combination with the 92% methanol (synergist) fraction. It is probable that the primary antioxidant activity of tomato lipids is caused chiefly by one or more of the tocopherols.

To elucidate further the nature of the "synergist" present in tomato lipids, several acidic compounds were tested (Table IV). Of these, phosphoric acid was the only one which with alpha-tocopherol exhibited synergistic action of the same order of magnitude as the 92% methanol fraction of tomato lipids. The exact effective concentration of the phosphoric acid in the lard sample is not known because of its low solubility.

TABLE IV

A Comparison of the Synergistic Action of Phosphoric, Citric, Ascorbic, and Succinic Acids with *alpha*-Tocopherol in Lard (100°C.)

Material tested	Keeping time (hrs.)	Protective factor
Lard	1.0	1.0
Lard + 0.073% α-toc.	4.0	4.0
Lard + 0.073% α-toc. + 0.05% H_3PO_4	58.0	58.0
Lard + 0.073% α-toc. + 0.05% citric acid	26.0	26.0
Lard + 0.073% α-toc. + 0.05% ascorbic acid	26.0	26.0
Lard + 0.073% α-toc. + 0.05% succinic acid	9.0	9.0

The phosphorus content of the 92% methanol fraction was determined colorimetrically by the A.O.A.C. method (11) after digestion with concentrated sulfuric and nitric acids. The results of this analysis indicated that the 92% methanol fraction contained 2.4% phosphorus. This means that the 92% methanol fraction added to the lard in the tests in Tables II and III was equivalent to adding 0.00132 g. of phosphoric acid. The solubility of the tomato synergist fraction in both petroleum ether and lard indicates that the phosphorus is not present as free phosphoric acid but rather that it exists as an organic phosphorus compound. The peroxide-reducing properties of tomato lipids is similar to that reported for phosphoric acid and wheat germ phosphatides (8, 9) and may be caused by this phosphorus-containing fraction.

Summary

Highly active antioxidants for lard were extracted from dried tomato fruits with petroleum ether. When added to fresh lard, the tomato lipids protected against autoxidation at 100°C. for long periods with little accumulation of peroxides during the induction period. When added at a 2% level to actively oxidizing lard (peroxide values 20 to 130), the tomato lipids effected a rapid drop of approximately 25% in titrable peroxides. When added at a level of 8%, this immediate drop was followed by a second, more gradual, drop to a constant low value (*ca.* 5), which was maintained for a long period at 100°C.

The antioxidant activity of the tomato lipids was separated into two fractions, one consisting of a primary antioxidant and the other of a synergistic (potentiating) substance. The primary antioxidant activity was accountable in the tocopherol content of the extracts. The synergistic activity was qualitatively similar to phosphoric acid, and the synergistic fraction was found to contain 2.4% phosphorus. However solubility data indicated the phosphorus was present in an organic form, probably phosphatides.

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Recent Developments in Screw-Press Operations

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THIS PAPER is primarily a brief summary of developments related to cooking and continuous mechanical screw presses which have in recent years improved the efficiencies which can be expected from this method of processing. It is helpful and interesting in discussing these developments to trace the history of mechanical processing techniques since some of the basic fundamentals which were learned several decades ago are responsible for several of the more recent developments.

When pressing of cottonseed was first attempted, no cooking was done; then gradually it was discovered that a moderate cooking or tempering resulted in a higher degree of extraction. From this was evolved the idea of using a steam-jacketed conveyor to warm the material en route to the pressing machinery. Other types of horizontal cookers with paddles having steam-jacketed outer walls were also developed and were found to provide definite improvements over methods which used no cooking at all. All of these methods however gave way and were superseded by the individual batch-type of cooking kettle, which at the turn of the century was the most widely used type of cooking apparatus. The advantage of this type of cooking was that it provided absolute control over temperature, moisture, and length of time during which the material was processed. From this type of cooking arrangement evolved the stack cooker which super-imposed one kettle on another, providing greater capacity and greater control. This type of cooking arrangement was readily adapted to the continuous process by using automatic floating-type gates, which maintained the desired level of meats in each kettle continuously and thereby provided the exact control over the cooking process which is required for an efficient operation.

It is interesting to observe that when continuous mechanical presses were first introduced, processors labored through the same long cycle which they had experienced with hydraulic presses some years before with regard to cooking equipment. As with hydraulic presses, the first continuous mechanical presses were installed with no cookers, then with steam-jacketed conveyors and sometimes with a combination of steam tube-dryers or horizontal cookers and steam-jacketed conveyors; but again all of these methods left much to be desired in providing the necessary control of the cooking process, and the stack cooker has again become recognized as the best possible cooking arrangement for this type of continuous process.

It was soon discovered that considerably more cooking capacity was required to prepare a material properly for screw presses than was necessary for hydraulic presses because of the additional drying which must be accomplished prior to pressing in a continuous screw press. The early installations of screw presses used a 72-in. diameter cooker, having four kettles mounted on each screw press to handle 20 to 25 tons of cottonseed. As the capacity of the screw presses increased to nearly twice this amount, the need for more and more cooking capacity was evident. To increase cooking capacity additional kettles were added to the cookers, making them 5 or 6 high and eventually larger diameter cookers of 85-in.- and recently 100-in.-diameter were introduced to eliminate the need for stacking kettles 8, 9, or 10 high.

To provide sufficient cooking equipment in converting a mill from hydraulic presses to screw presses, space is often a problem. If an unusually high cooker were required, extensive modifications of existing buildings might be required. Using 85-in.- or 100-in.-diameter cookers, capacity per kettle is increased, avoiding the need to stack the cookers so high. This has proven to be very helpful in providing efficient, compact installations at a minimum installation cost.

The use of an 85-in.-diameter cooker mounted over a single press, as shown in Figure 1, provides a flexible and compact arrangement of the press room and at the same time provides maxium ease of operation. To simplify an installation of several presses, it has often become desirable to install two or more presses under a single cooker, as shown in Figure 2, where a 100-in.-diameter cooker with four kettles is mounted over and between two presses. Such a unit has a rated capacity of up to 80 tons of cottonseed per 24 hours and provides a compact, easily controlled and flexible arrangement which can be readily adapted to most existing oil mills. This type of arrangement with the cookers mounted directly over the presses reduces installation cost by eliminating the need for elaborate cooker supports and cutting down the amount of conveying equipment required by feeding the cooked material directly to the presses. The arrangement also allows the cooked material to be pressed immediately as it comes from the cooker, thereby preventing chilling in conveying from cooker to press.

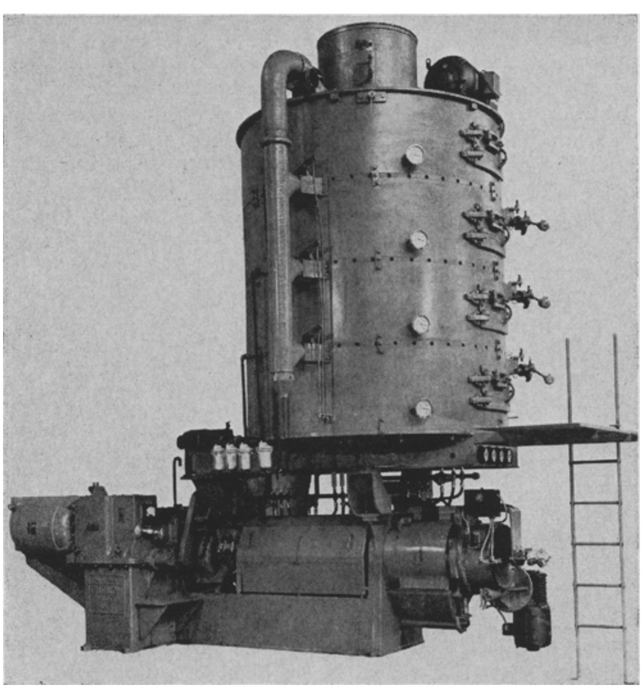

FIG. 1. Four high cooker dryer and four-section mechanical screw press with two extension cages and easy-view discharge cone.

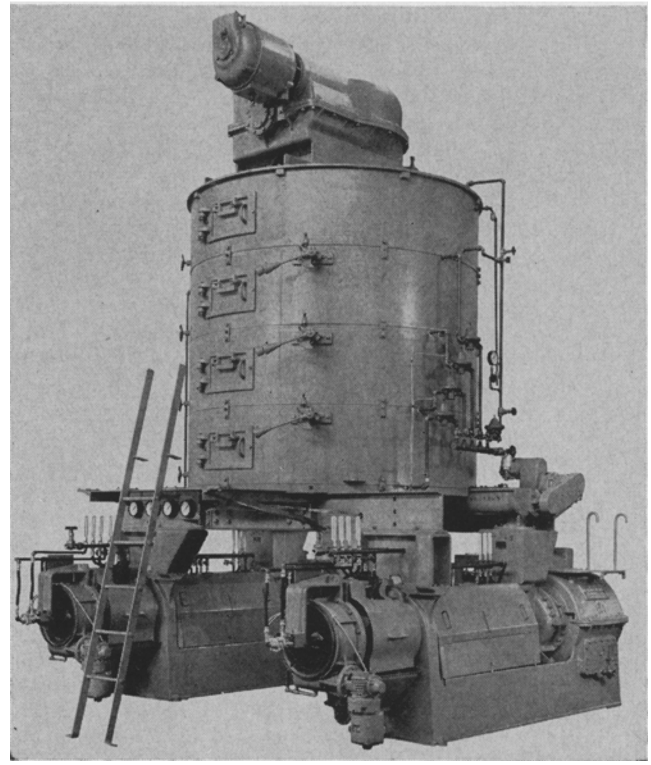

FIG. 2. Two 4-section mechanical screw presses with extension cages having a 4 high, 100-in. diameter cooker, serving both presses.

Perhaps the most important step in the development of mechanical-pressing methods was the introduction of the continuous mechanical screw press, replacing the hydraulic pressing process; and strange as it may seem, there is a great deal in common between the hydraulic press and the screw press in their action on cottonseed meats and their reaction to cottonseed meats. It has always been important to control the length of time of the application of low pressure in a hydraulic press, and it was considered advisable not to subject the cake to too sudden an application of pressure but rather to let the cake set up gradually before the high pressure was applied and then to apply the high pressure gradually and constantly. The screw press reacts in much the same manner. It was found that the same principles relating to application of low pressure in a gradual manner with a continuous and ever-increasing pressure yield the best results on screw presses as well as on hydraulic presses.

Just as in a hydraulic press, a high oil yield from a screw press is also dependent upon drainage time and pressure. The emphasizing of one and the neglecting of the other of these two elements beyond certain proportions is unwise for both of these elements are vitally important.

In hydraulic pressing, oil mills went through a cycle where it was first learned that thin cake seemed to give better extraction than did heavier cake with the same time under pressure. Then it was discovered that by increasing the weight of the cake by 25% and increasing the time under pressure by 25% and by keeping the tonnage the same, an improvement in extraction could be obtained, thanks to the longer drainage time. During the past few years the trend in screw presses has been toward higher capacity per machine in order to keep investment costs at as low a level as possible and to obtain as high a return on investment as possible when converting from hydraulic to screw presses. Several years ago when the demand for higher capacity machines became evident, what was already known pertaining to the advantages of long pressing time on hydraulic presses was applied to screw presses. At that time the standard screw press had a main cage consisting of four sections, each 11 in. long, in addition to the low pressure auxiliary cage. A 9-in.-long extension cage was added to the press, having the effect of increasing the drainage time by approximtaely 16%. In general, this resulted in improvement in extraction even as press capacity was increased slightly.

From this early attempt to increase drainage time in screw presses evolved the idea of increasing drainage time still farther. A 9-in. extension cage was replaced by an 11-in.-long water-cooled extension cage and later another 11-in., water-cooled extension cage was added to that. The addition of 22 in. of drainage cage to the standard 4-section screw press provided approximately 40% longer drainage time than had formerly been available on screw presses and approximately 50% longer drainage time under high pressure.

Considerable effort was expended to incorporate into the design of extension cages complete water-cooling for extensions to provide adequate cooling for the material as it is subjected to higher pressures in the extension cages. This is important in order to produce products of at least as high a quality as have been produced by water-cooled presses which did not have extension cages.

In addition, the design permits the addition of extension cages to a press without increasing the over-all length of the press. This was largely made possible by simplifying and improving the design of the "cone" or "choke" mechanism, which controls the thickness of the cake as it is discharged. This mechanism had formerly been such that the cake was discharged in a dark recess and required a cake breaker to cause the cake to spout easily. All of this made it dangerous for the operator to reach in to get a sample of the cake.

The new "easy-view" safety-cone mechanism, as shown on the discharge end of the press in Figure 1, discharges the cake directly from the front of the press and requires no dangerous cake-breakers. This makes it possible for an operator to observe the cake as it is being discharged from any position in front of the press, offering another convenience to the operator for maximum ease of operation.

The development of extension cages and the theory of longer drainage time in screw presses, while it was merely the application of the principle which had been learned many years before in hydraulic presses, proved to be one of the major advances in the continuous mechanical pressing-process. It was conclusively proven that it is possible for a mill to operate continuously, leaving consistently less than 3.0% oil in finished meal while making high quality products, a meal as bright in color as hydraulic meal and an oil of superior quality. It was also proven that, at lower capacities and increased drainage time, additional improvements in extraction could be made to less than 2.5% residual oil in meal. This is evidenced by the average results from a recently installed cottonseed oil mill, as shown in Table I. In fact, it has been proven by tests in mills all over the country that, regardless of operating conditions, the addition of extension cages to increase the length of the high pressure drainage time improved extraction lowering the residual oil content of the cake from between .7% to 1.5%.

TABLE I
Chart Showing Average Results of a Cottonseed Oil Mill Installed in August of 1954

Equipment: 2 French mechanical screw-presses with 100 h.p. motors; 22-in. water-cooled extension cages; individual 4 high, 85-in cookers. Installed August, 1954.
Daily Mill Throughput: 80 to 85 tons of cottonseed.

Average for Period	Cake Analyses		
	Moisture	Oil @ 8.00% Ammonia	Standard
1st Week of Operation	4.45	2.82	.36
4th Week of Operation	4.65	2.49	.31
6th Week of Operation	3.92	2.50	.31
8th Week of Operation		2.40	.30

This represents a gain of oil over what was formerly expected from screw presses equal to the gain of screw presses several years ago over hydraulic presses, which at that time justified replacing hydraulic presses with screw presses. The oil gain over hydraulic pressing processes therefore has been approximately doubled in the past few years.

It has long been the wish of oil-mill operators that some method be provided to vary the speed of the press quickly and easily. There are occasions when an oil mill finds itself in the position where it would be very advantageous to be able to increase the throughput of their machines for a short period of time. It is also possible that, as the worm flights which force the material through the press become worn, the slippage of material over the flights will reduce capacity. This generally has no effect on extraction, but often a reduction in throughput can seriously increase the operating cost of a mill. Since it is not always possible for a mill to shut down a machine for an extended period for repairs to the worn parts during its operating season, it is convenient to be able to increase the speed of the press to make up for any loss in throughput, providing this can be done quickly and easily. An increase in the over-all speed of the press will recover the loss in capacity caused by worn worm flights without affecting extraction and allow the mill to continue its operation at peak efficiency until such time as it is convenient to shut down.

One of the recent developments in the design of the screw press is the introduction of the vari-speed motor mount, as shown in detail in Figure 1. It permits changing the over-all speed of the press quickly and easily in a matter of minutes without requiring external lifting devices or special equipment.

It has been possible to discuss only briefly a part of the improvements in the mechanical extraction of vegetable oils. Current and proposed developments will certainly lead to further improvements in the efficiency of the vegetable oil mill industry, which is constantly demanding finer machinery to meet the high standards established by the industry for a more profitable operation.

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Methods of Analysis for Functional Groups in Earth (Mineral) Waxes[1]

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SINCE J. KOETTSTORFER (1) utilized the saponification value as a controlling factor in determining the quality of butter, this and other methods of analyses such as acid, carbonyl, and hydroxyl values have been used generally to characterize and identify natural fatty products. However the reliability of these analyses when applied to mineral waxes is usually overestimated.

This investigation reports some available as well as some modified methods useful for characterizing earth waxes. These have been applied to crude peat wax and to known control chemicals. Crude peat wax in this report refers to that part of bitumen which is soluble in boiling ethanol (94 wt. %) but is precipitated at —7°C.

Experimental

Equipment and Materials. The Fisher Titrimeter, steam bath, and saponification reflux vessels[2] were used in a majority of the following experiments. Thiophene-free benzene and 94 wt. % ethanol were used as solvents; 94 wt. % ethanol was also used in standard and indicator solutions.

Acid Value. The first analytical procedure studied was the direct titration of the sample with 0.1 N potassium hydroxide, using an indicator (2). This method, although the most common one, is limited to solutions of materials where a color change can be easily discerned. The other color-indicator method studied is suitable for dark solutions because the high molecular weight acids found in such samples are converted into insoluble calcium salts by treating the solution with sodium acetate and then with calcium chloride. Simultaneously an equivalent quantity of acetic acid is liberated and therefore easily titrated (3).

In the potentiometric titration method the sample was weighed into a saponification vessel and dissolved in a solvent mixture of 30 ml. of benzene and 120 ml. of ethanol. The solution was refluxed for five minutes on a steam bath, cooled to 30–40°C., then titrated with 0.5 N alcoholic potassium hydroxide, using the titrimeter. Because the maximum rate of change in pH was difficult to observe, the end-point was taken at meter readings pH 9 and at pH 10. pH 9 gave results closer to the theoretical value.

In the above methods a 1.00 ± .01-g. sample was used. The results are compared in Table I.

TABLE I
Acid Value

Material analyzed	Method	Mean value	Number of determinations	Range	Standard deviation[c]
Salicylic Acid[a]	Fischer (2)	409.8	10	408.2–411.4	1.4
	Pschorr *et al.* (3)	410.4	10	405.8–419.7	4.8
	Potentiometric (a) pH 9	407.6	6	406.3–408.5	0.9
	(b) pH 10	408.2	6	407.4–409.2	1.3
Crude wax[b]	Pschorr *et al.* (3)	50.8	9	43.3– 56.9	4.3
	Potentiometric (a) pH 9	51.9	10	48.8– 56.1	2.1
	(b) pH 10	59.3	10	54.2– 61.2	2.1

[a] Theoretical acid value for Salicylic Acid, 406.3 BDH Analytical Reagent "Analar."
[b] From Hydro-Peat of Kivisuo.
[c] Calculated using the equation, $\sigma = \pm \sqrt{\frac{\Sigma (m-x)^2}{n-1}}$

Saponification Value. In the "Ordinary Method" a 1.00 ± .01-g. sample was weighed into a saponification vessel and then dissolved in a 20-ml. benzene-80-ml. ethanol solvent mixture. Exactly 20 ml. of 0.5 N alcoholic potassium hydroxide was pipetted into the solution. The sample was refluxed for 3 hrs. on a steam bath, cooled, then titrated with 0.5 N alcoholic acetic acid, using 1% alcoholic phenolphthalein as an indicator. This procedure can be used only for clear or faintly colored solutions.

The Pschorr, Pfaff, and Berndt Method (3) was modified slightly by employing *0.5 N* solutions instead of *0.1 N* solutions. This is suitable for the analysis of dark products.

In the Potentiometric Method, preparation of the sample was the same as in the "Ordinary Method." After the saponification was completed, the excess potassium hydroxide was acidified with alcoholic sulfuric acid (1 part concentrated sulfuric acid in 5–10 parts ethanol) to pH 3.

Using the titrimeter, the sample was then titrated with 0.5 N alcoholic potassium hydroxide; the amount consumed was indicated by two inflection points, the first being the end-point of the strong acid (H_2SO_4), and the second, the end-point of the liberated, high-molecular organic acids. The saponification value is calculated from the amount of hydroxide used between the two points described. The results are given in Table II.

Hydroxyl Value. The method of Freed and Wynne (4), an outgrowth of procedures by Verley and Bölsing (5) and others, was found to be suitable, with slight modification, for the determination of the hydroxyl value in peat wax.

A 100 ± 2-mg. sample was weighed into a 25-ml. Pyrex test tube, and exactly 2 ml. of pyridine-acetic anhydride reagent (12 wt. % pure anhydride in dry redistilled pyridine) were added. The mixture was heated until the solution boiled, and for one minute

[1] Edited and revised by W. E. Parker, Eastern Regional Research Laboratory, Philadelphia, Pa.
[2] Described in A.S.T.M. D 399-52.