vary with the nature of the substance being analyzed. In the work done 6-hour extractions have been more than sufficient for the wide variety of substances examined. It is believed that the 6-hour figure can be safely reduced for many types of materials; however the method as proposed is designed to analyze a wide range of products (See Table V).

No emulsion difficulties were encountered during the extraction of unsaponifiable matter from any of the samples examined. Although with some substances, still pitches for example, slight emulsion will tend to form if the agitator is rotating too fast or if the solvent is refluxing too rapidly. This difficulty can be corrected by making appropriate adjustments in agitation.

Washing of the final extract solution with 10% aqueous alcohol as required in the A.O.C.S. procedure can be eliminated since no soap is entrained in the solvent if the extractors are operating properly. However one alcohol wash is recommended as a precautionary measure. The A.O.C.S. Method (1) requires correction for traces of fatty acids. This correction is included in the method proposed here; however for most purposes the correction is so insignificant that it could be eliminated. A similar conclusion was reached by Wood and Roschen (3).

Summary

1. A continuous liquid-liquid extractor was designed and procedures were devised for the determination of total fatty acids, oxidized acids, and unsaponifiable matter. These procedures give results that are in agreement with the present A.O.C.S. Methods.

2. The continuous liquid-liquid extractor has the following advantages:

a) It is simple in design, easy to operate and keep clean. The inner tube which gives better solvent dispersion than standard commercial pieces can be made out of ordinary glass tubing at relatively low cost.

b) The operation is automatic. Once a bank of these extractors is set up, the operator is free to do other work.

c) The extractions are made rapidly without troublesome emulsions.

d) Greater accuracy is possible because of limited handling.

e) The enclosed system minimizes health and fire hazards inherent in the present A.O.C.S. Methods.

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A New Batch Solvent-Extraction Pilot Plant[1]

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AN all-stainless steel, batch solvent-extraction pilot plant has been designed, constructed, and test-operated at the Southern Regional Research Laboratory. The plant is illustrated in Figure 1. It is an improvement over an earlier plant (4), which was used for more than 200 extractions. Its stainless steel construction makes it particularly useful as a research tool for the extraction of a variety of vegetable and other materials, either native or specially prepared, with suitable solvents for the production of meals, oils, pigments, and other constituents, of essentially unmodified quality and purity or with special qualities. Features of the plant are an improved batch extractor, a high-velocity rising-film type evaporator with its vacuum and other auxiliary equipment, and pneumatic control and mechanical recording instrumentation.

Design and Installation

The plant is located outdoors to minimize dangers when solvents, such as ethyl ether are used, and is installed on a structural steel frame mounted on reinforced concrete pedestals, which are monolithic with a reinforced concrete slab. Figure 2 is a flow chart which defines the major equipment units and their positions in the process. Designed for versatile and labor-saving operations, and in accordance with the ASME Code for Unfired Pressure Vessels, one of the existing extraction units with a capacity of 5 cubic feet is shown at the center of Figure 1. Although the extractors have capacities of 3.5 and 5.0 cu. ft., found during the past 8 years to be the most practical sizes for the extraction of small lots of new and established materials, extractors of this design with other capacities can be made and used by increasing or decreasing the length of the unit. Figure 3 illustrates the detail of the extractor. It is made of 14-in. 304 stainless steel pipe and is jacketed with 18-in. carbon steel pipe. Its accessories are of 316 stainless steel construction. Hot water can be circulated through the jacket when extractions are to be made at other than ambient temperatures, and the extractor is provided with an agitator which is manually operated at intervals to reduce channeling and at the same time not create too many fines. The agitator shaft has two sections to permit removal of the upper section along with the top head when charging flakes into the extractor. Two filter frames covered with cloth are mounted in the top head for filtering the miscella as it leaves the extractor on its way to the evaporator. For easy discharge of the extracted meal into a large can, the bottom head is hinged to the body with silicon-bronze hinges. Immediately above the bottom flange on the interior of the extractor body is a retaining screen which restrains the meal from discharging while the bottom head is swung down and the collector is slid beneath the extractor. Since tilting of the extractor is not required for discharging, it is bolted rigidly to the steel through two support lugs. There is a solid baffle above the solvent inlet in the bottom head to reduce the channeling of the solvent.

[1] Presented at the fall meeting, American Oil Chemists' Society, Nov. 2-4, 1953, in Chicago, Ill.

[2] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

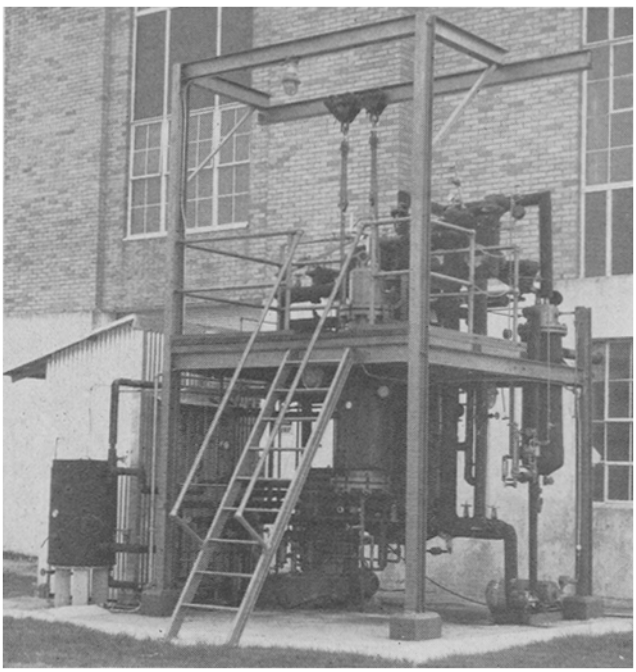


FIG. 1. New batch solvent extraction pilot plant.

The design makes the extraction vessels useful for other experimental work also, such as the chemical treatment of fibrous materials. For example, they can be adapted for the treatment of cotton fiber with monoethylamine to increase the elongation of the fiber in studies being conducted to improve the resilience of cotton cloth.

The evaporator is of the long-tube, rising-film, high-velocity type and is of 304 stainless steel construction on the tube side. It is designed for operation at low absolute pressures (vacuums as high as 27 in. of mercury) and low temperatures to avoid damage to heat-sensitive products such as concentrated cottonseed oil miscellas (7). The evaporator with its vacuum and other auxiliary equipment can be operated independently of the extractor for recovery of liquids and concentration of various liquid mixtures produced in other pilot plant operations. Six tubes of ¾-in. outside diameter and of 9-ft. length comprise the tube bundle, and space is available for two additional tubes. On the separator, two sight glasses 180° apart permit the operator to observe the action of the evaporator and to watch for foaming. Automatic control of the diaphragm-type steam-feed valve immediately before the evaporator is obtained by means of a pneumatic recorder-controllor with a heat-sensitive bulb located in the head immediately above the tube bundle of the evaporator.

The plant is not equipped with a stripping column. Stripping of small quantities of materials may be carried out in a Pyrex laboratory-sized stripper.

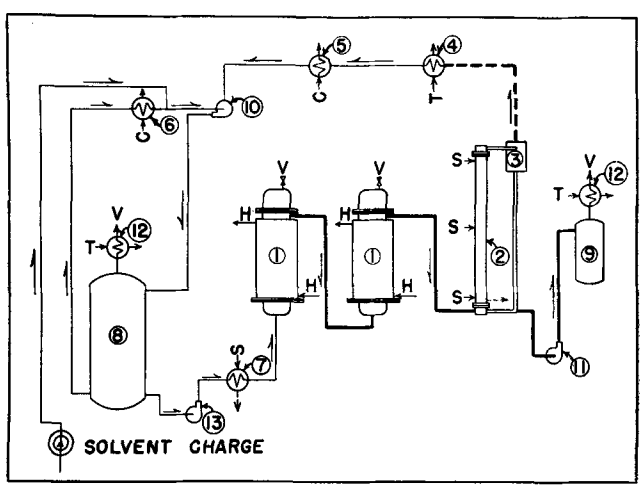
Heat-exchange units of standard design and manufacture are used for the evaporator condenser, the solvent after-cooler, the vacuum-pump-seal cooler, and the solvent preheater. All are four-pass units except the evaporator condenser, which is a single-pass unit. Two vent condensers are provided, one in the solvent tank vent line and the other in the miscella tank vent line. Construction is of jacketed 304 stainless steel pipe.

Solvent tank and miscella tank are designed in accordance with the ASME Code for Unfired Pressure Vessels. The solvent tank has a capacity of 150 gal. and is made from 24-in. 304 stainless steel pipe and two dished heads. It is equipped with an 8-in. handhole near the bottom for cleaning. The miscella tank has a capacity of 35 gal. and is made from 12-in. 304 stainless steel pipe and 2 dished heads. It consists of two sections with a flange located near the top head. Fifty-five-gallon steel drums are used for the cold and hot water tanks.

The vacuum pump is the wet rotary type of 302 stainless steel construction and is suitable for operation with the solvents processed as combined sealing and lubricating liquids. The evaporator-discharge pump is a rotary gear pump constructed of 304 stainless steel and has 316 stainless steel impellers. Both solvent pumps are rotary gear pumps, and each is coupled to a hydraulic variable-speed transmission. Centrifugal pumps of all-steel construction were used for the hot-water and cold-water pumps. A tap-water booster pump (bronze gear pump) is used to overcome occasional fluctuating city-water pressure.

The solvent and miscella piping systems consist of 304 and 316 stainless steel fittings, and light and standard wall pipe in most cases soldered together with low-melting-point silver-alloy-solder. There is only a minimum and probably a negligible area of silver solder exposed to the solvents and liquids. Because screwed stainless steel joints are difficult to maintain leak-proof, they are used in these systems only in cases in which it is considered impractical to solder, for example, for coupling to equipment units which are not provided with pipe flanges. Pipe and fittings of appropriate materials constitute the service piping systems.

Temperature and pressure indicating and recording instrumentation are installed strategically



1. EXTRACTOR
2. EVAPORATOR
3. SEPARATOR
4. EVAPORATOR CONDENSER
5. SOLVENT AFTERCOOLER
6. PUMP SEAL COOLER
7. SOLVENT PREHEATER
8. SOLVENT TANK
9. MISCELLA TANK
10. VACUUM PUMP
11. EVAP. DISCHARGE PUMP
12. VENT CONDENSERS
13. SOLVENT PUMP

— SOLVENT
— MISCELLA
--- VAPOR
--- CONDENSATE
S - STEAM
C - COLD WATER
H - HOT WATER
T - TAP WATER
V - VENT

FIG. 2. New batch solvent extraction plant.

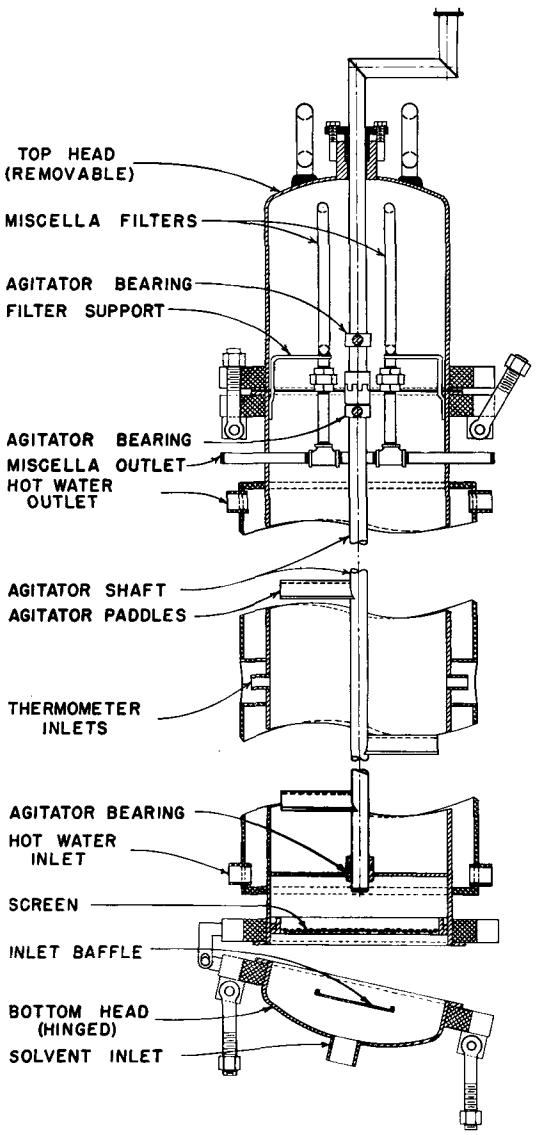


FIG. 3. Improved batch extractor.

throughout the plant so that each unit can be observed and controlled independently. A pneumatic recorder-controller, already mentioned, regulates the flow of steam to the evaporator. Similar in design to that described by Decossas *et al.* (1) is the electrical installation which consists of Class I, Group D, equipment. An operator's house located at the end of the plant shelters the electrical starting switches and the recording instrumentation.

Acid-resistant blue asbestos is used as gasket and packing material in the solvent and miscella systems. All exterior metal surfaces are protected from corrosion with two coats of heavy duty industrial stainless steel paint. Equipment and piping are thoroughly insulated with either standard or double-standard-thickness magnesia on the hot surfaces, and brine-thickness cork on the cold surfaces.

Test Operation

During five test runs conducted, a total of 660 pounds of cottonseed meats from three lots were flaked to approximately 0.010 in. thick on cracking and flaking rolls and extracted. None of the runs was made on a round-the-clock basis. In each case, with the exception of the first run, the bottom of the extractor had been charged with fresh glass wool and tightened in place immediately after discharging the extractor at the conclusion of each previous run. Prior to each extraction, clean extractor filters were installed.

Commercial hexane was the solvent used during the first three runs for the extraction of cottonseed oil, and 2-butanone (methyl-ethyl-ketone) with 1.5-2.5% water (by volume) and preheated to 145°F., was used during the other two runs for the extraction of gossypol. In all of the runs initial filling of the extractor with solvent was accomplished at the rate of 1 gal. per minute. After initial filling this solvent rate was reduced to a rate equal to the maximum capacity of the evaporator, either approximately 0.7 gal. per minute of hexane or 0.5 gal. per minute of butanone. Continuous evaporation at 15½ in. Hg. vacuum was accomplished with concentration of miscella to approximately 50% oil by weight in a single pass and to as much as 97% oil by weight in a double pass. Two rapid passes of the miscella through the evaporator has been found preferable to one longer pass. Boiling point data obtained by Pollard *et al.* (3), and specific gravity data obtained by Magne and Skau (2) were used in determining and setting conditions for operation, and for evaporator control.

Data from the Test Runs are compiled in Table I. In Runs 2 and 3 lipide extraction efficiencies were 97.3% and 97.1%, respectively, and the residual lipides in the marc after hexane extraction were 0.85% and 1.07% by weight, respectively. A prime oil was

TABLE I

Batch Solvent Extraction Data

Test Run No.	Material[1]	Batch wt. lb.	Flake Thickness mils.	Analysis after Extraction				Solvent Used	Extr'n Time[6] Hrs.	Avg. Solv. Rate GPM	Total Solvent[7] Gals.	Temp. Solv. to Extractor Deg. F.
				Lipides[2] %	Moisture[3] %	% Gossypol						
						Free[4]	Total[5]					
2	Cottonseed	135	10-12	0.85				Hexane	5	0.80	250	.....
3	Cottonseed	125	8-12	1.07	6.91			Hexane	4	0.71[8]	170	90
4	Cottonseed	130	8-12			0.01	0.14	Butanone	9	0.45	240	145
5	Cottonseed	142	8-12			0.01	0.19	Butanone	17	0.45	460	145

[1] Analysis of flakes before extraction was as follows: Run No. 2, lipides[2] 32.02%; Run No. 3, lipides[2] 36.59% and Moisture[3] 7.07%; and Run No. 5, free gossypol[4] 0.51% and total gossypol[5] 0.54%.

[2] Lipides were determined by A.O.C.S. Official Method Ba 3-38.

[3] Moistures were determined by A.O.C.S. Official Method Ba 2-38.

[4] Free gossypols were determined by method of Pons and Guthrie (5), now A.O.C.S. Official Method Ba 7-50.

[5] Total gossypols were determined by method of Pons, Hoffpauir, and O'Connor (6).

[6] Extraction time is exact extraction time exclusive of overnight soaking. Overnight soaking resulted from the work schedule employed and consisted of the following: Run No. 2, 1 night for 17 hours; Run No. 3, 1 night for 17 hours; Run No. 4, 1 night for 18 hours; and Run No. 5, 3 nights for a total of 70 hours. The residual lipides could have been reduced to the amounts shown without overnight soaking by extracting continuously for a longer time.

[7] Indicates the quantity of solvent pumped through the extractor.

[8] Reduced solvent rate can be attributed to the severe foaming characteristics of the miscellas.

obtained in Run 2. Analyses of the laboratory stripped oil from Run 2 are: refined color, 35 Y, 5.32 R; bleached color, 20 Y, 1.19 R; lowest refining loss 6.49%. Refinings were done by A.O.C.S. Official Method CA 9a-52 and bleaching tests were made in accordance with A.O.C.S. Official Method Cc 8a-52.

In Runs 4 and 5 free gossypol was reduced in the marc by extraction with butanone to 0.01%, and total gossypol was reduced to 0.14% and 0.19%, respectively. It was shown in Runs 4 and 5, using cottonseed from the same lot, preparing the flakes the same, using the same solvent and the same solvent rate, that the percentage free and total gossypol in the extracted meal from Run 5 was as high as that from Run 4 although twice the quantity of butanone was used and the time of extraction (including soaking time) was more than tripled.

Cost

Material and equipment for the plant cost $15,511 and the installation was $6,512.

Summary and Conclusions

A new improved batch solvent extraction plant and its versatility of design have been described. Data have been given on the cost and initial test operations. The performance of the plant has been tested, and its value as a research tool has been demonstrated by the production of free gossypol and meals low in gossypol for use in research. Simplicity of operation is borne out by the fact that only one operator is needed. A limited number of detail drawings of the plant are available on written request from the Southern Regional Research Laboratory.

Acknowledgment

All publication drafting work by Clyde P. Martin Jr., of the Engineering and Development Division, the assistance given in the design of the plant by F. A. Deckbar Jr., and J. E. Hawkins, both formerly of the Engineering and Development Division, and the assistance and cooperation of A. J. Crovetto in the test operation of the plant are acknowledged. The authors express appreciation to the Mechanical Service Division of this Laboratory for the installation work and their close cooperation, particularly during the installation phase of the project.

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The Effect of Electrolytes on Soil Redeposition in Laboratory and Laundry Practice

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J. POWNEY and R. W. Noad (1) examined the deposition of ilmenite black on cotton fabric in the presence of soap, synthetic detergents, and certain sodium salts, such as chloride, carbonate, hydroxide, silicate, and phosphate. They found that soap gave good protective action which decreased with rise in pH and with the addition of sodium chloride or carbonate. Synthetic detergents of the long chain alkyl sulfate type were found to possess low protective action. Similar conclusions were reached by Weatherburn *et al.* (2, 3), using a similar procedure.

To ascertain the contribution of redeposition of soil in total detergent action, deposition of carbon from Aquadag suspensions on cotton cloth was examined in the presence of mono- and divalent cations as well as some protective colloids. Using solutions of the same compositions, these results were correlated with multiple-cycle soiling and washing on hand towels under ordinary home-laundry conditions. The results of Powney and Noad were confirmed and extended.

In the present work synthetic anionic detergents exhibited an inferiority in the laundering of cottons as compared to ordinary soap. It was demonstrated that synthetic detergents wash more poorly in hard than in soft water. Improvement in hard water detergency was noted when synthetics were mixed with phosphate and when certain protective colloid agents were added. The latter also improved soft water detergency. Soap did not show a washing deficiency in hard water, a fact which was apparently due to the removal of hard water ions of calcium and magnesium as insoluble fatty acid salts while an excess of sodium soap remained. Builder salts used either with soap or detergent enhanced soil removal but at the same time tended to increase soil deposition from the wash liquor. The present work also showed that a most important function of builders in the washing process is to suppress the effect of calcium or magnesium ion.

Experimental

I. Soil Deposition Test. A suspension was made containing 0.5 g. of Aquadag[1] in 1 liter of solution containing the salts or detergent systems to be examined. The Aquadag used in these experiments was a 22% solids paste of colloidal graphite in water containing a small amount of suspending agent. The density of the paste was 9.35 lbs./gal. The fineness of the Aquadag particles was arbitrarily designated by the letter "A," which means a maximum particle size of 4 microns with the majority of particles either 1 micron or less and the average size of particles over 1 micron being 2 microns. To the salt- or detergent-Aquadag suspension was added a clean cotton swatch (6 in. x 6 in.), which was stirred in a Tergotometer

[1]Acheson Colloids Corporation, Port Huron, Mich.