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Smoke, Fire, and Flash Points of Cottonseed, Peanut, and Other Vegetable Oils*

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Introduction

The smoke, fire, and flash points of a fat or oil are measures of its thermal stability. Their relation to thermal stability may be stated as follows: (a) The *smoke point* is that temperature at which appreciable decomposition begins, when a body of the oil is heated in contact with the atmosphere. It is the temperature at which volatile decomposition products are evolved in sufficient quantity for them to become visible. (b) The *flash point* is that temperature at which the decomposition products are evolved in such quantity as to be capable of ignition but not of continued combustion. (c) The *fire point* is that temperature at which decomposition products are evolved in such quantity and at such a rate that they will support continued combustion.

In any operation involving the heating of an oil to a high temperature, the thermal stability of the oil is a matter of manifest importance. The degree to which an oil or fat may be heated without undergoing undue breakdown or evolution of volatile substances determines not only the temperature range within which it may be effectively used, but also the fire hazard attendant upon its use. Among the commercial operations which require the use of fats or oils and which are conducted at temperatures within the range of their thermal instability, are deep fat frying of food, the manufacture of "bodied" oils, varnishes and similar products, and the manufacture of tin plate, and of tin and terne-plated articles.

Since both cottonseed and peanut oils have long been prized for their edible qualities, their industrial use has for many years been insignificant. Consequently, little interest was attached to their high temperature behavior except under the conditions of food frying. Recent dislocations in the world supply of fats and oils, however, have given a new interest to the possible technical uses of these and other domestic oils. It does not seem likely that oils of foreign origin will be available in this country in any quantity for an extended period of time. As a consequence, the more expensive, domestically produced edible oils, such as cottonseed and peanut, may be expected to find increasing use in the future in technical or non-edible fields.

From time to time, proponents of various cooking oils have made claims of superiority of one oil over another with regard to its smoke point. Olive oil has long been widely used for cooking purposes and one of the chief claims made for it was its high smoke point. Later, cottonseed oil came into general use for deep fat frying and again one of its chief advantages was said to be its high smoke point. More recently, peanut oil has appeared on the market in appreciable volume and again a high smoke point has been advanced as one of its principal virtues.

It has, however, been generally known that the smoke point of any fat or oil is dependent on the degree of refinement of the product, and especially on its freedom from fatty acids (1). In fact, it has been considered that the initial free fatty acid content of a fat or oil, and the rate at which additional free fatty acids are formed under conditions of use, are the determining factors affecting the smoking temperature of a fat or oil.

A survey of the literature revealed little pertinent information on the smoke, flash, and fire points of peanut oil, and relatively few references to the smoke, flash, and fire points of cottonseed oil. Blunt and Feeney (1) mention two smoke points for peanut oil, namely, 325° F. and 300° F., but they make no mention of the flash or fire points of this oil. Dickhart (3) reported the determination of the smoke point (464° F.), flash point (632° F.), and fire point (692° F.) on a single sample of peanut oil.

No data on the smoke, flash, and fire points of peanut oil are reported in the monographs by Jamieson (6) and Lewkowitsch (8), or in the International Critical Tables (5). Detwiler and Markley (2) did not include peanut oil in the series of oils which they examined with respect to the smoke, flash, and fire points.

The data on cottonseed oil are somewhat more extensive. The International Critical Tables record the flash and fire points of cottonseed oil as 582° F. and 644° F., respectively. Jamieson (6) and Lewkowitsch (8) report a range of temperatures for these properties. Vollertsen (12) also reported data on the smoke points of various oils, including cottonseed. None of these authors reported the determination of the free fatty acid content of the same sample of oil on which the smoke, flash, and fire points were determined.

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Both Blunt and Feeney (1) and Lowenstein and Vollertsen (9) have investigated the relationship between the free fatty acid content of an oil and its smoke, flash, and fire points. The first mentioned authors found that the smoke point of cottonseed oil was depressed by the addition of various amounts up to 2 per cent of the mixed free fatty acids derived from olive oil. The effect of the addition of free fatty acids above 2 per cent was not investigated. Lowenstein and Vollertsen found that the flash and fire points of various animal fats varied inversely with their free fatty acid content over the range 0 to 100 per cent of free fatty acids.

Several investigators have reported work on the effect of the time and temperature of heating various cooking fats on such characteristics as the smoke point, acidity, iodine number, and acetyl value. Most of these investigations, exemplified by the work of Blunt and Feeney (1), Morgan and Cozens (10), Porter, Michaelis, and Shay (11), and Lantz and Carlin (7) have been concerned with the deterioration of cooking fats under actual conditions of deep fat frying. However, the investigation of the effect of dry heat alone on cooking fats has been somewhat neglected. Fulmer and Manchester (4) investigated the changes in acidity, iodine numbers, and other properties on heating cottonseed oil, but the time of heating was only thirty minutes or less, whereas the temperatures were relatively high, 180 to 270° C.

Standardized conditions and official methods were not always applied in the various determinations reported in the literature and in most cases the degree of refinement of the oil or fat used was unknown or

was not mentioned in the published report, consequently, it is difficult to evaluate or intercompare the few data which are available.

Because of the unsatisfactory state of the published information on smoke, flash, and fire points of cottonseed and peanut oils, a number of commercial and experimentally prepared samples of these oils were examined with respect to their thermal stability properties for the purpose of determining what effect certain processing treatments and the presence of free fatty acids had on these properties.

Experimental

In all cases the smoke, flash, and fire points were determined according to the official method of the American Oil Chemists' Society, using a gas-heated, Cleveland open cup. Duplicate determinations were made in all except two instances where the quantity of oil available was insufficient.

Free fatty acid determinations were carried out in accordance with the official American Oil Chemists' Society method except in the case of the dark colored oils produced by prolonged heating. The method used for dark colored oils will be discussed later.

Effect of free fatty acids: Mixed fatty acids derived from refined cottonseed or peanut oils were added to a series of samples of the respective oils in varying amounts to give a series of fatty acid-oil ratios ranging from 0 to 100 per cent. The smoke, flash, and fire points were determined on each sample of refined oil to which the free fatty acids had been added. The effect of the addition of the mixed free fatty acids on the smoke, flash, and fire points of the

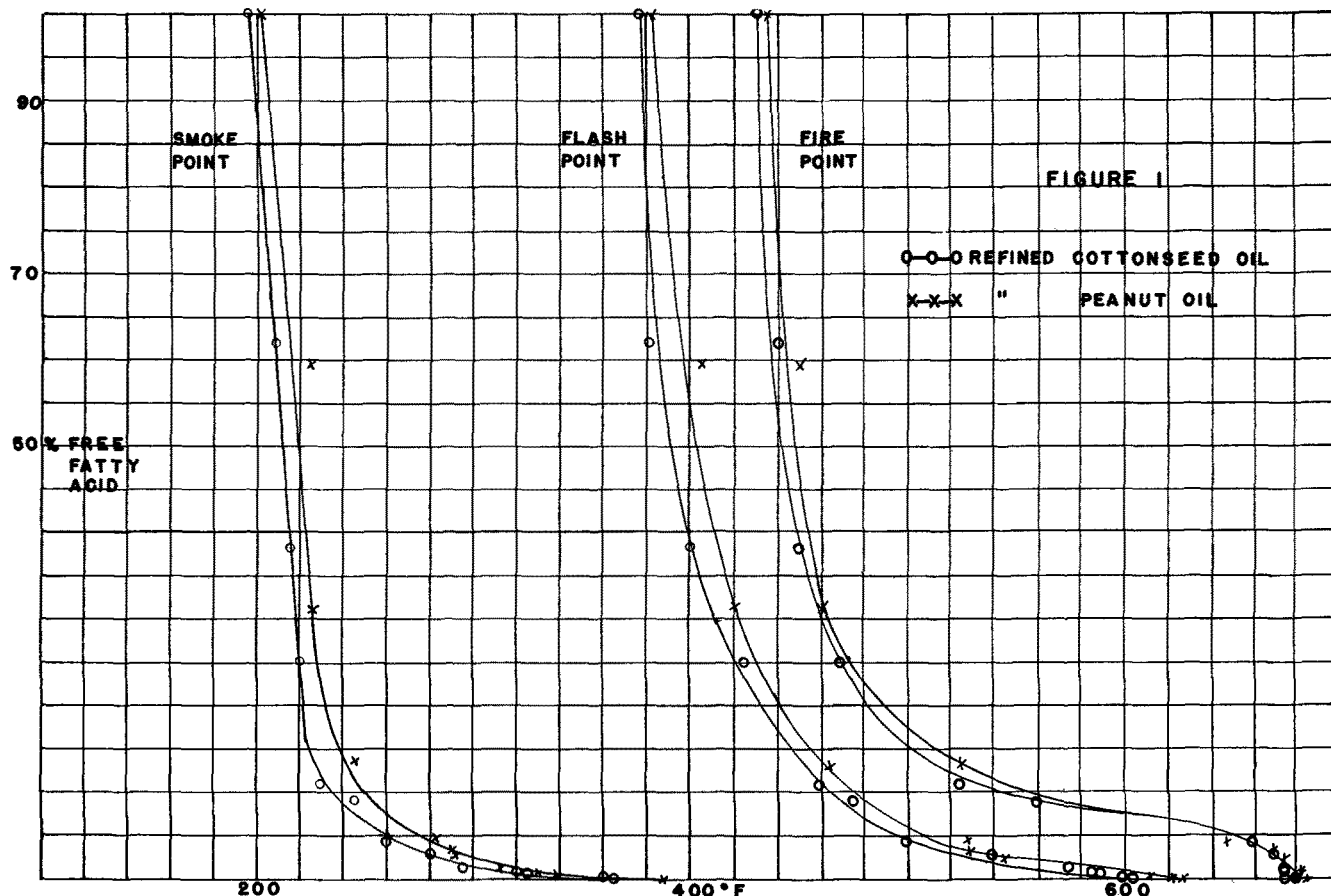


FIG. 1. Relationship between Free Fatty Acid Content and Smoke, Flash, and Fire Points of Cottonseed and Peanut Oils.

oils is shown in graphic form in Figures 1 and 1a. Figure 1a is the lower portion of the smoke point curve in Figure 1 but reproduced on a scale sufficiently large to permit easy determination of differences in smoke point corresponding to small differences in the free fatty acid content of the oil.

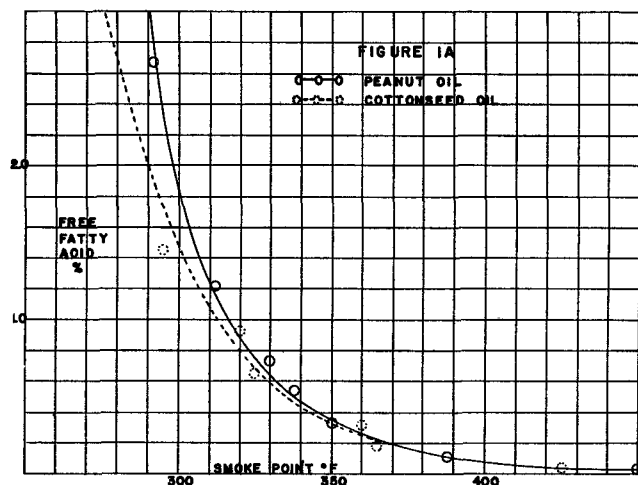


FIG. 1a. Relationship between Free Fatty Acid Content and Smoke Points of Cottonseed and Peanut Oils.

It is apparent from the curves in Figure 1 that cottonseed and peanut oils do not differ greatly in their respective smoke points, flash points, or fire points when both oils have the same free fatty acid content. In fact, these differences are close to the allowable limit of error of the method of determination. However, the constancy of the trend would indicate that the differences are significant above about one per cent of free fatty acids. Peanut oils containing more than about one per cent of free fatty acids generally exhibit a slightly higher smoke, flash, and fire point than cottonseed oils of corresponding free fatty acid content. Below one per cent free fatty acid content, the curves of the two oils virtually coincide.

Effect of processing: In order to evaluate the effect of the degree of processing on the smoke, flash, and fire points of cottonseed and peanut oils, a number of samples representing various stages of commercial and laboratory processing were examined.

Oils numbered 1, 11, 14, and 16 in Table 1 were commercial products; the others were laboratory sam-

ples. The observed smoke, flash, and fire points of the peanut oil samples are recorded in Table 1.

Inspection of the data in this table indicates that in general the smoke points of alkali-refined peanut oils can be determined within the allowable limits of the American Oil Chemists' Society's official method, from the curve in Figure 1, if the corresponding values of the free fatty acid content of the oils are known. However, in the case of oils which have not been alkali-refined, the experimentally determined values may depart appreciably from those represented by the curve for free fatty acid content vs. smoke point.

The smoke point (408° F.) for the deodorized (steam stripped) crude peanut oil (Table 1, No. 3) which had a low free fatty acid value (0.11 per cent) is higher than would be predicted from the curve, although the smoke point (312° F.) of the original crude hydraulic-pressed oil is somewhat lower than would be predicted. After bleaching, which did not affect the free fatty acid content, the smoke point was observed to increase appreciably, namely, to 418° F.

A crude solvent-extracted oil (Table 1, No. 6) had a smoke point (293° F.) in good agreement with that predicted from the curve. After a moderate steam refining (Table 1, Oil No. 7), the smoke point increased to 333° F., which also corresponds to the value predicted from the curve. However, on subsequent bleaching (Table 1, Oil No. 8), the smoke point increased further to 348° F., although the free fatty acid content remained unchanged. Evidently, the bleaching removed some substance (or substances) which tends to depress the smoke point below the value corresponding to that expected from its free fatty acid content. This substance (or substances) can also be removed by alkali-refining and can partially be removed by steam stripping.

Since the more prolonged and more complete steam-refining process yielded an oil (Table 1, Oil No. 3) having a smoke point-free fatty acid relationship superior to that predicted from the curve, it is probable that this substance is more selectively removed by steam-refining than by alkali-refining, since the alkali-refined oils were generally found to conform to the behavior predicted from the curve.

The data relative to the smoke, flash, and fire points for the various samples of cottonseed and other oils are recorded in Table 2. The data for the oils other than cottonseed were determined in the course of an

TABLE 1
Free fatty acids, smoke, flash, and fire points of various peanut oils

| Oil No. | Type of Oil | Free fatty acid | Smoke point (Predicted) | Smoke point (Found) | Flash point | Fire point |
|---------|---|-----------------|-------------------------|---------------------|-------------|------------|
| | | per cent | F° | F° | F° | F° |
| 1 | Crude, hydraulic-pressed, (0-5)..... | 0.84 | 322 | 312 | 580 | 670 |
| 2 | Oil No. 1, bleached..... | 0.84 | 322 | 323 | 598 | 680 |
| 3 | Oil No. 1, deodorized, steam-refined..... | 0.11 | 388 | 408 | 613 | 678 |
| 4 | Oil No. 1, deodorized and bleached..... | 0.11 | 388 | 418 | 620 | 680 |
| 5 | Oil No. 1, alkali-refined and bleached..... | | | 445 | 625 | 680 |
| 6 | Crude, solvent-extracted, laboratory..... | 2.3 | 295 | 293 | 550 | 678 |
| 7 | Oil No. 6, deodorized, steam-refined..... | 0.51 | 337 | 333 | 610 | 680 |
| 8 | Oil No. 6, deodorized and bleached..... | 0.51 | 337 | 348 | 610 | 680 |
| 9 | Oil No. 6, alkali-refined..... | 0.030 | 430 | 450 | 620 | 680 |
| 10 | Oil No. 6, alkali-refined and bleached..... | 0.030 | 430 | 448 | 623 | 680 |
| 11 | Alkali-refined, bleached and deodorized (0-50)..... | 0.11 | 388 | 388 | 628 | 685 |
| 12 | Oil No. 11, hydrogenated to I. No. = 55.3..... | 0.12 | 385 | 380 | 620 | 680 |
| 13 | Oil No. 11, hydrogenated to I. No. = 1.6..... | 0.15 | 378 | 370 | 593 | 643 |
| 14 | Crude, hydraulic-pressed, (0-38)..... | 0.66 | 330 | 343 | 618 | 685 |
| 15 | Crude, hydraulic-pressed, water washed..... | 0.71 | 328 | 330 | 600 | 680 |
| 16 | Alkali-refined, bleached and deodorized..... | 0.105 | 388 | 413 | 615 | 685 |

TABLE 2
Free fatty acids, smoke, flash, and fire points of cottonseed and other vegetable oils

| Oil No. | Oil | Type of Oil | Free fatty acid | Smoke point (Predicted) | Smoke point (Found) | Flash point | Fire point |
|---------|-------------------|--|-----------------|-------------------------|---------------------|-------------|------------|
| | | | <i>per cent</i> | <i>F°</i> | <i>F°</i> | <i>F°</i> | <i>F°</i> |
| 1 | Cottonseed (0-47) | Crude, hydraulic-pressed, AOCS #1 | 0.7 | 325 | 338 | 603 | 680 |
| 2 | Cottonseed (0-58) | Crude, hydraulic-pressed, AOCS #3 | 1.1 | 310 | 310 | 585 | 680 |
| 3 | Cottonseed (0-52) | Crude, hydraulic-pressed, AOCS #2 | 1.8 | 293 | 293 | 560 | 673 |
| 4 | Cottonseed | Alkali-refined | 0.040 | 425 | 425 | 617 | 685 |
| 5 | Cottonseed (0-19) | Alkali-refined, bleached | 0.055 | 415 | 425 | 613 | 683 |
| 6 | Cottonseed (0-14) | Alkali-refined, bleached, deodorized | 0.040 | 425 | 428 | 613 | 680 |
| 7 | Cottonseed (0-49) | Alkali-refined, bleached, deodorized | 0.18 | 370 | 365 | 605 | 675 |
| 8 | Cottonseed | Oil No. 7, hydrogenated to I. No. = 1.6 | 0.25 | 360 | 360 | 595 | 650 |
| 9 | Cottonseed | Oil No. 7, hydrogenated to I. No. = 48.5 | 0.29 | 355 | 350 | 600 | 675 |
| 10 | Corn (0-51) | Refined and bleached | 0.065 | 405 | 400 | 618 | 678 |
| 11 | Soybean (0-15) | Refined and deodorized | 0.010 > | 450 | 453 | 623 | 685 |
| 12 | Soybean (0-17) | Refined and deodorized | 0.01 > | 450 | 443 | 625 | 685 |
| 13 | Soybean (0-16) | Crude, expeller-pressed | 0.51 | 335 | 365 | 565 | 660 |
| 14 | Safflower (0-45) | Crude, hydraulic-pressed | 1.7 | 295 | 318 | 603 | 683 |
| 15 | Pecan (0-92) | Water-washed, settled | 0.55 | 332 | 335 | 600 | 680 |
| 16 | Olive (0-112) | Edible grade | 2.1 | 290 | 280 | 550 | 670 |

investigation on the development of a domestic substitute to replace imported olive oil for use in the textile industry. Data on oils examined in the course of work on the development of a substitute for palm oil will appear in a later publication from this laboratory. Although the relationship between free fatty acid content and smoke point of Figure 1 would not be expected to apply for oils of such diverse types and treatments as are indicated in Table 2, nevertheless, the smoke points predicted from the curves in Figure 1 are in surprisingly good agreement with a majority of the experimentally determined values, as is evident from columns 5 and 6 of Table 2.

Effect of prolonged heating: In order to determine the effect of prolonged continuous heating on cottonseed and peanut oils, several of these oils were heated at a temperature of 350° F. for approximately two weeks during which time samples were withdrawn for analysis. Beakers of 2,000 ml. capacity were used to contain the oils. Heat was independently supplied at the bottom and over the entire side walls by means of resistance wire. The temperature was kept constant by means of a bimetallic thermostat immersed in the sample of oil under test.

The original oils were commercially refined, deodorized, and bleached. The hardened oils corresponding to the original oils were hydrogenated in the laboratory. The iodine value and other properties of these oils are recorded in Table 3.

TABLE 3
Properties of the peanut and cottonseed oils subjected to prolonged heating at 350° F.

| Oil | Iodine number | Free fatty acid | Smoke point | Flash point | Fire point |
|-----------------|---------------|-----------------|-------------|-------------|------------|
| | | <i>per cent</i> | <i>F°</i> | <i>F°</i> | <i>F°</i> |
| Peanut..... | 97.7 | 0.11 | 388 | 628 | 685 |
| Peanut..... | 55.3 | 0.12 | 380 | 620 | 680 |
| Peanut..... | 1.0 | 0.15 | 370 | 592 | 642 |
| Cottonseed..... | 98.2 | 0.18 | 365 | 605 | 675 |
| Cottonseed..... | 48.5 | 0.29 | 350 | 600 | 675 |
| Cottonseed..... | 1.6 | 0.25 | 360 | 595 | 650 |

Samples of 125 ml. were withdrawn periodically; usually a total of five samples were removed during an interval of two weeks. These samples were examined with respect to their smoke, flash, and fire points, as well as percentage of free fatty acids and iodine numbers.

Some difficulty was encountered in determining the free fatty acid content of the heated, dark-colored

oils owing to the masking of the phenolphthalein endpoint in these products. The use of the spot plate with phenolphthalein as an external indicator did not help materially. The procedure finally adopted was as follows:

Seventy-five ml. of neutral ethanol (95 per cent) was added to 7.05 grams of oil and the mixture was heated to boiling. Phenolphthalein solution was added and the mixture was titrated with 0.1 N aqueous sodium hydroxide solution to an approximate endpoint as indicated by the first appearance of a pink color in the supernatant alcohol layer. The mixture was vigorously shaken and the titration was then completed using a spot plate and thymol blue solution as an external indicator.

The same value for the free fatty acid content was obtained in duplicate analyses of unheated oils, or of heated oils containing little color, irrespective of the indicator used.

In order to check the phenolphthalein-thymol blue titration method, a 360-gram sample of a very dark peanut oil which had been heated for 14 days was subjected to steam deodorization to remove the free fatty acids. Titration of the heated, dark-colored oil, using phenolphthalein-thymol blue indicator, indicated the presence of 4.58 grams of fatty acids calculated as oleic acid. After steam deodorization, titration by the same method indicated the presence of 0.72 grams of fatty acids in the sample, or a loss of 3.86 grams of these acids. The recovered acids actually weighed 3.9 grams and had a neutralization equivalent of 308. Ordinary steam distillation of the fatty acid fraction obtained by deodorization gave 0.3 grams of distillate which consisted of myristic and palmitic acids. The residual undistilled portion of the fatty acids were found to consist of approximately 35 per cent of a mixture of palmitic, stearic, and arachidic acids and 65 per cent of a mixture of unsaturated acids.

The relationship between the smoke point and the free fatty acids developed on heating the peanut oils is reproduced graphically in Figure 2. A portion of the smoke point curve from Figure 1a representing peanut oil with added free fatty acids is included for comparison.

The smoke point of the highly hydrogenated oil (I. No. = 1.0) decreased with increasing acidity due to heating just as the smoke point increased when free fatty acids were added to the unhydrogenated oil,

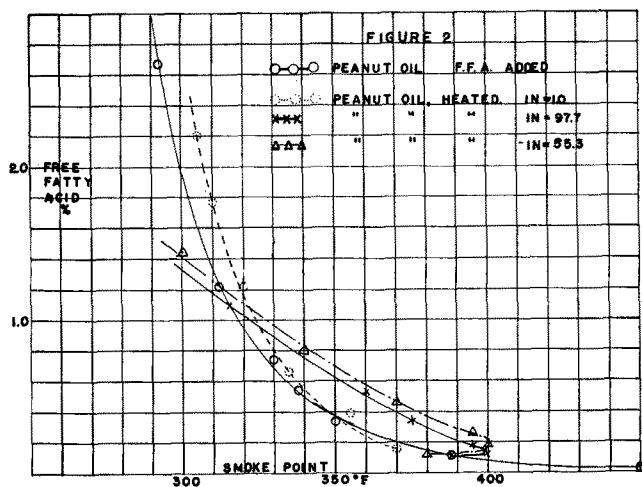


FIG. 2. Smoke Point vs. Free Fatty Acid Developed by Heating Peanut Oils of Various Iodine Numbers.

indicating that the decrease in smoke point was probably due to the presence of the same material, namely, free fatty acid. However, the curves representing the smoke points vs. free fatty acids depart appreciably from the reference curve in the case of the partly hydrogenated oil (I. No. = 55.3) and also in the case of the original oil (I. No. = 97.7). In these cases, as compared to the reference curve, the smoke points first increased slightly and then decreased more rapidly with increasing free fatty acid content of the oil.

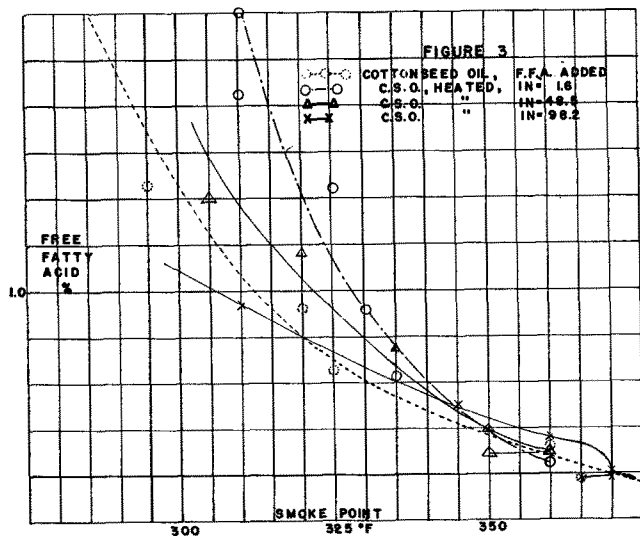


FIG. 3. Smoke Point vs. Free Fatty Acid Developed by Heating Cottonseed Oils of Various Iodine Numbers.

Cottonseed oil (Figure 3) exhibited a similar behavior. The curve of the highly hydrogenated oil (I. No. = 1.6) is similar to the reference curve, whereas the curves of the partly hydrogenated (I. No. = 48.5) and original (I. No. = 98.2) oils show at first a slight increase of smoke point, followed by a more accelerated rate of decrease.

The relationship between the time of heating and (1) the free fatty acid content and (2) the smoke point of peanut oil, is illustrated in Figure 4. The

smoke points of the partly hydrogenated and the original oils are again observed to increase in the early stages of heating in contrast to that of the highly hydrogenated oil which decreases continuously as the length of heating time increases. The shapes of the curves for free fatty acid vs. time for

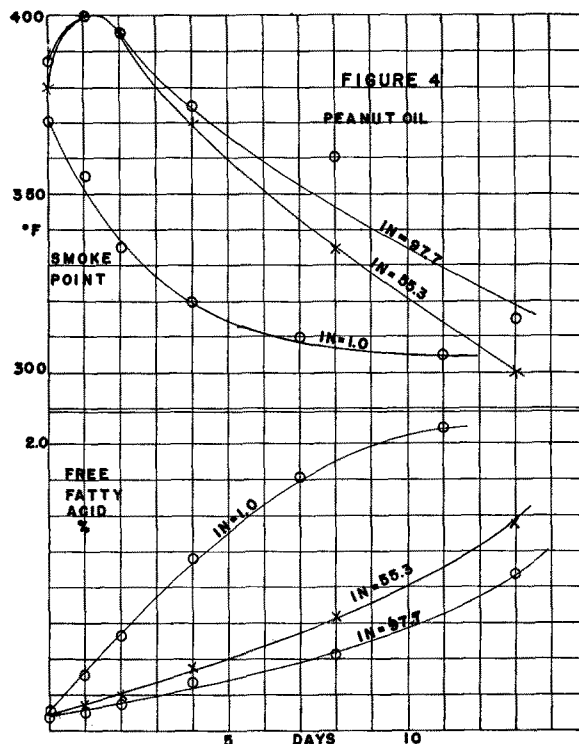


FIG. 4. Relationship between Time of Heating and (1) Free Fatty Acid Content and (2) Smoke Point of Peanut Oils of Various Iodine Numbers.

these oils also show distinct differences, the curve of the highly hydrogenated oil is convex to the time axis, whereas the other curves are concave.

The corresponding data for the cottonseed oils are illustrated graphically in Figure 5. The relationships between the smoke point and time of heating of the cottonseed oils are very similar to those observed with the peanut oils. The smoke points of the partly hydrogenated and original oils are observed as before in the early stages of heating in contrast to that of the highly hydrogenated oil which decreases continuously with increased time of heating. However, the curves representing the free fatty acid as a function of time exhibit a distinct difference. The curve of the highly hydrogenated oil (I. No. = 1.6) remains concave, whereas that of the unhydrogenated oil (I. No. = 98.2) is convex to the time axis. The partly hydrogenated oil, (I. No. = 48.5) first follows the curve of the unhydrogenated oil, being convex, then passes a point of inflection and follows the curve of the highly hydrogenated oil, being concave to the time axis.

The relationships between the iodine number and time of heating of the refined cottonseed and peanut oils are shown graphically in Figure 6. The curve for the change in iodine number with time of heating of the refined peanut oil is similar to the corresponding curve for cottonseed oil, except that in the later stages of heating the peanut oil exhibits an increasing

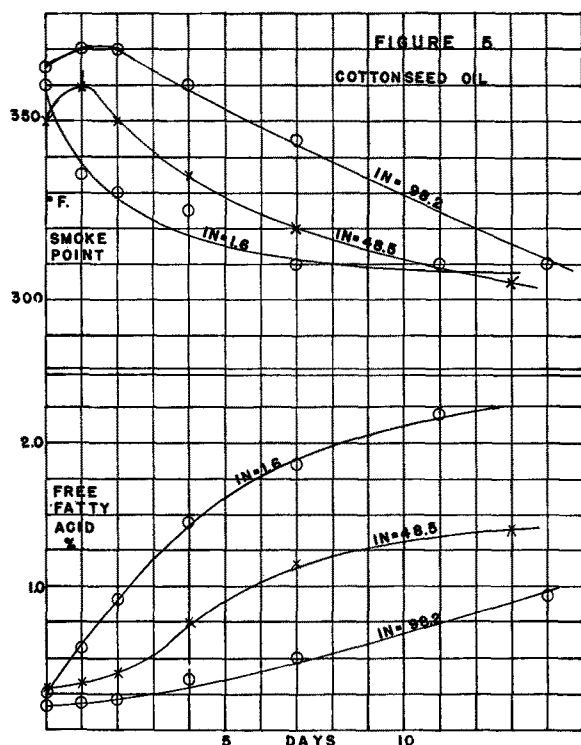


Fig. 5. Time of Heating vs. (1) Free Fatty Acid Content and (2) Smoke Point of Cottonseed Oils of Various Iodine Numbers.

stability in comparison with cottonseed oil, as measured by the iodine number of these oils.

In the case of the partially hydrogenated cottonseed and peanut oils, the curves for the iodine number vs. time of heating are quite similar and parallel throughout the entire period of heating.

Summary and Discussion

The smoke, flash, and fire points of a variety of cottonseed and peanut oils have been determined together with the effect which the free fatty acids, processing treatments, and prolonged heating at 350° F. have on these properties.

The smoke points of good quality cottonseed and peanut oils, processed under similar conditions, differ but little. The smoke point of peanut oil is only slightly, but probably significantly, higher than that of cottonseed oil of equivalent quality.

The presence of free fatty acids in either cottonseed or peanut oils influences the smoking temperature and appears to be the principal, but not the only factor, which tends to lower the smoke point. This conclusion is based on the observations that: (1) refined cottonseed and peanut oils of given free fatty acid content and smoke point exhibit an increase in smoke point after bleaching which operation does not affect the content of free fatty acids; (2) cottonseed and peanut oils subjected to prolonged heating exhibit an initial increase followed by a decrease in smoke point with increasing fatty acid content; and (3) the smoke point of cottonseed and peanut oils decreases at a more rapid rate on prolonged heating than can be accounted for by the rate of increase in free fatty acid content.

Since the highly hydrogenated oils do not exhibit the initial increase in smoke point on prolonged

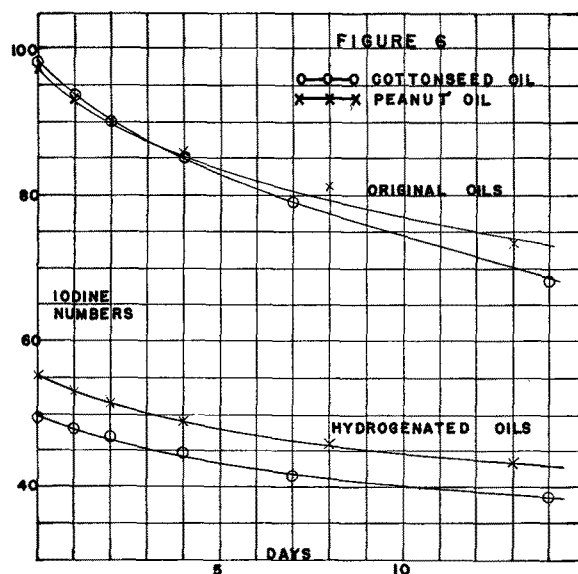


Fig. 6. Iodine Numbers vs. Time of Heating of Cottonseed and Peanut Oils.

heating which is observed in the case with the original and slightly hydrogenated oils, it would appear that the substance (or substances) other than free fatty acids, which is partially responsible for lowering the smoke point is more or less unsaturated. The type of unsaturated bodies present in peanut and cottonseed oils which may account for this phenomenon is exemplified by the carotenoid pigments.

In order to account for the initial slight increase in smoke point on prolonged heating, it must be assumed that the unsaturated substance (or substances) which is partially responsible for lowering the smoke point is polymerized, decomposed, or evolved during heating.

Acknowledgment

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