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# Technical Program Emphasizes Processing and Methods

UCH interesting and helpful information of both practical and theoretical nature was presented at the technical sessions of the 38th annual meeting of the American Oil Chemists' Society in New Orleans, May 20-22, 1947. A new antioxidant, nor-conidendrin, which is present in sulfite waste liquor from the manufacture of the pulp of western hemlock was disclosed. Extraction of cottonseed flakes with isopropyl alcohol was shown to reduce the gossypol content of the meal to approximately 0.01% and to produce a meal with double the nutritive value of hydraulic pressed meal. Gossypol was found to be nontoxic when fed to experimental animals in quantities normally present in any type of cottonseed meal feed. The toxic constituents are unknown factors present in the cottonseed pigment glands but they are not gossypol or gossypururin. Deodorizations of vegetable oils were shown to increase the stabilities as measured by the active oxygen methods rapidly to a maximum during the initial part of the deodorization. This increase is apparently caused by heat destruction of pro-oxidants rather than by removal of volatile materials during the steam distillation. A possible new source of oil with fast drying characteristics may be that from perennial gourds of the genus cucurbita. These products can be grown on the present arid waste land of the southwestern deserts.

### Processing Problems

Isopropyl alcohol as a solvent for cottonseed meal extraction was discussed by W. D. Harris (1). It was shown that a gossypol free meal could be obtained and that this meal which did not require excessive heating had a much higher nutritive value than did hydraulic meal which is always severely heated in order to inactivate the toxic constituents. Rather high solvent to meal ratios were used during this experimental study but practical application might lower these. Gossypol, fatty acids, phosphatides, raffinose, and neutral oil fractions were prepared from the miscella by combinations of liquid-liquid extraction

# 38th Annual Meeting in Pictures

- 1. Three young trainees from India enjoy the New Orleans meeting (l. to r.): P. B. V. Reddi, K. S. Murti, and S. A. Hussain. Mr. Reddi read a technical paper before the Society, of which Mr. Murti and R. O. Feuge were constants.
- 2. Among the prominent couples in attendance were the following: Mr. and Mrs. Lamar Kishlar, Mr. and Mrs. J. J. Ganucheau, Mr. and Mrs. S. O. Sorensen, and Mr. and Mrs. J. J. Vollertsen. (L. to r.) first row—Mrs. Kishlar, Mrs. Sorensen, Mrs. Ganucheau, and Mrs. Vollertsen; standing—Mr. Sorensen, Mr. Ganucheau, Mr. Vollertsen, and Mr. Kishlar.
- 3. This might be called the long and short of it, but instead it is a group of association representatives: (l. to r.) Francis Scofield, National Paint, Lacquer, and Varnish Association, Washington, D. C.; J. H. Mitchell, Southern Research Institute, Birmingham, Ala.; and C. E. Swift, National Cottonseed Products Association, New Orleans.
- 4. Past presidents of the Society gather at Arnaud's for their annual reunion. Three are missing: E. R. Barrow of Memphis, N. C. Hamner of Dallas, and T. C. Law of Atlanta.
- 5. Committee-man James A. Kime (publicity) confers with a visitor from Norway: Robert D. Nergaard, A/S Johan C. Martens and Company, Bergen.
- han C. Martens and Company, Bergen.
  6. J. C. P. Helm, for 20 years secretary-treasurer of the Society, meets with W. Doss Lumpkin, Filtrol Corporation (left), and George F. Clark, Bennett-Clark Company (right).
- 7. Russell R. Haire, Planters Manufacturing Company, Clarksdale, Miss., receives the Smalley cup as three-time winner of top ratings from J. J. Vollertsen, chairman of the Smalley Foundation Committee.
- 8. Leah Castillon of the Southern Regional Research Laboratory is comparing notes with fellow speakers (l. to r.): A. R. Baldwin, Corn Products Refining Company; W. D. Harris, Texas A & M College; and Wales Newby, Cotton Products Company, Opelousas, La.



with hexane and crystallizations. A study of both laboratory and commercial deodorizations of various vegetable oils was described by A. R. Baldwin (2). The oils were deodorized in a laboratory deodorizer equipped with a takeoff for removal of samples at various times during the deodorizations. It was found that the stability increased rapidly to a maximum when the oil temperature reached 190-195° C. and that further heating to higher temperatures or for longer periods of time caused a slow decrease in oil stability. Color reductions due to the heating followed the stability curves very closely. Stability increases and color reduction both occurred equally well whether the oil was heated in vacuo with or without steam stripping.

R. H. Fash (3) discussed the theory of the behavior of positively charged colloidal phosphatides and color bodies during the refining of vegetable oils by kettle, the Clayton continuous, and the "mist mixing" processes. The negative charges of the in situ formed soapstock particles neutralize the positive charges and precipitate the undesirable colloidal particles. The "mist mixing" process was found to give lower refining losses and much better quality oil than the other processes studied.

Miss E. Leah Castillon (4) described the color development in cottonseed oil during storage of both seeds and crude oil. The rate of increase in the bleach color in the stored seed and stored crude oils was found to be proportional to the original content of pigments in the seed and oils, and to the temperature and length of time of storage. The bleach color was found to develop more rapidly during storage of the crude oils than during storage of the seeds from which the oils were expressed. It was concluded that for bleach color alone it was better to store the seeds than the crude oil.

Large quantities of rice bran oil are potentially available from the dehulling operations in rice mills. However, it is necessary to extract the oil immediately after the bran has been obtained in order to decrease the deleterious effects of the powerful lipolytic enzymes present in the bran. K. S. Murti (5) showed that glyceride hydrolysis occurred in the bran at



the rate of 1% per hour. The wax content of the oil makes it somewhat difficult to process. Approximately 5% wax with a melting point of 75° C. is present in the oil. It is probable that the wax comes from the seed coat and the oil from the rice germ. The best method, at least on a laboratory scale, for refining the oil was presented.

Wales Newby (6) discussed the acid wash method for the determination of soap in oils during various stages of processing. Finished oils were shown to contain soaps in amounts of 0.004-0.008%. The presence of as much as 0.24% soap in the neutralized oil during the bleaching did not affect the Lovibond color of the bleached oil as measured by transmitted light. However, the color, as noted by reflected light, was appreciably different from that of the bleached oil which had contained no soap during bleaching.

Analytical Methods

One of the original purposes for founding the American Oil Chemists' Society was to develop and standardize methods of analysis for the fat and oil industry. This meeting, like many of the previous meetings, has brought forth several new methods of analysis and improvements over the old ones. R. T. Milner (7), the new president of the society, described a method for the determination of nitrogen in vegetable oils. The nitrogenous compounds are concentrated into an aqueous phase by preliminary acid hydrolysis of a large sample of oil. After separation the aqueous phase is digested and distilled with ordinary Kjeldahl equipment and the nitrogen determined with Nessler's reagent. With quantities of nitrogen as small as one p.p.m. reproducibility is within plus or minus 20%.

A method for the determination of saponification numbers in dark colored oils was discussed by V. L. Frampton (8). The oil is saponified in isopropyl alcohol. Enough propylene glycol or ethylene glycol is then added to give a 50-50 alcohol-glycol mixture and the excess potassium hydroxide is determined by back titration with an isopropyl alcohol solution of hydrochloric acid. Spirit blue is used as the indicator or the solution may be titrated potentimetrically using the glass electrode. A method for phosphorus determination in organic materials was also described whereby the oxidation is carried out in a muffle furnace in the presence of ammonium and magnesium nitrates. This procedure gave the author much better results than by oxidations with hydrogen peroxide or sulfuric and nitric acids. Final determination of phosphorus was made spectrophoto-

## 38th Annual Meeting in Pictures

9: The egg exhibit of G. R. Ingram showing the effect of gossypol or other substances in cottonseed meal upon the storage quality of eggs attracted many visitors.

10. J. J. Vollertsen thanks the Society for the citation just given him by J. P. Harris, outgoing treasurer (right). Col. H. P. Newton is at left. Seated are Mrs. S. O. Sorensen,

Mrs. T. H. Hopper, and Mr. Sorensen. 11. Snapped at the banquet on May 22, 1947 are some of the new officers of the Society, who will serve until May, 1948 (l. to r.), seated: J. J. Vollertsen, treasurer; R. T. Milner, president; C. P. Long, first vice president; standing: E. M. James, second vice president; V. C. Mehlenbacher, third vice president; and H. L. Roschen, secretary. Absent is L. B. Parsons, fourth vice president.

12. G. R. Ingram of the Alabama Polytechnic Institute,

Auburn, starts his egg-breaking demonstration.

13. Outside the University room, where the sessions were held on Wednesday and Thursday, were a group of members: (l. to r.) M. E. Whitten, U. S. D. A., Washington; C. B. Combs, Green Bros., Inc., Dallas; Charlotte H. Boatner, Southern Regional Research Laboratory, New Orleans; W. T. Coleman, West Texas Cotton Oil Company, Abilene; and A. M. Altschul, program chairman for the 38th annual meeting, New Orleans.

14. A stairway meeting was chosen by E. H. Chapin, advertising chairman for the Journal, Foster Wheeler Corporation, with George R. Fitts, Read Machinery Company, York, Pa.; and C. C. McInnes, American Mineral Spirits

Company, Chicago.

15. The registration desk was always busy. Going through the line is Potter Holmes, South Texas Cotton Oil Company, Houston. Behind the desk (l. to r.) are E. L. D'Aquin, W. S. Singleton, E. F. Pollard, and (standing) Mrs. T. H. Hopper and E. A. Gastrock.

16. Scientific discussion was not always confined to the

lecture hall, or committee meetings to the parlors.



metrically. Mr. Frampton also pointed out that gossypol should be removed from oil samples before the determination

of a-tocopherol.

J. P. Awapara (9) has applied the fluoridin adsorption technique to the determination of vitamin A in various foods. It is based upon the quantitative removal of vitamin A by means of activated floridin XXF. This absorbent reacts with vitamin A and the carotenoids and the reaction products are held tightly on the surface of the adsorbent. By using a small amount of the floridin in the form of a thin layer in an adsorbent column, other materials are not adsorbed. Hence a portion of the nonsaponifiable fraction, when treated with floridin, offers a blank which can be used against the untreated portion. The difference between the spectral curves of the blank and the sample then represents the amount of vitamin A and earotenoids present in the original product. The procedure has been applied successfully to margarine, butter, eggs, and milk. J. G. Baxter (10) reviewed the methods of tocopherol analyses and suggested that purification of tocopherol concentrates by molecular distillation before analysis greatly simplified the assay. He found that the tocopherol potencies for extracted, expelled, and degummed soybean oils were all the same. From 1-5% loss of tocopherol was encountered during centrifugal refining and 12-20% loss on batch refining. There was practically no disappearance of tocopherol on bleaching, and the average deodorization loss was about 5%.

Verification was offered by H. C. Black (11) for the hypothesis that the ability of an antioxidant to stabilize the fat in baked goods is a function of its solubility characteristics. On the basis of this observation a laboratory method involving the partition of antioxidants between equal amounts of fat and hot water has been developed. Good correlation was found between this laboratory test and actual bakeshop results.

#### Nutritive Effects of Cottonseed Pigments

A broad program involving 11 individual investigations in academic, commercial, and government laboratories is in progress on a study of the nutritive effects of the pigments and pigment glands separated from cottonseed meal by the gland flotation process. Charlotte Boatner (12) described some of the work from four of the collaborating laboratories (Ralston-



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Purina Co., Bureau of Animal Industry, Bureau of Home Economics and Human Nutrition, and Swift Company). Gland free cottonseed flour prepared by the gland flotation process or diethyl ether extracted cottonseed meal had much superior nutritive value over hydraulic pressed cottonseed meal or hexane extracted, uncooked cottonseed meal and screw press soybean meal. The deleterious factors of cottonseed are segregated in the pigment glands. These factors are inactivated during cooking of cottonseed, but the biological value of other thermally unstable components of the meal are simultaneously reduced. Removal of the contents of the glands by exhaustive extraction with diethyl ether or acetone or removal of the intact glands yields a protein meal of superior nutritive value. It was also pointed out that the toxicity of the cottonseed was not due to gossypol or gossypururin and that it would be very difficult to get enough of these compounds in ordinary feed to kill animals. It was also pointed out that lysine has been found in native cottonseed protein but not in cooked hydraulic press meal. G. R. Ingram (13) described the results of several different tests on the storage of eggs from chickens which have been fed various cottonseed meals and cottonseed meals treated by several different methods. It was proven that the cottonseed meal cooked in the presence of excessive moisture or the addition of  $\frac{1}{2}\%$  ferrous sulfate to the ration eliminated most of the dark yolks of the eggs stored for 3-4 months in the cold. A combination of these two treatments almost eliminated yolk discolorations. Results of one test conducted on cottonseed meal produced by the gland flotation process indicated that this meal causes little discoloration of egg yolks during cold storage. An accelerated method to note the discoloration of eggs during cold storage was presented. The eggs are stored in closed containers in which there is a tray of 8% aqueous ammonium hydroxide for 24 hours at room temperature. It was found that this time is equivalent to 6-8 months cold storage as far as egg discoloration due to cottonseed meal feeding is concerned. T. F. Zucker (14) has found that contrary to previous claims purified gossypol has no generally toxic properties and furthermore it has no action on the heart as measured by the electrocardiograph. A single dose has a transient effect on the stomach in delaying the passage of food. Daily administration leads to very low food intake and possible eventual starvation. When the whole pigment gland is fed, an unidentified factor which is neither gossypol nor gossypururin leads to intestinal irritation and serous exudates.

### General Subjects

A polyphenolic substance, conidendrin, is present in many coniferous woods. G. S. Fisher (15) described a method for obtaining appreciable quantities by extraction with trichloroethylene of the sulfite waste liquors from the manufacture of pulp from western hemlock. Treatment of this material with acetic and hydrobromic acids produces the compound nor-conidendrin which is similar in structure to nor-dihydroguaiaretic (NDGA). Its antioxidant activity is comparable to that of NDGA and it contributes no objectionable odor, color, or flavor to fats. Although its toxicity has not yet been measured, it was not believed to be more toxic than NDGA.

W. C. Ault (16) presented data on the oils from the seeds of perennial gourds, cucurbita palmata and cucurbita digitata. These grow wild in the southwestern states, particularly in arid regions, and it is possible that these plants may be successfully grown on otherwise commercially useless land. The seeds in general are similar to pumpkin seeds and represent \( \frac{7}{23} \) by weight of the whole dry fruit. The seeds contain 28-30\% oil which is characterized by the presence of 10-17% of a conjugated trienoic acid similar to the eleostearic acid of tung oil.

A series of hydrazides of the normal fatty acids was described by W. G. Bickford (17). Physical and chemical properties were determined, and it is possible that the preparation of hydrazides may be used for identification of small samples of fatty acids or methyl esters of fatty acids. The reaction of ethyl oleate and hydrazine led to the formation of the hydrazide of stearic acid whereas similar reaction with ethyl elaidate yielded the hydrazide of elaidic acid.

R. T. Doughtie, Jr (18) re-emphasized the need for care in sampling oil seeds for official analyses and he mentioned many precautions which should be taken during the sampling of cottonseed, soybeans, and peanuts.

-A. R. Baldwin.

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# Royal-Stafolife Opens New Laboratory at Memphis, Tenn.

ROYAL-STAFOLIFE MILLS, pioneer manufacturers of livestock and poultry feeds, have recently completed an office and laboratory building

joining their mills at 800 Provine avenue, Memphis, Tenn. Special apparatus has been installed for crude fibre determinations, extractions and solvent recovery, protein tests, moisture tests, ash determinations, and other assays and tests. The laboratory was planned by Joe N. Pless, chief chemist for the past 17 years. In 1932 he won the trophy for the top rating in both oil and protein determinations conducted by the



JOE N. PLESS

Smalley Foundation Committee of the American Oil Chemists' Society. Five times he has been awarded the certificate for highest accuracy in protein tests.

### DR. C. A. BROWNE DIES

Dr. C. A. Browne, honorary member of the American Oil Chemists' Society, died on February 3, 1947. He was a resident of Washington, D. C., and had been a member since 1925. He was a collaborator in the Bureau of Agricultural and Industrial Chemistry of the U.S. Department of Agriculture.



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