to peanut butter and other roasted peanut products (3,6,7), but the applicability of these two parameters has not been evaluated thoroughly. Furthermore, differences between oven stabilities of solvent extracted and cold-pressed peanut oils have been reported, and the relationship between stabilities and O/L ratios is somewhat uncertain (1,3,6).

The possibility that O/L ratios or oven stability tests might offer a simple, rapid, reliable means of predicting the shelf lives of roasted peanut products a priori has led us to investigate these two parameters more thoroughly. The oven stabilities of oils extracted with five common solvents and hydraulically pressed oils and their O/L ratios have been determined. Relationships between oven stabilities, storage period, and O/L ratios also have been determined. The results of these experiments are based upon five varieties grown in both a North and a South Texas location.

MATERIALS AND METHODS

Peanuts used in preliminary experiments were from a commercial lot of 1971 crop Starr peanuts (splits) from Gorman, Tex., which had been held in cold storage for ca. 1 year. Peanuts used in later experiments were 1972 crop Starr, Spancross, TP 716-2-1, Wilco I and Florunner grown at Pearsall (South Texas) and Stephenville (North Texas) on irrigated plots. Samples were received in shell shortly after harvesting and curing. They were shelled within 2 weeks of receipt and passed over standard screens (15/64 x 3/4 in. for Spanish and 16/64 x 3/4 in. for the larger seeded Wilco I and Florunner). Peanuts riding the screens were hand sorted to remove immature and off-color nuts, and the resulting sound mature kernels were stored at 4 C in sealed glass jars.

Reagent grade solvents were used. Peroxide-free ether was prepared by column chromatography on activated alumina. Oil samples were prepared in three ways: (A) cold-pressing, (B) pressing in polyethylene bags (bag-pressing), or (C) solvent extracting the raw nuts three times using two volumes of solvent/g comminuted nuts. Solvent-oil mixtures were obtained by vacuum filtration, multiple extracts pooled, and the solvents were removed at room temperature over a period of 36 hr with the aid of the air currents drawn into a closed laboratory fume hood. Solvent dependent differences in oil recoveries were noted during solvent extraction. Samples without skins were prepared by removing the skins from the seeds by hand prior to extraction.

Bag-pressing was accomplished by wrapping the nuts in cheese cloth and then placing the samples insdie a 5 x 8 in. polyethylene bag. The bag then was placed into a second 7 x 10 in. polyethylene bag so that the mouth of the smaller bag nearly touched the bottom of the larger bag. The whole package was placed between two wooden blocks on a Carver laboratory press so that the bottom portion of the larger bag formed a receptacle external to the blocks. This arrangement allowed the expressed oil to accumulate in the base of the larger bag external to the blocks and mass of peanuts which were maintained under a pressure of 20,000 lb for 30 min.

Oven stabilities were determined using the modified procedure of Holley and Hammons (2). Samples of each oil (0.5 ml) were pipetted into each of three 30 ml beakers and were placed in a convection oven at 60 C. Samples were weighed and randomized daily until a wt increase of 1.0 mg was recorded. The average number of days (oven days) was recorded as the keeping time. Free fatty acids and peroxide numbers were determined according to the Association of Official Analytical Chemists' procedures (8).

Fatty acids in the oil were determined by gas liquid chromatography (GLC) after transesterification to their methyl esters by the procedure of Metcalfe, et al. (9). Esters were separated using a Beckman GC-5 chromatograph equipped with a flame ionization detector using a 6 ft x 0.14 in. outside diameter stainless steel column packed with 10% EGSS-X on Gas Chrom P and with the respective oven, injection port, and detector temperatures of 180, 250, and 230 C. Peak areas were calculated using a Disc

integrator or by triangulation, and the O/L ratios were calculated from the peak areas.

RESULTS AND DISCUSSION

Results of our preliminary experiments using samples (with skins) drawn from the commercial lot of Starr peanuts are shown in Figure 1. An increase in oven stability of ca. 20% (11.3 vs 9.5 days) was noted when the samples were prepared by pressing in the polyethylene bags. On the average, solvent extraction more than doubled the oven stability of the extracted oils in comparison to the cold-pressed samples. Choice of solvents apparently influenced the oven stability of the oil. The average oven stability of the chloroform-methanol (3:1) extracted samples exceeded 37 days. On the other hand, the oven stability of the acetone extracted oil was considerably lower than the other solvent extracted oils, and the order of stabilities was chloroform-methanol≫cyclohexane≅ether>acetone>bag-pressed>cold-pressed.

The short keeping times of the pressed samples probably reflected the fact that the peanuts had been in cold storage for nearly a year and, as a result, some deterioration in quality had occurred. However, yearly and seasonal effects are known to affect oil stabilities strongly (10) and also may have contributed to the short oven keeping times.

To answer the above questions and to investigate the cause of the prolonged oven keeping times, additional experiments using five peanut varieites grown at Pearsall, Tex., in 1972 were undertaken. The nuts had been in cold storage for ca. 3 months, and the preparation of samples was similar to the earlier experiments, except that the skins were removed prior to pressing and extraction. Two different sets of ether extracted oil were prepared: one using commercial reagent grade peroxide containing ether, and the second using low peroxide ether. The results of the tests are shown in Table I.

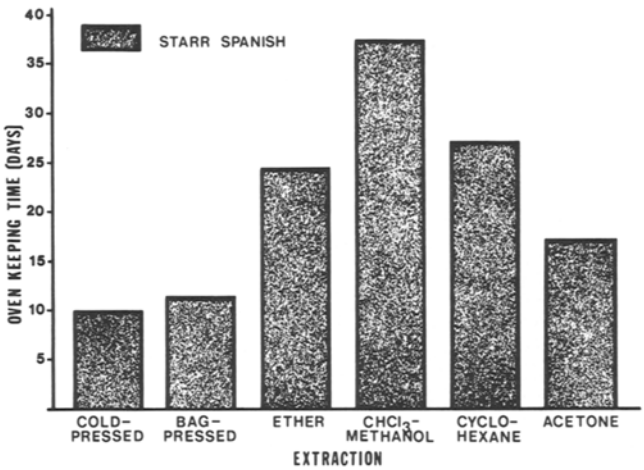

FIG. 1. Oven keeping times of oils from solvent extracted and hydraulically pressed 1971 crop Starr peanuts. Bag-pressed signifies that the sample was enclosed completely in a polyetylene bag during pressing to avoid direct contact with the press.

The order of stabilities for the oils from Pearsall, Tex., in 1972 was chloroform-methanol≫cyclohexane≅ether>acetone>bag-pressed>cold-pressed. The average keeping times of the oils extracted with reagent grade and low peroxide ether were ca. the same, although the values for individual varieites varied as much as 4 days. Solvent extraction again markedly extended, and bag-pressing slightly extended, the oven stabilities of the samples, but the increases were not as great as those recorded for the 1972 Starr peanuts. The use of chloroform-methanol (3:1) led to the greatest increase in stability in comparison to the cold-pressed oils. However, the average keeping times of the solvent extracted Spanish peanut oils were ca. 8 days shorter in 1973 than in 1972. Since the skins had been removed from the peanuts prior to

TABLE I

Oven Stabilities, Oleate/Linoleate Ratios, and Free Fatty Acid and Peroxide Numbers of Solvent Extracted and Hydraulically Pressed Oil Samples from 1972 Pearsall, Tex., Peanuts

Parameter	Variety				
	Starr	Spancross	TP 716-2-1	Wilco I	Florunner
			Cold-pressed		
Oven days	10.7	11.3	12.7	11.7	11.3
Oleate/linoleate ratio	1.22	1.33	1.21	1.41	1.60
Free fatty acids[a]	0.19	0.16	0.17	0.12	0.31
Peroxide number[b]	8.59	9.16	6.54	6.93	5.62
			Bag-pressed		
Oven days	12.3	12.0	11.3	14.3	11.3
Oleate/linoleate ratio	1.22	1.33	1.21	1.41	1.60
			$CHCl_3$-MeOH (3:1)		
Oven days	20.7	22.3	20.0	25.7	32.0
Oleate/linoleate ratio	1.25	1.30	1.26	1.52	1.69
			Ether (reagent)		
Oven days	16.0	17.3	19.0	23.0	19.7
Oleate/linoleate ratio	1.22	1.33	1.28	1.41	1.64
			Ether (low peroxide)		
Oven days	16.0	19.7	18.7	18.3	22.3
Oleate/linoleate ratio	1.22	1.33	1.28	1.41	1.64
			Cyclohexane		
Oven days	15.3	18.3	20.7	20.0	26.7
Oleate/linoleate ratio	1.22	1.33	1.25	1.41	1.64
			Acetone		
Oven days	15.7	15.0	15.3	---	---
Oleate/linoleate ratio	1.22	1.33	1.20	---	---

[a]Percent free fatty acids expressed as oleic acid in pressed samples at time of test. Freshly pressed values: 0.10, 0.13, 0.11, 0.15, and 0.12.

[b]Milliequivalents peroxide/kg sample in pressed samples at time of test. Freshly pressed values: 0.51, 0.63, 0.63, 0.46, and 0.61.

TABLE II

Oven Stabilities, Oleate/Linoleate Ratios, and Free Fatty Acid and Peroxide Numbers of Solvent Extracted and Hydraulically Pressed Oil Samples from 1972 Stephenville, Tex., Peanuts

Parameter	Variety				
	Starr	Spancross	TP 716-2-1	Wilco I	Florunner
			Bag-pressed		
Oven days	15.7	14.3	15.0	17.0	16.7
Oleate/linoleate ratio	1.14	1.18	1.15	1.21	1.43
Free fatty acids[a]	0.04	0.04	0.02	0.06	0.05
Peroxide number[b]	1.45	2.32	1.72	1.22	1.36
			$CHCl_3$-MeOH (3:1) without skins		
Oven days	26.7	19.0	20.3	23.3	33.3
Oleate/linoleate ratio	1.16	1.17	1.17	1.22	1.42
			$CHCl_3$-MeOH (3:1) with skins		
Oven days	22.7	14.3	26.7	36.0	36.0
Oleate/linoleate ratio	1.17	1.15	1.22	1.31	1.42
			Ether		
Oven days	16.3	16.7	20.0	17.0	24.3
Oleate/linoleate ratio	1.17	1.16	1.21	1.05	1.47
			Cyclohexane		
Oven days	18.7	12.7	17.7	19.3	25.3
Oleate/linoleate ratic	1.21	1.18	1.22	1.39	1.47
			Acetone		
Oven days	13.7	11.3	16.0	12.3	15.3
Oleate-linoleate ratio	1.24	1.19	1.23	1.15	1.43

[a]Percent free fatty acids expressed as oleic acid.

[b]Milliequivalents peroxide/kg sample.

oil extraction, the lower keeping times could incidate the presence of an extractable antioxidant in the skins or that the keeping quality of the 1973 oils was poorer than the oils from the Starr peanuts used in 1972.

Pressing the samples in polyethylene bags results in an average increase in oven stabilities of ca. 10% in comparison to the cold-pressed samples, and we consistently have recorded similar increases of 10-20% in favor of the bag-pressed samples in other experiments (unpublished results). It is also noteworthy that the oven keeping times of the cold-pressed oils from the Wilco I and Florunner peanuts were slightly lower than the average for the cold-pressed Spanish peanut oils. The opposite was true for the bag-pressed samples. The average oven keeping times of the bag-pressed oil samples from the Wilco I and Florunner peanuts were longer than the oven stabilities of the bag-pressed oils from the low O/L standard Spanish varieties.

Increased stabilities of the bag-pressed oil samples in comparison to the cold-pressed samples could be due to two phenomena: (A) the presence of an oil extractable antioxidant in the polyethylene bags or (B) contamination of the samples with catalytically active metals during pressing. Although the antioxidant butylated hydroxytoluene (BHT) is sometimes added to polyethylene following the polymerization step, the manufacturer of the stock from which the bags were made (U.S. Industrial Chemicals Co., Deer Park, Tex.) indicated that none was added by the manufacturer, and, in any case, the quantity which could be extracted from BHT containing polyethylene in 30 min using vegetable oil would be essentially nil.

On the other hand, trace amounts of iron, nickel, chromium, and copper salts in vegetable oils accumulated during commercial oil processing operations often act as strong prooxidants (11). Contamination of samples with oxidatively active metals during cold-pressing and subsequent lipid oxidation also has been suggested as the cause of low and erratic oven stabilities. In the present set of experiments, the cold-pressed and bag-pressed oils were prepared ca. 1 month prior to the initiation of the oven tests and were stored at 4 C until utilized, whereas the solvent extracted oils were prepared just prior to testing. The peroxide values and free fatty acid values, respectively, of freshly prepared oils ranged from 0.45-0.65 milliequivalents/kg and 0.10 to 0.15% free fatty acids and are shown in footnoes a and b of Table I.

Correlation of the oven keeping times of the cold-pressed and bag-pressed oil with both sets of peroxide numbers and the free fatty acid content of the freshly pressed oils was poor. However, a relatively high negative correlation with free fatty acid contents of the aged oils was obtained with keeping times ($r = -0.78$ and -0.65). A negative correlation with free fatty acid content might be expected if prooxidants in the form of metal salts (soaps) were responsible for deterioration of the oils and extensive deterioration had occurred. Thus, the high peroxide and free fatty acid values and low keeping times shown for the pressed samples in Table I tend to indicate that sufficient time had elapsed between pressing and testing to allow extensive autoxidation to occur in the pressed samples and to override the normally longer induction period of high O/L oils (2, 10). As the deterioration was apparently greater in the cold-pressed oil and metal catalyzed oxidations were strongly suspected as the cause, bag-pressing was adopted as the procedure for preparing pressed samples in subsequent experiments.

Some interesting relationships also were noted when the O/L ratios recorded in Table I were correlated with oven keeping times. The correlations between oven days and O/L ratios were: (A) cold-pressed, -0.69; (B) bag-pressed, 0.02; (C) chloroform-methanol, 0.91; (D) ether (reagent), 0.49; (E) ether (low peroxide), 0.88; and (F) cyclohexane, 0.89. It should be noted that the negative correlation for the cold-pressed samples is contrary to O/L theory and suggests that the use of O/L ratios to predict the stabilities of oils with relatively high peroxide contents could be quite misleading. Furthermore, we have observed (GLC analysis) that the fatty acid compositions of oils which had been subjected to the oven test were changed considerably. The O/L ratios for the samples which had been: Spanish (Starr and TP 716) 1.2, Wilco I 1.3, and Florunner 1.4 became: Spanish 1.6 and Wilco I and Florunner 2.6. Tai (7) recorded

TABLE III

Effect of Storage Period Prior to Extraction upon Average Oven Stabilities of Oil Samples from Stephenville, Tex., Peanuts

Months	Varieties	Treatment					
		Bag-press	$CHCl_3$-MeOH (3:1) (No skins)	$CHCl_3$-MeOH (3:1) (Skins)	Cyclohexane	Acetone	Ether
6	Spanish[a]	15.0	22.0	21.3	16.3	13.7	17.6
	All[b]	15.7	24.5	27.1	18.7	13.7	18.9
10	Spanish	9.4	21.8	24.1	23.7	13.4	22.1
	All	10.5	23.9	27.2	22.4	12.9	24.5
11	Spanish	9.8	17.0	24.2	16.3	10.8	11.8
	All	11.0	16.5	24.9	15.5	10.4	12.5

[a]Standard Spanish varieties: Starr, Spanhoma, and TP 716-2-1.

[b]All varieties: Starr, Spanhoma, TP 716-2-1, Wilco I, and Florunner.

the same phenomena during his dissertation research on peanut butter.

Our results also indicate that it is advisable to use ether of low peroxide content for solvent extraction of oils. The potential explosion hazard of peroxidized ether is well known, but the use of peroxidized ether apparently may lead to unexpected chemical changes as well.

A third series of extracted oils using five varieties grown in Stephenville, Tex., in 1972 was prepared to evaluate more fully the effects of skins upon the oven keeping times of peanut oils. The bag-pressed, ether (low peroxide), cyclohexane, and acetone extracted samples were prepared after removal of the skins, whereas two chloroform-methanol samples, one with skins and one without skins, were utilized. The results of the oven tests, O/L ratios, and free fatty acid and peroxide values of the samples are listed in Table II.

The oven tests were initiated, and the chemical tests were completed within a week after the sample were prepared. As a result, the peroxide numbers of the bag-pressed oils were low, ranging from 1.35 for Wilco I to 2.4 for Florunner. The free fatty acid percentages (0.02-0.06) were lower, and the oven keeping times of the cold-pressed oils from Stephenville were longer than those of Pearsall, Tex., samples.

The lower peroxide numbers and low free fatty acid numbers suggest that the bag-pressed samples were of better quality than the Pearsall samples (Table I) at the time of testing. The resulting oven stabilities of the hydraulically pressed oils were increased ca. 40% over the Pearsall values (15.7 vs 11.5 days). A positive correlation was found between the free fatty acid content and keeping times ($r = 0.74$), while a negative correlation was found with peroxide numbers ($r = 0.30$). However, the free fatty acid values are so low that they probably are not significant, and increasing free fatty acid content usually is associated with a decreasing quality. Therefore, the high positive correlation is probably a matter of chance.

The average oven keeping times of the oils extracted from the peanuts with skins using chloroform-methanol (3:1) were somewhat longer than the oven keeping times of the oils extracted from the peanuts without skins (24.5 vs 27.1 days), and large differences were recorded between individual varieties in the two tests. The oven keeping times of the ether extracted (low peroxide) and cyclohexane extracted oils were generally similar, while the oven keeping times of the acetone extracted and bag-pressed samples were generally lower than the other treatments.

The overall order of the oven stabilities was chloroform-methanol (with skins)> chloroform-methanol (without skins)≥ether≅cyclohexane>bag-pressed>acetone. The order was similar to the Pearsall samples, except that the acetone-extracted oils were more stable than the bag-pressed oils at Pearsall. The difference is probably due to the poor quality of the Pearsall hydraulically pressed oils, as noted previously. The increase in stability of Florunner and Wilco I oils in comparison to Starr, Spancross, and TP 716-2-1 was not as great as at Pearsall. This might be due to the relatively larger linoleate content of the Stephenville Florunner and Wilco I oils in comparison to the other three varieties grown at Stephenville.

Correlation coefficients were calculated for the relationship between oven days and the O/L ratios of the solvent extracted and pressed oils. The correlations were: (A) bag-pressed, 0.55; (B) chloroform-methanol (without skins), 0.83; (C) chloroform-methanol (with skins), 0.90; (D) ether (low peroxide), 0.89; (E) cyclohexane, 0.86; and (F) acetone, 0.62. These results, obtained after the nuts had been in cold storage for ca. 6 months, indicate that chloroform-methanol, ether, and cyclohexane are ca. equally suitable for extracting peanut oil and testing oven stabilities. Extraction with acetone and hydraulic pressing definitely lead to different results.

Increasing period in cold storage prior to extraction also can influence the oven stabilities and correlation coefficients obtained with solvent extracted and bag-pressed oils. The average oven stability values obtained for the Stephenville grown peanuts after 6, 10, and 11 months in cold storage are shown in Table III. The averages for the three Spanish varieties and the overall averages for the five varieties are shown for each treatment.

Comparison of the averages for the Spanish varieties and for the five varieties indicates that prolonged storage of the peanuts tended to shorten the oven keeping times and to make the differences in oven keeping times between the standard Spanish varieties and the large seeded Wilco I and Florunner varieties more erratic. These effects were much less noticeable in the samples with skins which had been extracted with chloroform-methanol. When the individual values were correlated with the respective O/L ratios, a similar trend was recorded. The correlations for all treatments became poorer with increasing storage period, but the results using the oils extracted from peanuts with skins with chloroform-methanol were affected to a lesser degree ($r = 0.52$ after 11 months).

The differences in keeping times of oil extracted with different solvents from blanched and nonblanched peanuts may be due to several factors. First, solvents may differ in their capacity to extract some of the minor constituents of the cotyledons and skins. The possibility that some peanut varieties differ in their content of solvent extractable nontocopherol antioxidants was suggested by Fore, et al., (5) nearly 20 years ago. Second, solvents may differ in their capacity to extract certain lipid components in peanuts. Although the small differences in O/L ratios, which generally were recorded, do not appear to be large enough to indicate a significant difference in the gross composition of the oils, they may indicate that the solvents differ in their capacity to extract one or more of the minor lipids, e.g. phosphatides or glycolipids. Glycolipids and phospha-

tides have been reported to convey pro-, as well as anti-, oxidant effects, depending upon the system and its pretreatment (12). Chloroform-methanol is an excellent general solvent for lipids, whereas acetone and ether are not (13). Thus, the differences in stability may reflect differences in the extent to which minor components are extracted.

The differences in stability might also be due to solvent related effects. Many commercial solvents, including ether, acetone, and methanol, contain traces of high mol wt fluorescent compounds which are presumably phenolic in nature (14). These high boiling compounds, which remain in the oil after the solvent has been evaporated, possibly could act as antioxidants and prolong the keeping times of the oils. The longer keeping times of chloroform-methanol extracted oils also could conceivably be due to residual amounts of chloroform or methanol in the oils. Methanol might serve as a hydrogen donor or inhibitor of free radical oxidation in the oil system. Alcohol (ethanol) is added to reagent grade chloroform for this purpose (15). It is also possible that chloroform itself might stabilize the lipid system by reacting with free radical initiators or by acting as a chain terminator (16). None of these suggestions, however, explain the relatively low stability of the acetone extracted oils.

Taken as a whole, our results tend to indicate that the presence of skins during solvent extraction of the oils increases the oven keeping times of the ensuing oils. The effects of skins appear to be more significant as the period in storage increases. Our experiments also indicate that the length of the oven keeping times can be affected by the method of solvent extraction. If the peanuts are relatively fresh, good correlations between O/L ratios and keeping times can be obtained using ether of low peroxide content, cyclohexane, or chloroform-methanol, but the values for one set of solvent extracted oils are not directly comparable to those for oils extracted with a different solvent system. The oven keeping times and correlations with acetone extracted oils were always lower than the other solvent extracted oils. Chloroform-methanol gave extended stabilities, and correlations remained higher after long periods in storage. Correlations between oven keeping times and free fatty acid values and peroxide numbers using freshly prepared samples were poor. Thus, predictions of oven stability from O/L data would appear to be confounded by the method of oil extraction, as well as the location of growth (1) and year variations (10).

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