containing safflower oil showed a slightly greater tendency to chalk but showed slightly less checking and cracking than the linseed oil control paints. In the red oxide series the results showed an opposite trend.

Outdoor exposures on alkyd varnishes and enamels, as well as phenolic varnishes showed no definite differences between safflower and linseed oils.

Commercial Production

Soon after the potential value of safflower oil as a paint vehicle had been pointed out, Harold Meggitt Ltd., of Sydney, Australia, commenced experiments on the commercial production of the oil, using Indian seed. The plant used is a batch-type solvent extraction unit, and I am indebted to Clive Meggitt for the following comments on his experience in the handling of safflower seed:

From the preliminary samples examined, it seemed likely that the safflower seed would treat satisfactorily in the plant without decortication. Initial extraction figures showed a residual oil content in the meal of 1 to 1.5%. Since the whole seed analysis showed an initial oil content of 27.5%, the result was considered satisfactory and economically sound. The oil was of excellent colour, had a low acid value, and was completely free from "foots."

The raw oil was found to refine very well indeed by the caustic soda process, with very small losses.

There was some doubt as to the market value of the meal by reason of the relatively high percentage of coarse hull, but despite this it was decided to enter full-scale production, and the results compared favourably with those of the initial trials. After some early rejection of the meal because of the hull, a few successful feeding tests established its value, and all meal since produced has been readily absorbed at 1 £ (A) per ton under current linseed meal prices. All users have reported favourably.

Summary

A brief account is given of experiments with safflower oil in Australia. Under practical conditions, the drying power of safflower oil equals that of linseed oil. The non-yellowing properties of the former render it superior to the latter as a vehicle in coatings for interior decoration and in stoving enamels. In the heat polymerization of safflower oil, temperatures 10° to 15°C. higher than those normally employed for linseed oil are recommended. During three years of outdoor exposure trials, paints based on safflower oil have performed at least as well as similar paints from linseed oil. The commercial production of the oil by solvent extraction presents no difficulty. Decortication prior to extraction is not necessary. The resulting oil has a very good colour and is free from "foots." On alkali refining, losses are very small. Notwithstanding the high hull content of the meal, it has proved valuable as a stock fodder.

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Utilization of the Seed of the Chinese Tallow Tree

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THE Chinese tallow tree, *Sapium sebiferum*, produces seeds which are unusual in that they contain both a highly saturated fat and a highly unsaturated oil. The saturated fat and unsaturated oil are physically separated in the seed and may be isolated independently. The tree was introduced into this country over a hundred years ago and can be found scattered in several of the southern states, particularly in the Houston, Texas, area. The principal value of the tallow tree in this country is ornamental although consideration is being given to the utilization of the seeds as a source of vegetable tallow, drying oil, and protein feed (12).

The seed, which has been described in detail by Kaufmann and King (10), Potts (12), and Potts and Bolley (11), is the size of a pea. An outer coating of vegetable tallow and fiber cover a hard, brittle shell which contains a small embryo and abundant endosperm. The endosperm is composed of a high protein meal and a drying oil (stillingia oil). In China the tallow is separated by placing the seed in hot water, whereby the tallow melts and floats to the surface, or by melting the tallow with steam and collecting it when it drops off (10). A convenient method used by various modern investigators (5, 6, 11) is to solvent extract the tallow from the seed. The tallow still adhering to the seed may be then removed by an alkali treatment (13). The rather thick hard shell prevents extraction of the oil in the interior. After removal of the tallow the seed is crushed and the stillingia oil obtained by pressing or solvent extraction. The composition of the tallow (4, 10), protein (6), and oil (5, 8, 9) has been established.

In the present study various processing procedures were tried that could be used with the addition of a minimum of equipment by oil mills at present processing other seeds. The various products prepared by the processing study were evaluated. Very little work was done with the tallow other than to observe that it appeared to be of good quality and similar to that which has been described in the literature. The protein was prepared and compared to soybean, peanut, linseed, and casein. Stillingia oil has had some limited use as a drying oil. However a complete study of its characteristics as a paint and varnish oil apparently have not been reported. We have therefore subjected the oil to an evaluation study, using a procedure previously described (1). The same procedure has been used to characterize a number of other drying oils (7).

Processing of Seeds

Approximately 200 pounds of Chinese tallow tree seeds were received from W. M. Potts of the Agricultural and Mechanical College of Texas. These were obtained from trees in the Houston, Texas, area. The seeds were examined in the laboratory by standard methods and found to be composed of:

Fiber-free tallow	23.4%
Fiber from the tallow	6.6%
Shell	41.2%
Embryo	1.1%
Oil-free endosperm	10.7%
Stillingia oil	17.0%

These results are in fair agreement with those obtained by other experimenters (10, 11, 12).

The seeds as received contained some twigs, dirt, and other trash. This was removed by screening through a one-half-inch screen to remove the large twigs and trash and then passed through an air separator to remove the dirt and small twigs. This clean seed was the starting material for the experimental work and all yields are based on it.

In the first large scale experiment the tallow was removed from the seed by solvent extraction. The "tallow-free seeds" were then crushed and the oil was solvent extracted. However the oil was of a poor quality since it contained tallow which had not been completely removed by the first solvent extraction. The oil free meal was air separated into shell and kernel. It was noted that the kernel was contaminated with the fiber. This experiment showed the desirability of complete coating removal prior to processing the seed for the drying oil and high protein kernel.

Tallow and fiber was completely removed by stirring the seed in warm water to disintegrate the coating. The slurry was screened, retaining the seeds on the screen while the disintegrated coating was washed through the screen. The slurry which contained tallow, fiber, and soluble constituents was filtered on a vacuum filter to recover the solid materials which were dried at 212°F. As a result of this procedure the seed was divided into 59% decoated seeds, 31% tallow and fiber, and 10% processing loss and soluble constituents. The tallow was removed from the fiber by solvent extraction (23% of the seed) and by expression in a Carver laboratory cage press (20% of the seed). Attempts were also made to separate the tallow and fiber by saponification and steaming; however the latter two methods gave very low yields.

A good grade of tallow may be prepared directly from the seed by solvent extraction with hot hexane. There are indications that benzene, dichloroethelene, and carbon tetrachloride may be better solvents. After extraction of the major part of the tallow by solvents, the remaining tallow and fiber is removed by water treatment as previously described. Attempts to decoat the tallow free seeds mechanically, using a rotating wire brush were not successful. The treatment of 83.5 pounds of seed by solvent extraction followed by water treatment gave 17.5 pounds of high grade tallow (21.0%), 54.3 pounds of decoated seed (65.1%), and 11.7 pounds of fiber, tallow and loss (13.9%). Thus by either this method or the one previously described a satisfactory tallow and decoated seed is obtained.

The next step in the process was crushing the decoated seed and solvent extracting the stillingia oil. The nuts would best be crushed by rolls, but since this equipment was not readily available, a large coffee mill was found to be satisfactory. The crushed decoated seed was readily extracted with hexane. Most of the extractions were carried out in a five-gallon pressure filter. The oil was recovered from the hexane by vacuum distillation of the miscella in a stainless steel still with the last traces of solvent removed by sparging the hot oil with carbon dioxide gas. In this manner an average of 20.0% oil on the total seed basis was obtained. The various oil fractions were combined and used for evaluation as described later. The crushed shell and meal contained less than 1% fat and oil and amounted to 45.1% of the original seed.

The de-fatted crushed seed (extraction residue) was easily air-separated into a high protein flour and crushed shell. The flour was white and by nitrogen analysis, 70% protein was indicated. The shell fraction retained a small amount of the flour, which probably could be further separated. The finely ground shell might find a use similar to ground walnut shell as a filler in plastics. The results of the processing are:

	% of Seed	Remarks
Tallow	21.0	Iodine value 20
Oil	20.0	Iodine value 191
Flour	8.9	70% protein
Shell	36.2	5% protein
Fiber and losses	13.9	Calc. by difference

Thus apparently about 2% tallow (seed basis) is lost while the amount of oil recovered is greater by 3% than found by laboratory examination of the seeds. The higher figure agrees however well with that of 20.3% previously reported by Potts and Bolley (6). The flour content is lower due to incomplete separation, some remaining in the shell fraction.

Preparation and Evaluation of Protein

Chemically purified protein was prepared from the flour by a different procedure than that used by Holland and Meinke (6) for amino acid and vitamin analysis. One part of the tallow seed flour, prepared as described above, was treated with 15 parts of dilute hydrochloric acid at pH of 4.7 to extract the soluble constituents, including soluble non-protein nitrogen. The solids were separated from the extract by centrifuging. The protein in the wet solids was extracted with sodium hydroxide solution adjusted to a pH of 11.0 in the ratio of 30 parts of solution to 1 part of original flour. Again the residual solids were removed by centrifuging. The protein was recovered from the dispersion by adjusting to a pH of 4.7 with sulfur dioxide to coagulate and precipitate the protein. The supernatant liquid was withdrawn and the settled protein spray dried at 125°F. outlet temperature. All extraction, separation, coagulation, and settling operations were carried out at room temperature. In order to compare the tallow protein with soybean, peanut, and linseed proteins these materials were prepared from their respective flours by the same procedure. Yields and nitrogen determinations calculated to a moisture-free basis were:

Preparation

	% N in Flour	% N in Protein	% Yield
Tallow protein	11.3	16.5	60
Soybean protein	8.9	15.1	47
Peanut protein	8.2	15.9	45
Linseed protein	8.9	15.0	34

The viscosity yield value and thixotropic gain of 50 g. of proteins dispersed in 200 ml. of a 0.12% sodium hydroxide solution were measured, using a standard Gardiner Mobilometer. The color of the dry powder was estimated according to a method of Doob, Wellmann, and Sharp (3). Results are shown below:

Physical Properties

	Viscosity Poises	Yield Value, g./sq. cm.	Thixotropic Gain,%	Color
Tallow protein	0.53	8.3	3,500	2+
Soybean protein	1.0	4.6	230	2+
Peanut protein	0.18	0	180	5
Linseed protein	0.22	0	1,240	4+

The adhesive property of Chinese tallow seed protein was compared with the other seed proteins and with casein, using the following glue formula (2) and procedure (14):

Mix (I)
- Protein .... 30 gm.
- Water .... 90 gm.

Mix (II)
- Sodium silicate (40°Bé) .... 15 gm.
- Water .... 25 gm.

Mix (III)
- NaOH (18% aqueous solution) .... 13 gm.

Mix (IV)
- $CuSO_4$ .... 1 gm.
- Water .... 10 gm.

Add (II) to (I) then add (III), next (IV), and mix thoroughly.

The glue was spread on three-ply veneer panels, 30.6 pounds per 1,000 square feet, and the panels were pressed for 5 hours at room temperature at 200 pounds per square inch. Test pieces were cut from the resulting plywood and tested in a Riehle Tester with the results following:

Plywood Glues

	Joint Failure, lb./sq. in.
Tallow protein	211
Soybean protein	172
Peanut protein	182
Linseed protein	187
Casein protein	277

From the physical properties and performance data given, some differences may be noted. Whether these truly reflect the nature of the proteins or are due to processing variations cannot be stated at this time. However the properties of Chinese tallow seed protein indicates that it has possibilities as an industrial protein.

Evaluation of Stillingia Oil

The oil which is commonly called stillingia oil, prepared from a crushed decoated seed as described previously, was evaluated according to the procedure of Bolley and Gallagher (1). About two gallons of the clarified oil were utilized. The results of a good grade of bleached linseed and unbodied dehydrated castor are reported in order to obtain a comparison. The constants are:

	Stillingia	Linseed	DCO
Viscosity	A	A—	H+
Color	7	6—	6
Appearance	Clear	Clear	Clear
Odor	Bland	Normal	Normal
Acid value	6.1	4.3	8.5
Saponification value	205.3	193.4	198.3
Acetyl value	7.5	6.2	19.4
Iodine value	191	180.4	137.3
% unsaponifiable	2.35	1.77	0.96
% Ash	0.0167	0.000	0.000
Refractive index	1.4843	1.4788	1.4820
Specific gravity	0.9404	0.9344	0.9377

Of particular interest was the high iodine value and the unexpected high saponification value. The latter value is understandable in view of Hilditch's (5) observation that stillingia oil contains a relatively short chain conjugated unsaturated acid. The above constants for stillingia oil are in fair agreement with those which have been previously reported in the literature (loc. cit.).

The performance of the oil is shown below:

Bodying Test	Stillingia	Linseed	DCO
Time to Q	43 min.	93 min.	12 min.
Color at Q	10+	9	7
Time to Z2	105 min.	186 min.	65 min.
Color at Z2	11—	11	8
Time to Z5	131 min.	225 min.	85 min.
Color at Z5	11	12	8
Time to Z8	157 min.	269 min.	105 min.
Color at Z8	11+	13	9
Time to Gel	170 min.	300 min.	111 min.
Drying Test			
Set to touch	2¾ hr.	4 hr.	1¾ hr.
Dryness			
24 hr.	10—	9+	9
48 hr.	10—	9+	9+
96 hr.	10—	8	8
192 hr.	9+	9	9
Sward hardness			
24 hr.	5	5	3
48 hr.	5	4	2
96 hr.	5	5	4
192 hr.	5	5	1
Film Solubility			
% soluble in water	10.5	14.2	16.7
Acid value of soluble	248.0	217.0	178.8
% soluble in hexane	14.2	14.1	22.4
Acid value of soluble	119.8	138.6	148.2
% soluble in acetone	35.1	53.6	80.4
Acid value of soluble	149.6	124.6	122.0
% soluble in alcohol-benzene	42.4	73.1	86.5
Acid value of soluble	156.6	116.0	132.0

Cold water resistance tests, hot water resistance tests, and alkali resistance on the three oils were similar with the possible exception that the hot water and alkali resistance of dehydrated castor oil was somewhat superior to linseed which in turn was slightly superior to stillingia. The standard reactivity test indicated that stillingia oil was stable in the presence of reactive pigments such as zinc oxide. Standard varnishes were prepared with the three oils as described in the previously cited paper (1). The results are given on the next page.

Two standard paints were made up, using in one case a single pigment zinc sulfide (S.P.) and the other a standard formula containing white lead, titanium dioxide, zinc oxide, and asbestine (M.P.). The results obtained from these paints are shown on the next page.

From the data given in the tables it can be observed that stillingia oil is a faster heat bodying oil than refined linseed and closely approaches dehydrated castor oil. The drying time of the stillingia oil is intermediate between that of linseed and dehydrated castor. The dried films of stillingia oil are more insoluble than either of the two comparison oils. Varnishes made with stillingia oil are quite satisfactory and in general tend to be faster cooking and produce harder films than either the linseed or dehydrated castor but are somewhat slower drying. The alkali, hot, and cold water resistance are satisfactory. Paints made with stillingia oil produce films having properties which compare favorably with linseed oil and dehydrated castor. However after-yellowing of stillingia oil is distinctly greater than the other paint oils. This feature should be carefully watched in the commercial application of the oil in protective

Bakelite BR 254 Varnish

	Stillingia	Linseed	DCO
Cooking time	82 min.	137 min.	86 min.
Set to touch	80 min.	44 min.	20 min.
Dust free	160 min.	254 min.	28 min.
Dryness			
8 hr.	9	8—	9
24 hr.	10—	10—	10—
48 hr.	10—	10	10
96 hr.	10—	10	10—
192 hr.	10	10	10
Sward hardness			
24 hr.	13	8	4
48 hr.	21	10	7
96 hr.	21	14	9
192 hr.	37	19	12
Cold water (time to whiten)	Unaffected	Unaffected	Unaffected
Cold water (time to fail)	Pass 192 hr.	Pass 192 hr.	Pass 168 hr.
Hot water (time to whiten)	Unaffected	Unaffected	Unaffected
Hot water (failure)	Pass	Pass	Pass
Alkali (time to whiten)	624 hr.	23 hr.	19 hr.
Alkali (time to fail)	1,176 hr.	672 hr.	66 hr.

Amberol 801 Varnish

	Stillingia	Linseed	DCO
Cooking time	15 min.	33 min.	43 min.
Set to touch	173 min.	202 min.	56 min.
Dust free	250 min.	333 min.	76 min.
Dryness			
8 hr.	9	7	10
24 hr.	10—	9	10
48 hr.	10—	10	10
96 hr.	10—	10—	10
192 hr.	10	10	10
Sward hardness			
24 hr.	15	8	5
48 hr.	30	13	6
96 hr.	30	20	7
192 hr.	46	23	19
Cold water (time to whiten)	24 hr.	192 hr.	Remained clear
Cold water (time to fail)	Pass 192 hr.	Pass 192 hr.	190 hr.
Hot water (time to whiten)	Unaffected	43 min.	Remained clear
Hot water (failure)	Pass	Pass	Soft, dull
Alkali (time to whiten)	6 min.	2 min.	11 min.
Alkali (time to fail)	25 min.	157 min.	15 min.

Limed Rosin Varnish

	Stillingia	Linseed	DCO
Cooking time	73 min.	100 min.	75 min.
Set to touch	340 min.	278 min.	58 min.
Dust free	400 min.	333 min.	120 min.
Dryness			
8 hr.	8	7	9+
24 hr.	10—	10	10—
48 hr.	10—	10	10—
96 hr.	10—	10	9+
192 hr.	10	10	10
Sward hardness			
24 hr.	17	19	5
48 hr.	28	20	11
96 hr.	28	27	13
192 hr.	40	33	15
Cold water (time to whiten)	5½ hr.	23 hr.	43 hr.
Cold water (time to fail)	Pass 192 hr.	Pass 192 hr.	43 hr.
Hot water (time to whiten)	1 min.	6 min.	Immediate
Hot water (failure)	Blistered	White, dull	White, dull
Alkali (time to whiten)	Immediate	14 min.	2 min.
Alkali (time to fail)	10 min.	18 min.	12 min.

coatings. In general, stillingia oil is an excellent general purpose drying oil for use in paints and varnishes and would rate for many purposes superior to bleached linseed oil, while approximating the desirable characteristics of dehydrated castor oil.

Conclusions

The experiments described above indicate that the seed of the Chinese tallow tree can readily be processed in equipment available in many oil mills. The most promising products are tallow, stillingia oil, and high protein flour. There is also the possibility of utilizing the shell and the fiber. Thus it has been established that the seed has a definite market value. Whether or not Chinese tallow tree culture can be profitable will depend on the cost of raising the trees and harvesting the seeds in this country. This problem is being actively studied by Dr. Potts of Texas A. & M. and others. It has been estimated (12) that 160 trees can be grown per acre which would yield after six years somewhat over 60 pounds of seed per tree or 10,000 pounds per acre. The Chinese tallow tree as a source of valuable seeds should continue to receive serious consideration.

Paint Tests

	Stillingia	Linseed	DCO
Appearance			
S. P.	Good	Good	Excellent
M. P.	Fair	Fair	Good
Brushing			
S. P.	Fair	Fair	Poor
M. P.	Good	Fair	Poor
Leveling			
S. P.	Fair	Poor	Good
M. P.	Fair	Poor	Good
Consistency			
1 day S. P.	700+ g.	665 g.	250 g.
7 days S. P.	700 g.	645 g.	240 g.
1 day M. P.	302 g.	260 g.	260 g.
7 days M. P.	290 g.	260 g.	260 g.
Dry to touch			
@ 25°C. S. P.	6½ hr.	7½ hr.	3¾ hr.
@ 25°C. M. P.	6 hr.	7½ hr.	3½ hr.
Dry to touch			
@ 5°C. S. P.	26 hr.	42 hr.	42+ hr.
@ 5°C. M. P.	26 hr.	42 hr.	26 hr.
Dry to touch			
@ 110°C. S. P.	13 min.	14 min.	10 min.
Drying odor			
S. P.	Strong	Strong	Moderate
M. P.	Strong	Strong	Moderate
Hardness 1 day			
S. P.	Firm	Soft	Firm
M. P.	Soft	Firm	Hard
Hardness 3 days			
S. P.	Firm	Firm	Hard
M. P.	Firm	Hard	Very hard
Yellowing			
S. P.	Considerable	Very slight	Very slight
M. P.	Considerable	Very slight	Slight
Water permeability			
S. P.	1.34	1.34	1.08
M. P.	1.07	0.63	0.64
Flexibility			
M. P.	20.0	28+	28+
Elongation			
S. P.	12.6	21.4	25.5
M. P.	4.2	5.0	9.0
Tensile strength			
S. P.	58.0	29.0	14.7
M. P.	70.5	35.2	38.7
Taber abrasion.			
M. P.	206	254	264
Shear hardness.			
S. P.	590	467	288
M. P.	600	600	428
Sward hardness			
S. P.	7	6	3
M. P.	9	7	9
60° gloss			
S. P.	54.3	70.1	80.0
M. P.	5.4	16.7	36.2
Daylight reflectance			
S. P.	86.5	84.1	87.0
M. P.	75.9	79.0	81.0

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