Pilot-Plant Fractionation of Cottonseed. II. Differential Settling[1,2]

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Introduction

A COTTONSEED fractionation method termed "Differential Settling" has been developed to produce, from either defatted or undefatted flakes, a fine cottonseed meal essentially free of oil, hulls, and pigment glands. The laboratory development of the process has been completed and the pilot-plant-scale development initiated. This process was developed to avoid difficulties encountered with the mixed-solvent fractionation flotation process reported for a laboratory scale (3) and a pre-pilot-plant scale (15).

Interest in methods of fractionation of cottonseed meal (3, 4, 13, 15) has increased as a result of the recent development and industrial adaptation of the solvent extraction process for cottonseed (6, 7, 9, 10, 12, 14) using primarily commercial hexane as the solvent. This increased interest is further enhanced by the fact that in the solvent extraction process, in contrast to hydraulic and screw pressing, the protein and pigment glands in the meal are not essentially altered. A solvent-extracted meal of high nutritional value and possibly suitable as a source of protein for industrial uses may be obtained by removal of the pigment glands. Furthermore, the pigment glands which are a by-product of the process show promise of having pharmaceutical uses (16).

The structure and behavior of pigment glands have been described elsewhere (3, 4, 15). For the purpose of the present report the high mechanical strength of the glands and their apparent detachment from the remainder of the kernel tissue must be emphasized, as must also the fact that the pigment glands are unaffected by a few solvents, such as some of the chlorohydrocarbons and the low-boiling petroleum cuts, but are ruptured rapidly in the presence of water and most organic solvents.

An ideal method for processing cottonseed would be one in which practically all the pigment glands would be removed intact from the meal and oil without being ruptured. The meal and oil would then be separated by conventional industrial methods, producing a meal of low oil content practically free of deleterious pigments, and an oil easily refined to a light color. Such methods may be based upon a difference in physical properties of the various components of the cottonseed.

The mixed-solvent flotation method (3, 15), which was the first to approach these ideal conditions, resulted from laboratory experiments to separate the pigment glands from surrounding tissue so that the pigments could be evaluated in the absence of other substances. It takes advantage of the difference in densities of the solid components, namely the meal, pigment glands, and hulls. Cottonseed flakes in a slurry of mixed solvents are disintegrated violently to detach the pigment glands from the meal tissue. Separation is then effected by flotation of the pigment glands (sp. gr. less than 1.36) in the solvent medium (sp. gr. 1.378) and settling of the meal and hulls which can be further separated by increasing the specific gravity to 1.45, inducing flotation of the meal and settling of the hulls.

Pre-pilot-plant development of the flotation process incorporating necessary modifications such as the use of 80-mesh and 230-mesh wet-screening resulted in the production of the first sizable quantities of defatted cottonseed flour essentially free of pigment glands (gossypol content as low as 0.006%) for nutritional (2, 5, 8), pharmacological (16), analytical (11), protein dispersions (1), fiber and adhesives, and for other investigations.

The following inherent disadvantages of the mixed-solvent flotation process led to the development of the "Differential Settling" process:

1. The high percentage of fine meal produced (up to 70% of 2-40 microns in size) during disintegration caused interference and entrapment in the separation operation. This difficulty is further enhanced by the slight overlap between the densities of the heavier glands and the apparent densities of the finer meal particles.

2. Owing to the small differences in specific gravities of the components to be separated, results obtained by centrifugal tests showed no improvement in the process.

3. Cost of the heavier solvent to obtain the proper gravity was about five times that of commercial hexane. Moreover, the heavier solvents (perchlorethylene, trichlorethylene, and carbon tetrachloride) are toxic to varying degrees.

4. The miscella (when using undefatted flakes as a feed) is a three-component system complicating the evaporation, stripping, and fractionation operations.

5. The relatively high temperature required in stripping increases the possibility of darkening the resulting oil (14).

6. The high temperatures required for desolventizing the meal has a denaturing effect on the protein in the meal.

Development of the Differential Settling Process

Preliminary study of the characteristics of the solid components of cottonseed (meal, hulls, and pigment glands) showed that the hulls were dense, solid particles with relatively smooth surfaces; the pigment glands were compact, ovoid-shaped particles with a granular appearing surface; and the fine meal (2-40 microns) had no definite shape, resembling a fluffy, feathery, amorphous material with a relatively large surface area per unit weight. The larger meal particles (over 40 microns) were irregular in shape, had a rough surface, and a relatively small surface per unit weight.

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

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The slow settling characteristics of the fine meal particles noted during the flotation experiments suggested a possible separation by frictional resistance of the liquid medium.

The effectiveness of the frictional resistance of the liquid medium (commercial hexane) to the fine meal particles was attributed to two main factors: the texture of the particles and their total area per unit weight. Experiments showed that the fine meal particles could be separated from the other solid components in the slurry by taking advantage of this resistance of the liquid medium to these smaller particles to produce a slower settling rate for the latter as compared to that of the hulls, glands, and larger meal particles.

Figure 1 shows three slurries of cottonseed flakes in commercial hexane in three different stages of settling. The first slurry, settled overnight, shows all solids settled in layers: hulls, coarse meal, glands, and fine meal, respectively. The second slurry, settled for 20 minutes, shows the fine meal in suspension and the remainder of the solids settled. The third slurry, settled only a few seconds, shows all of the solids in suspension, except for a few hulls. Figure 2 shows an enlarged view of the components of cottonseed in the slurry settled overnight.

The hulls, having a specific gravity above 1.45, settled very rapidly; the whole pigment glands, having a specific gravity of below 1.36, settled slightly more slowly than the hulls; but the meal particles, which have a specific gravity between that of glands and hulls (1.41 to 1.44), settled more slowly than either the hulls or glands. Settling rate of the meal particles is chiefly dependent upon their texture and particle sizes. Slurries prepared with either defatted or undefatted flakes settled similarly.

Further experimentation showed that:

1. Time required for complete settling of hulls and practically complete settling of glands could be established for various conditions.
2. To detach pigment glands from 92 to 95% of the meal tissue in a slurry containing a ratio of 1 gram of cottonseed flakes (solid basis) to 1.5-1.8 ml. of solvent (such as commercial hexane), a disintegration (13) is necessary which reduces 70% of the meal tissue to a size of 2-40 microns.
3. Over 90% of these fine meal particles (2-40 microns) were in suspension at the end of the settling time, the amount of pigment gland fragments remaining in suspension being negligible.
4. Meal particles over 40 microns settled at rates intermediate to those of pigment glands and hulls.
5. With undefatted cottonseed flakes the oil content of the slurry may go up to at least 30% by weight without appreciably affecting the yield of fine meal by the increase of viscosity.
6. The solvents exceptionally suitable for the process are those in the low specific gravity range of 0.67 to 0.78, such as commercial hexane. However, any inert solvent of low viscosity with a specific gravity of below 1.25 or above 1.55 will affect the separation. Solvents that can be used are: petroleum ether, solvent naptha, benzene, and such commercial cuts as normal heptane, normal pentane, normal hexane, cyclohexane, etc.
7. The settled or coarse meal fraction can be redisintegrated and resettled to increase the yield of fine meal recovery.

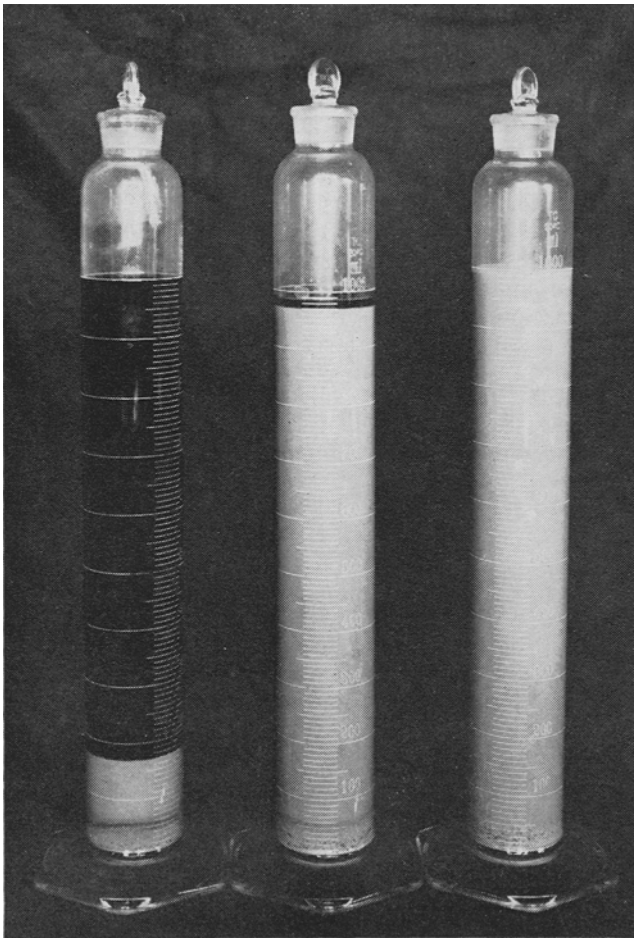

FIG. 1. Slurries of cottonseed flakes in different stages of settling.

On the basis of the above, two laboratory methods of Differential Settling were developed. A brief description of each follows:

Tube Differential Settling. A given quantity of cottonseed flakes in a solvent slurry is disintegrated sufficiently to pass an 80-mesh screen. Slurry is then diluted to a predetermined concentration by the addition of solvent, and the mixture settled for a period of time determined by observation of a test sample. The suspension of fine meal is then separated from the sediment by syphoning or decanting; and the fine meal subsequently recovered by filtering, washing with solvent if undefatted flakes were used, and desolventization.

Centrifugal Differential Settling. The principles of centrifugation were investigated. Preliminary work showed that by using a relative centrifugal force of about 50 R.C.F. and a time period normal to the operation of industrial continuous centrifuges, a separation similar to tank differential settling could be obtained. Centrifugal tests using slurries similar to those prepared for tube differential settling showed that the fine meal remained suspended in the effluent and the hulls, glands, and larger than 40-micron meal particles packed in the sediment. Recovery of the fine meal could be attained as in tube differential settling.

In addition to recovery of fine meal by filtering and washing for both methods of differential settling,

the use of centrifugal forces in the range of 2,000 R.C.F. was investigated. This was found to separate 95% of the fine meal when using undefatted material and 98% of the fine meal when using defatted material in original disintegration.

The settled fraction or sediment from either method can be redisintegrated and resettled or centrifuged to enhance the total yield of fine meal. Due to the nature of the physical characteristics of the solid components, the meal tends to disintegrate more readily than the hulls or pigment glands thereby making a substantial recovery possible.

The sediment from this second processing consists of hulls, pigment glands, and coarse meal and is a potential source of both pigment glands and a feedstuff. If pharmaceutical uses for the pigment glands are realized (16), the pigment glands can be separated and purified as described in the pre-pilot plant mixed solvent flotation process (15). Further investigation is required if the material is to be used as a feedstuff.

The fine, essentially pigment-gland-free cottonseed flour produced by differential settling is equal or superior in quality, judged by gossypol content, to that produced by the pre-pilot plant mixed solvent flotation process. It is therefore apparent that this meal will have at least as high a nutritive value and as much adaptability for industrial use.

Experimental Data

Some preliminary experiments had shown that the particle size of fine meal obtained in differential settling was in the range of 2 to 50 microns; that with good disintegration a yield of 70-75% of fine meal could be obtained whereas insufficient disintegration resulted in correspondingly poor yields; and that with similar settling periods the portion of fine meal suspension taken from the top of the settling tube contained less gland material than the portion from the bottom of the tube.

A systematic series of experiments was then conducted to determine the effects of varying the time of settling and the per cent solids and to determine the difference in behavior of slurries prepared from defatted and undefatted flakes.

Effect of Conditions on Yield. In studies of tube differential settling (experiments 6 through 13) two types of slurries were used: one prepared by mixing 4.8 pounds of undefatted flakes with one-half gallon of commercial hexane; and the other, by mixing 5.5 pounds of dried hexane-extracted flakes with 1 gallon of commercial hexane. Slurries were disintegrated in a modified commercial-type blender and then wet-screened through an 80-mesh screen to remove the excess coarse meal and hulls. A 30-mm. glass tube was used for all samples. Only one removal of fine meal suspension was made in each case, the fine meal adhering to the coarse particles being reported as sediment.

The results in Table I show the effects of time settled, per cent solids, and per cent oil on the yield of fine, gland-free meal obtained from the meal suspension in tube differential settling. It is apparent that the yield varies inversely with the time of settling. In increasing the settling time from 60 to 135 minutes a decreased yield of 3.4 to 11.9% is noted for defatted slurries and 3.5 to 8.1% for undefatted slurries. Small gland fragments present in the fine meal were negligible. The effect of total solids, on yield, is negligible except for the 135-minute defatted samples with 10.9% solids which showed a yield of 6.5% greater than the 5.0% solids sample. The presence of oil has little or no effect on the yield. For example, the 60-minute sample of undefatted 5.2% solids slurry and the 60-minute sample of defatted 5.0% solids slurry yielded 77.5 and 77.6% fine meal, respectively. Consequently, either defatted or undefatted cottonseed flakes can be used in the process.

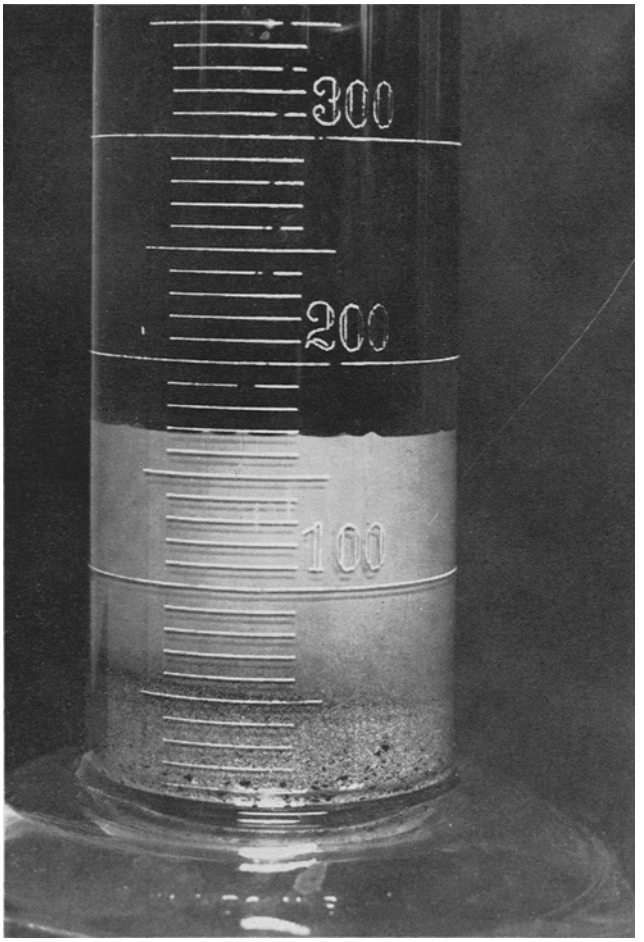

FIG. 2. Enlarged view of cottonseed slurry settled overnight.

In studies of centrifugal differential settling (experiments 14 through 19), tests were made using a laboratory batch-type centrifuge. Slurries prepared with defatted and undefatted flakes were the same as those used in tube differential settling. Samples of approximately 70 ml. each were centrifuged at 50, 75, and 100 R.C.F. (Relative Centrifugal Force). Pigment glands, hulls, and coarse meal settled out. The fine meal remaining in suspension was recovered by centrifuging at 2,000 R.C.F.

The effect of relative centrifugal force and of type of slurry is shown in Table II. For both types of slurries the per cent yield of fine meal increases with a decrease in centrifugal forces. At 100 R.C.F. the yield is appreciably lower for the defatted than for the undefatted material; and at 50 R.C.F. the yields are comparable. It is apparent therefore that the viscosity of the slurry has some effect on the yield of fine meal at the higher R.C.F. However, since the

TABLE I

Effect of Conditions Upon Yield of Fine, Gland-Free Meal

Experiment No.	Constant Conditions				Variables	
	Type Slurry	Depth Slurry	Solids	Oil	Time Settled	Yield
			pct.	*pct.*	*min.*	*pct.*
6	Defatted	24 inches	10.9	None	60	75.6
7	Defatted	24 inches	10.9	None	135	72.2
8	Defatted	24 inches	5.0	None	60	77.6
9	Defatted	24 inches	5.0	None	135	65.7
10	Undefatted	24 inches	13.1	31.0	60	74.0
11	Undefatted	24 inches	13.4	31.6	135	70.5
12	Undefatted	24 inches	5.2	11.5	60	77.5
13	Undefatted	24 inches	5.2	11.5	135	69.4

purity (per cent pigment gland in meal) of the fine meal for both 50 and 100 R.C.F. were the same, zero per cent, as shown in Table III, operation at R.C.F. of even lower than 50 R.C.F. would be desirable. On the basis of the above, the effect of oil in the slurry would be negligible and the yield higher for any proposed process at the preferable lower R.C.F.

TABLE II

Effect of Relative Centrifugal Force and Type of Slurry on the Yield of Fine, Gland-Free Meal

Experiment No.	Constant Conditions			Variables	
	Solids	Oil	Type Slurry	R.C.F.	Yield
	pct.	*pct.*			*pct.*
14	12.4	29	Undefatted	50	65.1
15	12.4	29	Undefatted	75	59.2
16	12.4	29	Undefatted	100	58.9
17	10.7	None	Defatted	50	63.0
18	10.7	None	Defatted	75	52.0
19	10.7	None	Defatted	100	44.9

Product Quality. The percentage of total glands in both the fine meal fractions and in the sediment fractions of a cottonseed slurry fractionated by the two methods of differential settling are shown in Table III, experiments 20 through 23.

The undefatted slurry was prepared from flakes disintegrated in commercial hexane and, as in the previous experiments, wet-screened through 80-mesh for removal of excess hulls and coarse meal.

For the centrifugal differential settling tests two samples of the resulting slurry were centrifuged at 50 and 100 R.C.F., respectively, followed by centrifugation of the suspension of fine meal at 2,000 R.C.F. to recover the fine, gland-free meal. To determine the per cent pigment glands, each fraction (the original sediment and the fine meal) was diluted with a mixture of commercial hexane and perchlorethylene to a specific gravity of 1.378 and allowed to settle for separation of glands. The glands were recovered, washed in commercial hexane, vacuum-dried, and weighed. The approximate per cent purity of the glands was microscopically determined, and on this basis the per cent of the total glands in each fraction determined. The weight of the meal in each fraction was determined by filtering the slurries after removal of glands, washing with hexane, and drying.

For the tube differential settling tests two samples of the original slurry were settled for 60 and 135 minutes, respectively. At the end of the settling periods the meal suspensions were withdrawn, filtered, washed in commercial hexane, and vacuum-dried. These meal and sediment fractions were processed as in the centrifugal experiment for separation and determination of the per cent pigment glands. Table III presents the yields of fine meal, essentially pigment gland-free, and the percentage of total glands in the fine meal and in the sediment.

The results in Table III show that by both tube and centrifugal differential settling a fine meal fraction practically free of pigment glands can be obtained. Although the yields of fine meal obtained with centrifugation in the present work are lower than those obtained in tube differential settling, comparable results can be obtained by using a lower R.C.F. The enriched gland fraction produced by both methods of differential settling can be further processed for additional recovery of fine meal fraction and for the recovery of the glands if desired.

TABLE III

Centrifugal and Tube Differential Settling Data

	Differential Settling			
	Centrifugation		Tube	
Experiment No.	20	21	22	23
How processed	50 R.C.F.	100 R.C.F.	60 min.	135 min.
Wt. meal in final effluent, gms.	0.205	0.19	.....	.....
Wt. solids in fine meal cake	3.43	2.48	22.13	20.18
Wt. solids in sediment, gms.	2.10	3.08	7.54	9.54
Total wt. solids recovered, gms.	5.735	5.75	29.67	29.72
Wt. glands in meal cake, gms.	0.000	0.000	0.002	trace
Wt. glands in sediment, gms.	0.10	0.16	0.86	0.86
Total wt. glands recovered, gms	0.10	0.16	0.862	0.86
% glands in original solids	1.74	2.78	2.89	2.89
% glands in sediment	4.76	5.20	11.4	9.0
% of total glands in fine meal	0.00	0.00	0.23	0.00
% of total glands in sediment	100.00	100.00	99.77	00.00
% of total meal in final effluent	3.61	3.40	.....	.....
% of total meal in meal cake	60.9	44.4	76.8	69.9
% of total meal in sediment	35.5	52.2	23.2	30.1
% total solids in slurry	12.0	12.0	12.0	12.0

Commercial Process Possibilities

These results have warranted the development, engineering, and construction of the full-scale pilot plant for the purpose of determining the applicability of the differential settling process to commercial production on an economical basis. Equipment for the various unit operations was selected after consultation with many manufacturers and their engineers. The pilot-plant was designed and constructed for maximum flexibility to permit a thorough chemical engineering study of the following unit operations and processes: material preparation, disintegration, separation, filtration, desolventization, and oil recovery. Continuation of this fractionation study will further determine whether the process can be used at a reasonable cost to obtain both a cottonseed oil comparable in grade (if undefatted flakes are used) to that produced by other methods, and a light-colored meal suitable as a high-grade protein feed and as a source of light-colored protein for industrial utilization. Pigment glands will be a by-product of the process.

The differential settling process can possibly be used in conjunction with continuous solvent extraction systems in either of two ways. One method would be to feed defatted, solvent-damp cottonseed meal from a solvent extraction plant directly into the disintegration equipment. Meal would then be disintegrated, differentially settled, and the resulting products recovered as previously outlined. By this procedure desolventization would be done only once —at the conclusion of the entire process. Furthermore, since additional extraction of oil would take place during the subsequent disintegration and settling steps, extracted flakes could be fed to the disintegrator at an oil content of about 3-4% instead of

1% or less, as is the aim in present industrial solvent extraction of cottonseed. The capacity of the extractor would thus probably be doubled since the last 3 or 4% of oil in flakes is removed during the diffusion stage, the most lengthy, difficult, and expensive part of the extraction. The weak miscella from the differential settling process containing the 3 or 4% oil could then be used in place of solvent in the solvent extraction plant. The resulting savings could be applied to the cost of the differential settling process when combined with an existing solvent extraction installation. The enhanced value of the meal obtained by the use of the combined process should result in additional economic gain.

The second possible commercial process is one in which undefatted flakes would be fed directly to the disintegrator. After the usual disintegration, settling, and product recovery steps, the emerging meal containing 6 to 10% oil could be extracted by solvent-washing, the meal recovered by centrifugation, and the oil and solvent recovered in the conventional manner. Fresh solvent would be used on this meal permitting efficient extraction, and the resulting miscella could in turn be used in preparing the slurry for the original disintegration.

Summary and Conclusions

A fractionation process termed "differential settling" has been developed to produce a cottonseed meal substantially free of oil, pigment glands, and hulls from either defatted or undefatted flakes. The development of the process was initiated during the work on a mixed solvent flotation method of fractionation which showed several inherent disadvantages. The investigation of the flotation method had shown that cottonseed meal essentially pigment-gland-free (gossypol content as low as 0.006%) had a high nutritional value and was a source of protein for industrial uses. The meal produced by the differential settling process has as low a gossypol content as the meal produced by the flotation principle and overcomes the disadvantages.

The advantages of the differential settling process are as follows: 1. It requires only one solvent and is more readily adaptable to present solvent-extraction plants than a process using mixed solvents—the preferred solvent, commercial hexane, is commonly used in practically all vegetable oil solvent-extraction plants, and is much cheaper and less toxic than the high specific gravity solvents, such as tetrachlorethylene, used in the mixed-solvent method; 2. fractionation of solvents and control of specific gravity is eliminated; 3. power consumption for the disintegration and centrifugation steps is less since a lighter weight slurry is used; 4. less total heat and lower temperatures are required in desolventizing both the oil and meal, thereby decreasing the possibility of darkening the oil and denaturing the protein of the meal; and 5. the time required for the separation of the slurry into the fine meal suspension and sediment is considerably less than required for the separation of glands from the meal fraction in the mixed-solvent flotation process.

Two methods, centrifugal and tube settling, of the differential settling process have been developed which show promise for commercial separation of the components of cottonseed, namely meal, pigment glands, hulls and oil. Removal of the pigment glands from the coarse fraction or sediment will depend on pharmaceutical or other uses developed.

Commercial possibilities of using the differential settling process in combination with present cottonseed solvent-extraction processes have been outlined.

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Studies on Candelilla Wax. I. Its n-Acids and n-Alcohols

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DESPITE its wide technological use candelilla wax has not been very intensively investigated. A need exists for a re-examination of published work—some of it could profitably be reviewed, particularly the statement that it contains a lactone (6)—and the application of some of the newer analytical techniques to the problem of filling some of the obvious gaps in our knowledge of the composition of this substance. That which follows is the first of several communications in which are reported results of such a study.

This plant wax covers the entire surface of several species of Euphorbiaceae, *Pedilanthus pavonis* and *Euphorbia cerifera*—the latter is probably *E. antisyphilitica* (4) of an earlier day—that grow in the semiarid regions of northern Mexico and southern Texas. In form they are leafless, reed-like stems, one to three feet high and from one-eighth to five-sixteenths inch in diameter. Sometimes as many as 100 of these spring from a single root. The plants grow without benefit of cultivation, interspersed with other desert flora, and must be harvested by hand.