

of expeller used, a considerable amount of shell in the meal is essential for efficient expelling.

Bags of nuts with hulls removed but with the shells intact showed no deterioration after two months' storage in a well-ventilated shed.

Acknowledgments

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Analysis of the Fruit of the Chinese Tallow Tree in Texas*

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THE Chinese tallow tree, *Sapium sebiferum*, of the Euphorbiaceae family, was introduced into Texas about 1910 by Edward Teas, although by 1848 many of the trees were growing in places in Florida, Louisiana, and South Carolina (6). The tree thrives on various types of soils but requires a somewhat semi-tropical climate. Experience has shown, for example, that in and about Houston they thrive whereas many other trees planted there fail to establish themselves satisfactorily.

Sapium sebiferum is a deciduous tree that attains a height of 40 feet or more. The tree branches freely, and the branches have a tendency to be pendant. The leaves are alternate and have slender petioles. The leaves are dark green on the upper surface and a lighter green below. In the fall the leaves are brilliantly colored, usually red.

The small flowers are 4-6 mm. long and are without petals and are unisexual. The male flowers are produced in groups of threes throughout the flexuous spike-like inflorescence. The female flowers are solitary and are usually found in the lower portion of the inflorescence.

The fruit is a 3-lobed capsule and is about 15 mm. in diameter. It is somewhat fleshy when green, but dry when ripe. When the capsule dehisces, the trees are defoliating or are in the process of losing their leaves. There is one large, white seed in each cavity of the capsule. The mature seed occupies practically all of the space in the cavity. The seed adheres to the placenta long after the capsule has opened.

The seed is small and covered with a white aril-like coating. When covered with the wax it is somewhat triangular, convex on the outer face, and flattened on the two inner faces. When the wax is removed, the seed is found to be orbicular in outline, but truncate on the micropylar end. It is somewhat flattened. The testa is brown and very hard. There is no caruncle present. In the mature seed the nucellus is papery and the endosperm abundant. The small embryo occupies the central axis of the seed.

Since the tallow tree thrives in parts of Texas, these investigations were carried out further to characterize the fruit and products obtained from it. The methods

of the Association of Official Agricultural Chemists were employed in the analyses.

The composition of the seed and three of its parts are reported in Table I.

TABLE I.

	Whole seed	Extracted meats	Extracted hulls	Fiber
Moisture and volatile.....	4.58	7.62	7.95
Ash.....	1.74	6.78	2.79	7.86
Protein (N × 6.25).....	11.27	76.43	2.78	5.22
Crude fiber.....	4.90	31.98
N.f.e. (by difference).....	4.27	54.94
Insoluble SiO ₂045
Soluble SiO ₂020
Total SiO ₂065	1.21
Potassium.....943	.016
Calcium.....27	.43
Magnesium.....875	1.06
Iron.....032	.011
Phosphorus.....	1.60	.073
Nitrogen.....	1.75	12.23	.44	.84
Tallow and oil (ether extract)...	45.42
Hull (fiber, shell, tallow).....	68.52
Kernel (meat, oil).....	31.48
Shell and kernel.....	67.97
Seed-coat (fiber, tallow).....	32.03

The following data were also obtained: tallow in seed-coat 74.75%, fiber in seed-coat 25.25%, tallow in hull 34.94%, oil in the kernel 64.10%. The percentage composition calculated from these data: oil 20.3, tallow 23.9, meat 11.3, fiber 8.1, shell 36.4.

A number of investigations have been reported in the literature on the characteristics of the oil and tallow and the fatty acids obtained from both the oil and tallow. The two most significant of these for the oil are those by Jamieson (3) and by Jamieson and McKinney (5). The most significant results on the tallow are reported by Jamieson (4) and by Hilditch and Priestman (2). There is little agreement among the data from other investigators. This is due to the difficulties encountered in obtaining samples of one component that are uncontaminated by the other.

For experimental purposes uncontaminated samples of each may be obtained by cutting the individual seed and picking out the kernel. The tallow and oil can then be obtained without contamination by extracting these two fractions. Oil and tallow samples numbered four were obtained by this method.

The oil and tallow are obtained in China by suspending the seed in water and heating this mixture

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TABLE II.

Tallow

Sample No.	Gm. of seed	Solvent*	Gm. of tallow	Iodine No.	Acid No.	Sapon. No.	Soluble acids	Hehner No.	m.p.	Softening pt.
4.....	600	1	136.	16.3	11.01	218.5	54.4	52.5
9.....	600
9.1.....	1	33.5	10.7	11.92	221.4	3.41	54.1	52.1
2.....	1	25.5	55.6	52.5
3.....	1	17.5	10.5	11.98	221.5	2.40	91.92	56.8	52.3
4.....	1	14.0	58.0	52.7
5.....	1	20.0	10.4	56.3	53.2
6.....	1	12.0	222.2	2.91	55.3	52.8
7.....	1	5.5	57.2	56.1
8.....	1	4.0	58.1	56.3
9.....	1	1.0	58.4	52.0
10.....	1	4.5	11.7
11.....	3	19.1	63.0	59.9
12.....	3	14.4	61.7	59.4
13.....	2000	562.
1.....	2	164.	24.8	6.65	51.8	48.0
2.....	2	134.	25.1	56.2	53.2
3.....	2	104.	56.8	53.4
4.....	2	70.	24.1	6.22	57.8	55.9
5.....	2	35.5	57.5	53.1
6.....	2	18.5	58.8	55.3
7.....	2	6.0	20.9	56.0	55.6
8.....	3	4.0	20.8	56.2	54.3
9.....	3	1.0	26.4	55.5	55.0
10.....	4	2.0	34.0	53.3	51.5
11.....	6	2.0	34.3	51.0	46.9
12.....	5	2.0	48.6	53.0	52.6
13.....	5	2.0	49.4	46.7
15.....	5	2.0	48.6	41.0
17.....	5	4.0	121.3	47.5	46.7
18.....	5	6.5	168.6	40.9	34.3

*Solvents: 1) Barnsdall E5A; 2) Solvesso No. 1; 3) benzene; 4) ethyl ether; 5) mixed (Solvesso No. 1, chloroform, ethyl ether, methyl alcohol, and benzene); 6) benzene.

(6, 8). The melted tallow is skimmed from the surface of the mixture. The shells of the seed are then broken by grinding them between stones and the shells and kernels are separated in winnowing machines. The kernels are ground and the oil is obtained by pressing the hot material. A low-grade oil is obtained by this process.

Quinby (7) obtained a United States patent on a process involving the heating of the seed in water and, finally, in alkali to remove the last visible traces of tallow. The oil is then extracted from the ground seed by solvents. Davis and Fleming (1) removed most of the seed-coat by first boiling the seed in alkaline solution. They were then stirred in hot alkaline solution to remove the last traces of the seed-coat.

In the present investigation it was found that the seed-coat could be removed by suspending the seed in water heated from 60 to 70°C. and stirring the mixture. The fiber and tallow were recovered by filtering off the water and then extracting the dried material with mixed solvent. Approximately 90% of the tallow was recovered in this process which was employed with sample 16. It was also found that the tallow could be removed by repeated extractions of the whole seed with solvents. The lowering of the melting points and softening points of fractions 13.10 through 13.18 indicated that some oil was extracted from the kernel but this was not extensive. The fiber was removed

from these extracted seed by suspending them in water and stirring the mixture. The dried seed were cracked and the meats were separated from the shell. The oil was then extracted from the ground meats with solvents. The seed of sample 9 were extracted and, without removing the fiber, they were ground and extracted. The extracted seed of sample 20, with fiber, were broken and after the meats were separated from the hulls, the ground meats were extracted. Sample 15 was prepared by removing the tallow with solvents and the fiber was removed by stirring the extracted seed in water. The dry seed were passed through crushing rolls to break the shell and the crushed seed were separated into two fractions in an air separator. The smaller fraction, the kernel concentrate, consisted principally of kernels together with a small amount of shell. The larger portion, the shell concentrate, was principally broken shells, but it also contained a large quantity of kernel. These two fractions were ground further and the oil was obtained by four extractions with "Skellysolve A." The solvent was removed from the oil by distillation and sparging with carbon dioxide. The color of the oil from the kernel concentrate was lighter than that obtained from the shell concentrate and the former fraction of oil was characterized as indicated for sample 15 in Table III. Test tube heat bodying tests showed that the oil gelled in four hours as compared

TABLE III

Oil

Sample No.	15	4	13	16	20
Color (Gardner 1933 Standards).....	6+
Viscosity.....	0.5 poises
Appearance.....	slightly cloudy
Refractive index at 25°C.....	1.4827
Specific gravity at 15°C./15°C.....	0.9413
Acid value.....	10.0	0.14
Saponification value.....	203.9	202.0
Unsaponifiable.....	1.03%
Volatile.....	0.0
Acetyl value.....	7.5
Iodine value.....	183.3	181.0	187.3	183.4	185.6
Diene value.....	none
Qualitative break test.....	negative
Ash.....	0.019%
Soluble acids.....	3.23
Hehner No.....	88.89

TABLE IV

	Reichert-Meissl	Polenske	Unsaponifiable
Tallow C15.....	.42	.53	.73
Tallow 16.....	.48	.46	.88
Tallow 20.....	.48	.45	.82
Oil 16.....	.66	.10

to five and one-half hours for a regular alkali refined linseed oil. The color, after two hours at temperature, was 12+ and that of alkali refined linseed oil was 10+. Drying tests were made by measuring the gain in weight and the results indicate that this oil dries similarly to a high quality linseed oil.

The whole seed, the extracted meats and hulls, and the fiber of the Chinese tallow seed were analyzed.

Six samples of tallow and five samples of oil were characterized as shown by the data recorded in Tables II to IV.

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Lipids of the Cottonseed

I. Some Quantitative Observations on Yield

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EVIDENCE has been obtained that in the extraction of cottonseed with several organic solvents, the composition of the extract obtained with a given solvent will vary from seed specimen to seed specimen, and the composition of the extract obtained with a given seed specimen is dependent on the nature of the solvent used. The quantity of material extracted with a given specimen is also dependent on the nature of the solvent, and it is suggested that the selection of any particular solvent as the standard or "official" solvent to be used in the assay of oil with cottonseed is arbitrary.

At the time of the adoption of the refractometric method for oil assay with linseed oil (1) as an official method by the Association of Official Agricultural Chemists, it was suggested that the procedure might be adapted to the determination of the oil content of other oil-bearing seeds. In the course of the investigations carried out in this laboratory it has been found that the refractometric method of oil assay may be used with cottonseed, but the results obtained for the quantity of oil in any given specimen are not in agreement with those obtained on the extraction of the seed with diethyl ether or petroleum ether, as recommended in the official methods of the Association of Official Agricultural Chemists and the Cottonseed Crushers Association respectively. In turn, the determinations of the oil content of cottonseed with these solvents are not in agreement with each other.

The refractometric scheme for oil assay with vegetable oil seeds involves the grinding of a weighed quantity of the seed in the presence of a fat solvent of low volatility which differs sufficiently in its index of refraction from that of the vegetable oil that small quantities of the oil in the solvent may be determined by means of index of refraction measurements. The grinding is effected in the presence of a prescribed quantity of the solvent, washed sea sand, and a small quantity of Na_2SO_4 (anhydrous). After sufficient grinding, the ground sand, the seed residue, and the

salt are removed by filtration, and the index of refraction is determined with a drop of the filtrate. The quantity of oil per unit weight of seed is then determined by reference to the proper calibration curve.

It was not feasible to use the mixture of 1-bromonaphthalene and chloro-naphthalenes, as recommended in the official method, as these substances have indices of refraction beyond the range of the instrument available to us. A mixture of 1-bromonaphthalene (Eastman) and ethyl benzoate (redistilled to constant index of refraction, $n = 1.50529_D^{20}$) having an index of refraction of 1.61128_D^{20} was adopted instead as the nonvolatile solvent for use in the determinations reported in this communication. The grinding was effected with a power-driven mortar and pestle grinder, and the requisite time of grinding, etc., were determined to yield maximum quantities of oil. The standard volume of the solvent was taken as 5 ml., the weight of seed as 0.5 g., the weight of sea sand as 2 g., the weight of sulfate as 2 g., and the time selected for the grinding was 20 minutes.

Two different procedures were used in determining the oil content of the seed specimen with the data obtained from the refractometric method of analysis. The one procedure involved the use of seed samples which were prepared by using two aliquots of seed, one of which had been exhaustively extracted with petroleum ether with a Soxhlet extraction apparatus. These aliquots were mixed in the desired proportions. The calibration curve in Figure 1 was prepared by plotting the index of refraction of the filtrates, as indicated above, from these several samples against the oil contents of the samples, as determined on extraction with petroleum ether. A linear relationship between the oil contents of the samples and the refractive index was observed. This fact in itself would indicate that the refractometric method of oil assay is applicable to cottonseed, if one assumes uniform composition of the lipid-soluble fraction from specimen to specimen. One may refer the indices of refraction obtained with his