

unpublished data (4). The latter were obtained at 171°, 216.1°, and 253.2°F. for Skellysolve B solutions of soybean oil by the method of Calingaert and Hitchcock (2). The comparison of pressure data was made for the two sets of data representing different hexane fractions by applying a factor. This was the ratio of the vapor pressures of the two solvents at the temperature of comparison. The two sets of data became comparable when the pressures of one set were multiplied by the factor at constant temperature and composition.

Comparison in the manner described indicated that the two sets of data agreed as closely as the data reported in this paper conform to smooth curves and within the estimated uncertainty of $\pm 5\%$. Between 171° and 253.2°F. the average difference in the results of the two investigations was $\pm 3.3\%$; the individual differences were $\pm 1.3\%$ at 171°, $\pm 3.1\%$ at 216.1°, and $\pm 5.4\%$ at 253.2°F.

The relationship of pressure and concentration indicates that Henry's law, $p = HX$, is applicable to the experimental data over part of the concentration range. Henry's law commonly represents the relation between partial pressure and mole fraction of a solute in dilute solution. The partial pressure in the equation is the corrected total pressure which was measured as the oil is relatively non-volatile. In this case the solute is hexane.

Calculation of the constant, H , in the equation and its correlation with temperature facilitates the interpolation and smoothing of the experimental data. The constant should vary with temperature according to the Clausius-Clapeyron equation in the same manner as vapor pressure. Average values of the constant have been calculated and plotted on a Cox type graph in Fig. 4. The approximate upper limit of concentration below which Henry's law is applicable is also shown in the figure. Within the limiting concentration the equation $\log H = 6.045 - [997.0/(t + 230)]$ is applicable; t is temperature in °C.

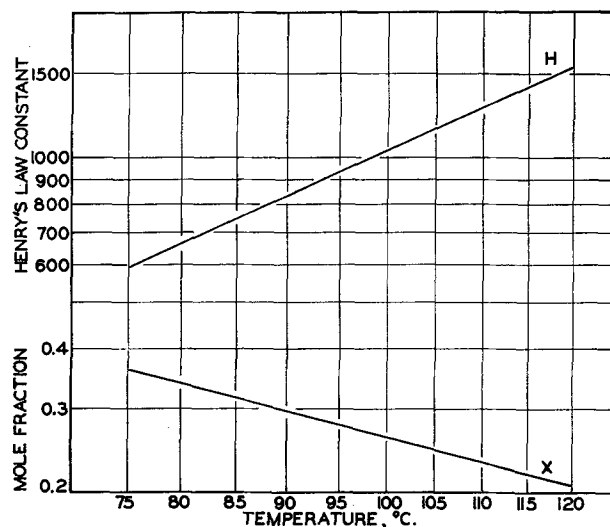


FIG. 4. Henry's law constant and concentration below which the constant is applicable in soybean oil-hexane solutions.

Acknowledgment

The work reported in this paper has been taken from a thesis submitted by the junior author in partial fulfillment of the requirements for the Master of Science Degree. It was supported in part by, and carried out as part of a program under a fellowship provided by the Central Soya Company, Decatur, Ind. The authors express their appreciation to the Central Soya Company, represented by J. P. Miller, for the fellowship and for permission to publish the results of this investigation.

REFERENCES

1. Armour and Company, Technical Data, 1944.
2. Calingaert, G., and Hitchcock, L. B., *J. Am. Chem. Soc.*, **49**, 750 (1927).
3. Pollard, E. F., Vix, H. L. F., and Gastrock, E. A., *Ind. Eng. Chem.*, **37**, 1022 (1945).
4. Smith, Allen S., Funk, J. E., and Braun, Theo. B., presented at the 1946 meeting of the American Chemical Society, Chicago, Ill.
5. Sweeney, O. R., Arnold, L. K., and Hollowell, E. G., *Iowa Engineering Experiment Station Bulletin 165* (1949).

[Received February 20, 1950]

Pilot-Plant Fractionation of Cottonseed: V. A Preliminary Cost Study^{1,2}

R. M. PERSELL, H. L. E. VIX, C. G. REUTHER and E. F. POLLARD, Southern Regional Research Laboratory,³ New Orleans, Louisiana

MUCH has been written on the overall economic aspects of the cottonseed industry, but comparatively little on the direct application of economic thinking to specific engineering developments. In an industry where the price of the seed and of the seed products fluctuates widely in comparison with the general price level (6), the attention of management is naturally focused on buying and selling at the right time. A mill which processes cottonseed with low efficiency can still prosper if the

seed is purchased low and the products sold high. However an increase in the process efficiency of a mill will obviously result in a saving to the mill. For instance, savings have resulted in the past from such developments as improved methods of cooking meats, the introduction of the screw-press, and the beginning of commercial continuous solvent extraction (7, 8, 9). Recently a new process for the fractionation of cottonseed meats has been reported on a pilot-plant scale (12). This process, termed differential settling, removes the whole pigment glands from the meats, producing oil, a pigment gland fraction, and a cottonseed meal essentially free of oil, hulls, and pigment glands (10, 11, 12). The purpose of this paper is to propose a technologically feasible industrial ap-

¹ Report of a study made under the Research and Marketing Act of 1946.

² Presented at the spring meeting of the American Oil Chemists' Society, May 1-3, 1950, in Atlanta, Ga.

³ One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

TABLE I
Estimated Original Cost of Model 200-Ton Per Day
Combination Plant

	1948 Basis
Buildings	\$
Seed Storage, Lint Room, General Office.....	314,318
Extraction-Fractionation Plant.....	52,386
Screw-Press Plant.....	17,868
Equipment	
Extraction-Fractionation Plant.....	354,000
Screw Presses (5).....	184,132
Lint Room.....	330,616
Meal Room.....	74,800
	1,328,120

plication of this fractionation process and to examine such a plant in terms of dollars and cents. This analysis is intended to be a practical guide to the engineer in his research and development and an indication to management of the overall cost considerations of the process. For this purpose a model 200-ton per day combination screw-press extraction-fractionation plant was planned. This size may or may not be the most advantageous size economically for a particular situation. The utilization of the screw presses makes possible the operation of the extraction-fractionation system, using whole meats only as a feed. Such an operation presents a minimum of technologic difficulties and produces purer end products (11).

The flow diagram shown in Figure 1 was selected as a possible industrial application. The cottonseed are run through conventional preparation machinery operated so that half of the seed are fed to the extractor as practically hull-free whole meats; the remainder of the meats are processed by continuous screw presses. The extractor is operated so that the defatted meats contain 4 to 6% oil; the remainder of the oil is recovered in the fractionation system. The elimination of the last and most difficult phase of the extraction cycle increases the capacity of the extractor considerably over that obtained when the meats are defatted to less than 1% oil. In the fractionation system the defatted meal is further defatted to less than 1% oil and separated into a pigment

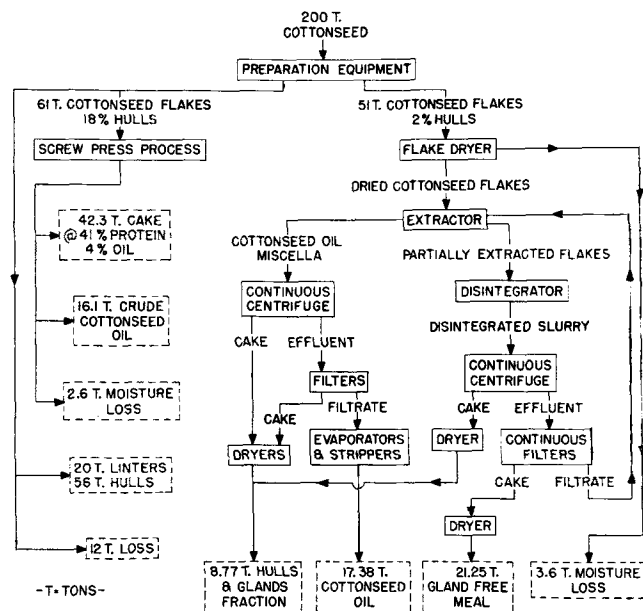


FIG. 1. Flow diagram of model 200-ton per day combination plant.

TABLE II
Operating Expenses of Model 200-Ton Per Day
Combination Plant

	Cost Per Ton Cottonseed Processed		
	Screw Press	Extraction-Fractionation	Plant Av.
Current Operating Expenses	\$	\$	\$
Wages.....	3.0065	3.3495	3.1780
Power, Light, Steam, and Water.....	1.3836	1.9245	1.6541
Solvent Loss.....		0.9250	0.4625
General Repairs.....	0.9800	1.2335	1.1067
Mill Expense.....	0.4554	0.5731	0.5143
Total.....	5.8255	8.0054	6.9156
Fixed and General Expenses			
Depreciation.....	0.9415	1.7507	1.3461
Salaries.....	1.5000	1.5000	1.5000
Interest and Exchange.....	0.2996	0.4086	0.3541
Insurance.....	0.2809	0.3831	0.3320
Taxes (other than Federal).....	0.4682	0.6385	0.5534
Workmen's Liability, Social Security and Unemployment.....	0.3605	0.3880	0.3742
All Other Expenses.....	1.0000	1.0000	1.0000
Total.....	4.8507	6.0689	5.4598
Total Operating Expense.....	10.6762	14.0743	12.3754
Cost of Linter Bagging and Ties.....	0.3600	0.3600	0.3600
Total Conversion Cost.....	11.0362	14.4343	12.7354

gland fraction and a fine meal essentially free of oil, hulls, and pigment glands. The products from the combination plant are linters and hulls from the seed preparation equipment; crude oil and meal cake from the continuous screw presses; and crude oil, purified meal, hulls, miscella cake, and pigment gland fraction from the extraction-fractionation system.

The major items of equipment for such a plant include conventional preparation equipment for 200 tons per day of cottonseed; screw-presses with auxiliary equipment for 61 tons per day of cottonseed meats; a dryer for 51 tons per day of cottonseed flakes; an 80-ton per day solvent-extraction unit with auxiliary equipment; a 50-h.p. dissolver-type disintegrator; a continuous horizontal 32" x 50" bowl centrifugal for separating the disintegrated slurry into an effluent containing the finer than 300-mesh meal and a meal cake containing the pigment glands and coarser than 300-mesh meal; two pressure leaf filters for recovery of the fine meal from 231 tons of

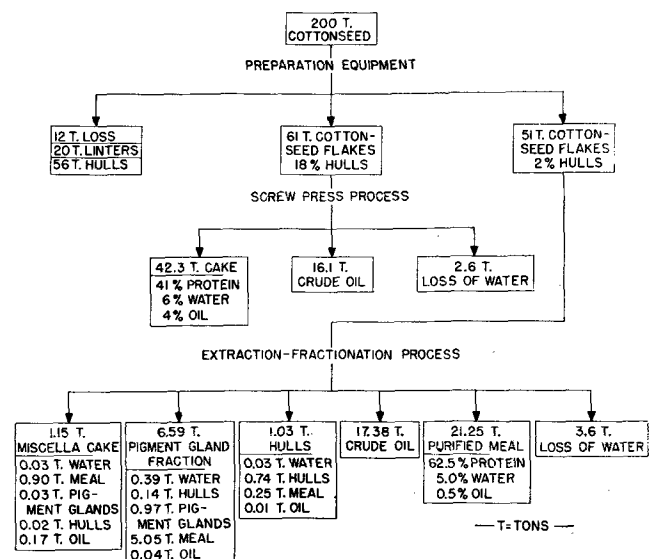


FIG. 2. Material balance for model 200-ton per day combination plant.

TABLE III
Value of Products of Model 200-Ton Per Day Combination Plant

	Income Per Ton Cottonseed Processed								
	1948 Basis			1944 Basis			1939 Basis		
	Screw Press	Extraction-Fractionation	Plant Average	Screw Press	Extraction-Fractionation	Plant Average	Screw Press	Extraction-Fractionation	Plant Average
	\$	\$	\$	\$	\$	\$	\$	\$	\$
Cottonseed Preparation									
Linters.....	8.8200	8.8200	8.8200	8.8600	8.8600	8.8600	6.4800	6.4800	6.4800
Hulls.....	4.1440	4.1440	4.1440	3.6400	3.6400	3.6400	3.1668	3.1668	3.1668
Screw Press									
Crude Oil.....	67.2980	33.6490	41.0550	20.5275	18.1608	9.0804
Meal Cake.....	28.7640	14.3820	20.5155	10.2578	11.6748	5.8374
Extraction-Fractionation									
Crude Oil.....	72.6484	36.3242	44.3190	22.1595	19.6046	9.8023
Hulls.....	0.1524	0.0762	0.1339	0.0669	0.1165	0.0583
Purified Meal.....	22.9738	11.4869	15.3985	7.6993	8.1025	4.0513
Miscella Cake.....	0.8740	0.4370	0.5980	0.2990	0.3324	0.1662
Gland Fraction.....	3.9540	1.9770	2.7053	1.3527	1.5036	0.7518
	109.0260	113.5666	111.2963	74.0705	75.6547	74.8627	39.4824	39.3064	39.3945

effluent per day; and a dryer for removing the solvent from 21 tons per day of purified fine meal.

THE estimated original cost of this plant, the cost of converting the seed, the values of the products, and the calculated spread between the value and the cost of the products are shown in Tables I-IV, respectively. All estimates and calculations were based on 1948 prices. Engineering News Record construction cost indexes were used when it was necessary to convert prices of other years to a 1948 basis. These indexes have been found quite satisfactory for this purpose (5). The calculations and tabulations upon which the totals are based are omitted to simplify the presentation of the overall data.

The figures on converting the cottonseed show the average plant costs and the allocation of these costs to the screw-pressing and extraction-fractionation operations. These costs were calculated from equipment and labor needs based for fractionation on pilot-plant experience and for solvent extraction and screw pressing on industrial experience. Wages were taken from Engineering News Record prevailing wage rates.

The values of the products (Table III) were calculated for the three years 1939, 1944, and 1948 to obtain an estimated spread between the value of the products and their cost under varying economic conditions. This spread can be estimated in a similar manner for the price and cost structure existing today. The products and quantities were obtained from the material balance in Figure 2. The price of the purified cottonseed meal was assumed to be the same as soybean meal and that of the gland fraction as

slightly less than the soybean meal (for 1948, 3.8c per pound for the purified meal and 3c per pound for the gland fraction).

In the calculation of the spread between the value and the cost of the products, per ton of cottonseed (Table IV), the conversion cost for 1948 was taken from Table II and the total value of all products from Table III. The spread for the combination plant as a whole is given for each of the three years to indicate the possible profits under varying economic conditions. The spreads are also shown for those portions of the seed which are produced by the screw-press process and those produced by the extraction-fractionation process. These spreads give the distribution of profit and cost between these two interdependent parts of the combination plant.

The spreads for the combination plant for 1939, 1944, and 1948 show that this combination operation is economically feasible. A comparison of the spreads for the screw-press and extraction-fractionation operations indicates that the cost of fractionation is in line with other types of processing. However the economic advantage of the combination plant over the screw-press operation as shown is small in comparison with the large additional investment required for the combination plant. The present value of the fractionation process lies in the production of two new products, a purified high-protein meal and a concentrated pigment gland fraction, and in the possibility of producing a high-grade oil as the removal of the whole pigment gland prevents the pigment material from coming in contact with the oil. The quantity production of the purified meal and the pigment glands is making possible the investigation of

TABLE IV
Spread Between Value of Products and Cost of Products Per Ton of Cottonseed

	1948 Basis			1944 Basis			1939 Basis		
	Screw Press	Extraction-Fractionation	Plant Average	Screw Press	Extraction-Fractionation	Plant Average	Screw Press	Extraction-Fractionation	Plant Average
	\$	\$	\$	\$	\$	\$	\$	\$	\$
Cost of Cottonseed per Ton.....	67.4000	67.4000	67.4000	52.7000	52.7000	52.7000	21.1400	21.1400	21.1400
Conversion Cost per Ton of Cottonseed.....	11.0362	14.4343	12.7354	7.1580	9.3619	8.2599	5.6498	7.3894	6.5196
Total Cost per Ton of Cottonseed Processed.....	78.4362	81.8343	80.1354	59.8580	62.0619	60.9599	26.7898	28.5294	27.6596
Total Value of All Products per Ton of Cottonseed.....	109.0260	113.5666	111.2963	74.0705	75.6547	74.8627	39.4824	39.3064	39.3945
Total Cost (above).....	78.4362	81.8343	80.1354	59.8580	62.0619	60.9599	26.7898	28.5294	27.6596
Spread.....	30.5898	31.7323	31.1609	14.2125	13.5928	13.9028	12.6926	10.7770	11.7349

the meal and pigment glands for possible industrial or other uses (1, 2, 3, 4).

This discussion has been based on the present stage of development of the fractionation process and the present possible utilization of the products. Pilot-plant work under way indicates improvements in the process, such as a higher recovery of the meal as purified fine meal. The development of additional and more profitable uses for the products would increase their value and provide a broader and more stable market for cottonseed. Such developments would make fractionation, already technologically interesting, a field economically advantageous.

Summary

A preliminary cost study was made of a combination screw-press extraction-fractionation plant. The economic advantage of the combination plant over the screw-press operation as shown here is small in comparison with the large additional investment required for the combination plant. The present value of the combination process lies in the production of two new products, a purified high-protein meal and a concentrated pigment gland fraction, and in the possibility of producing a high-grade oil as the removal of the whole pigment gland prevents the pigment material from coming in contact with the oil. Pilot-plant work under way indicates improvements in the process such as a higher recovery of the meal as purified meal. The development of additional and

more profitable uses for the products would increase their value and provide a broader and more stable market for cottonseed.

Acknowledgment

The authors wish to express their appreciation to Joseph L. Hecker and to Jack E. Hawkins for making the drawings for the charts.

REFERENCES

1. Arthur, J. C., Jr., and Karon, M. K., *J. Am. Oil Chem. Soc.*, **25**, 99-102 (1948).
2. Boatner, C. H., Altschul, A. M., Irving, G. W., Jr., and Pollard, E. F., *Poultry Science*, **27**, 315-328 (1948).
3. Castillon, L. E., Hall, C. M., and Boatner, C. H., *J. Am. Oil Chem. Soc.*, **25**, 233-236 (1948).
4. Eagle, E., Castillon, L. E., Hall, C. M., and Boatner, C. H., *Archives of Biochemistry*, **13**, 271-277 (1948).
5. Hoppel, J., Aries, R. S., and Borns, W. J., *Chem. Eng.*, **53**, (10), 99-102 (1946).
6. McVey, D. H., and Scarce, J. L., Working Manual for Cooperative Cottonseed Oil Mill Operators, F.C.A., U.S.D.A., Miscellaneous Report 128 (1949).
7. Markley, K. S., *The Cotton and Cotton Oil Press*, **47**, (17), A-3 to A-6 (1946).
8. Markley, K. S., and Lynch, D. F. J., *Proceedings of the First Cotton Research Congress*, 211-224 (1940).
9. Moore, N. H., *Food Industries*, **38**, 471-473 (1947).
10. Spadaro, J. J., Persell, R. M., Murphey, C. H., Jr., Vix, H. L. E., McCourtney, E. J., Hecker, J. L., Pollard, E. F., and Gastrock, E. A., *J. Am. Oil Chem. Soc.*, **25**, 345-353 (1948).
11. Spadaro, J. J., Persell, R. M., Reuther, C. G., Jr., Vix, H. L. E., Laborde, E. J., Latham, J. W., Jaeger, R. L., Pollard, E. F., and Gastrock, E. A., "Pilot-Plant Fractionation of Cottonseed. III. Process Development of Differential Settling." *J. Am. Oil Chem. Soc.* (In press.)
12. Vix, H. L. E., Spadaro, J. J., Murphey, C. H., Jr., Persell, R. M., Pollard, E. F., and Gastrock, E. A., *J. Am. Oil Chem. Soc.*, **26**, 526-530 (1949).

[Received October 10, 1949]

Countercurrent Distribution Studies on Fat Soluble Plant Pigments¹

CATHERINE R. LANCASTER, EARL B. LANCASTER, and HERBERT J. DUTTON, Northern Regional Research Laboratory, Peoria, Illinois²

PARTITION of immiscible solvents, the procedure used by Willstätter in his classical work on the separation of plant pigments (8), has been almost entirely replaced within the past two decades by chromatographic adsorption methods (6). With the development of systematic extraction procedures such as are made possible by the countercurrent distribution apparatus of Craig (2), the merits of solvent partition procedures again need to be re-examined. In the present paper the countercurrent distribution of a model system composed of chlorophyll-a, chlorophyll-b, and carotene is described. Separations obtained experimentally are discussed in relation to those predicted by the theory of Martin and Synge (4) and to their equations as modified herein. A simple nomograph has been constructed.

Materials and Methods

Chlorophyll-a and chlorophyll-b were isolated from frozen spinach by chromatographic adsorption on powdered sugar as described by Zscheile and Comar (9). Each pigment was further purified by two suc-

cessive adsorptions on powdered sugar columns. The effectiveness of the separation was then established by chromatographic and spectrophotometric methods. Carotene used in this work consisted of a mixture of alpha and beta (Eastman Kodak Co. 2702) and of crystalline α -carotene and crystalline β -carotene (General Biochemicals inc., No. 15849 and 16447).³

Countercurrent distributions were carried out in the 25-tube Craig analytical apparatus, using equal volumes of aqueous ethanol and hexane (90% ethanol and hexane, mutually saturated). On completion of the countercurrent distribution operation, both layers of each tube (8 ml. hexane and 8 ml. aqueous ethanol) were withdrawn into 25, 50-ml. volumetric flasks and diluted to volume with absolute ethanol. This dilution procedure resulted in a single phase solution for subsequent spectrophotometric determinations of chlorophyll-a, chlorophyll-b, and carotene. It was necessary to determine therefore the absorption coefficients of the pigments in that solvent mixture. The resulting equations for determining the concentrations of chlorophyll-a, chlorophyll-b, and carotene are similar in form to those of Comar and Zscheile (1):

¹ Presented at the fall meeting of American Oil Chemists' Society, November 2, 1949, in Chicago, Ill.

² One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.

³ The mention of these products does not imply that they are endorsed or recommended by the Department of Agriculture over others of similar nature not mentioned.