

Safflower Oil

G. WINTER, Defense Research Laboratories, Maribyrnong, Australia

THE possibility of using safflower oil as an ingredient of paints and varnishes has been investigated intermittently during the past 30 years, but probably owing to economic reasons, as well as to a lack of appreciation of its potential value, safflower has never been considered as a major oil crop for paints. Only a few investigators, notably Rabak (1) and later Claassen (2), continued their research work. New varieties were developed yielding up to 37% oil as compared with previous yields of 20 to 25%, thus challenging the economic difficulties that had been encountered. The continued world shortage of oils and fats has given this work added stimulus, and further valuable research data have been reported in recent years.

In the past few years considerable interest has also been shown in Australia regarding the possibility of growing safflower as an oil crop since it is now realised that safflower oil is a true drying oil and not a semi-drying oil, as was formerly believed. Some of the earlier Australian experiments on the growing of the crop, on oil yields, and on the properties of the oil have been reported elsewhere (3).

Composition of the Oil

The composition of safflower oil is simpler than that of most other drying oils because the mixed fatty acids are composed only of linoleic, oleic, and saturated acids. A large number of samples of oil obtained from seed grown over a number of years by the Waite Agricultural Research Institute, Adelaide, South Australia, have been examined by spectrophotometric methods and the linoleic acid content found to vary between 72 and 79%. The oil should therefore be expected to have reasonable drying properties.

One of the shortcomings of linseed oil as a paint vehicle is the yellowing of the ageing film, which is particularly pronounced when the film is not exposed to direct sunlight, as with interior decorative finishes. It is generally agreed that the yellowing of films from oils such as linseed and perilla is due to the presence of substantial proportions of linolenic acid; oils such as sunflower and soyabean from which linolenic acid is either absent or is present in small proportions only do not show this defect.

The presence of linolenic acid in safflower oil has been reported by a number of workers (4, 5, 6, 7), but their evidence has since been criticised (8). The absence of linolenic acid in a sample of safflower oil from an experimental crop has been demonstrated conclusively by the author on the basis of examination by the thiocyanogen method, the insoluble bromide method, and the spectrophotometric method (9). A large number of additional samples have since been examined spectrophotometrically, and none contained linolenic acid.

Drying Power

Many workers have assessed the value of an oil by its rate of drying in the absence of accelerators, without considering the effect of metallic driers of this property. It is generally accepted that the drying of oils depends on the initiation of a chain reaction either by peroxidic compounds or metallic catalysts (10). Since linolenic acid forms peroxides more rap-

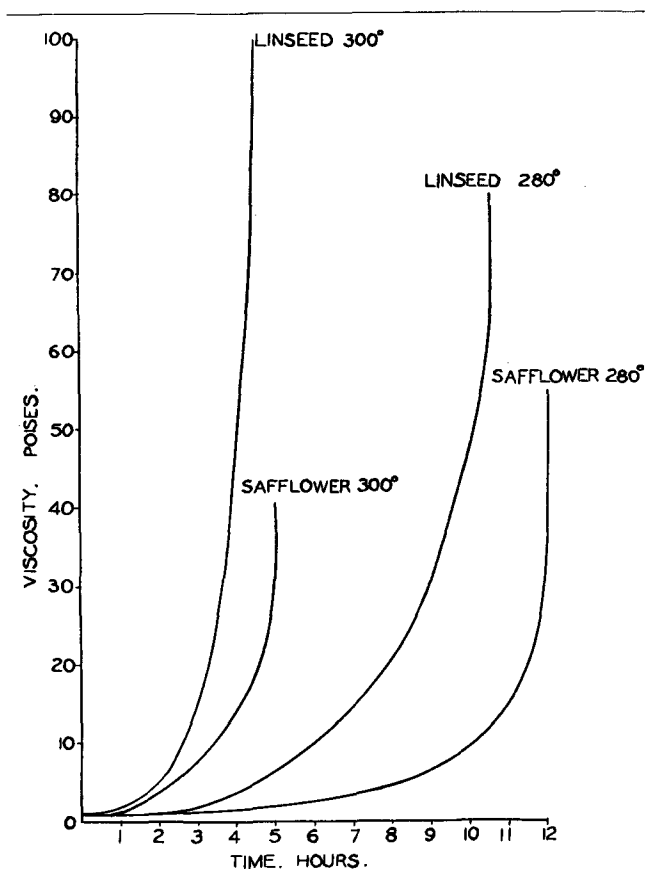


Fig. 1. Heat polymerization of linseed and safflower oils.

idly than linoleic acid, it has been suggested (11) that it is the presence of this acid that is responsible for the rapid drying of oils such as linseed and perilla. However these oils, although rich in linolenic acid, still require the addition of metallic driers to dry within a time acceptable to paint users. The catalytic effect of linolenic acid is therefore of little importance in the technological evaluation of the drying power of oils. Since linolenic acid will cause yellowing and possibly embrittlement of the films, its absence from a drying oil should be of advantage unless it imparts other valuable properties.

TABLE I
Drying Time of Safflower Oil in Comparison With Linseed Oil

Naphthenate Driers Calculated on Metal	Safflower Oil	Linseed Oil
Without Driers	Not dry after 5 days	3 days
0.05% Cobalt (on metal)	8-16 hours	8-16 hours
0.1% Cobalt	8 hours	7 hours
0.1% Manganese	8-16 hours	8-16 hours
0.2% Lead plus 0.05% Cobalt	7 hours	7 hours

In Table I are shown the drying times of safflower oil films, with and without driers, in comparison with linseed oil films. The drying times were determined by the silver sand method. Without the addition of metallic driers, safflower oil dries very much more slowly than linseed oil, but when the same amounts of driers are added to safflower and linseed oil, they dry at approximately the same rate. Similar results

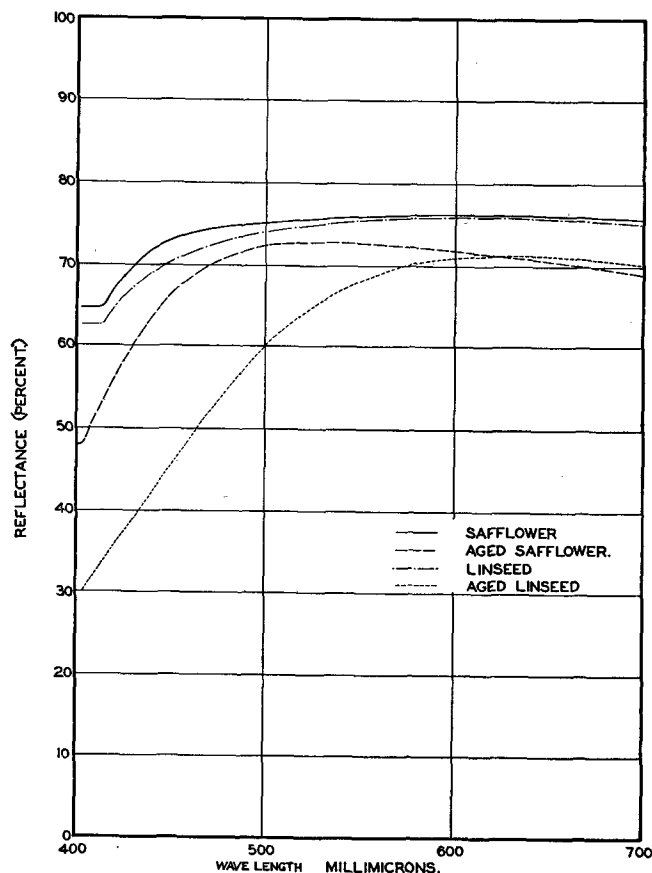


Fig. 2. Reflectance spectra of white linseed and safflower paints before and after ageing.

have been reported recently by English workers who used bodied safflower and linseed oils (12).

Heat Polymerisation

The rate of thickening of safflower oil in comparison with linseed oil is shown in Figure 1. The colours and acid values of the heat-treated oils are shown in Table II.

TABLE II
Colour and Acid Value of Heat-Treated Oil
"J" Viscosity

Oil	Temperature °C.	Lovibond Colour Units 1 cm. cell	Acid Value mg. KOH per g.
Safflower	280	1.5 R 10 Y	2.8
	300	4.6 R 28 Y	4.9
Linseed	280	3.5 R 27 Y	2.9
	300	6.2 R 27 Y	4.3

Safflower oil polymerises somewhat more slowly than linseed oil, but in practice it has been found that when safflower oil is bodied at temperatures 10° to 15°C. higher than those normally employed for linseed oil, heavy bodied oils may be obtained at approximately the same rate. The small amount of extra fuel required for the higher bodying temperature is offset by the better colour of the stand oil.

Alkyd Resins

Alkyd resin varnishes of various oil lengths prepared from safflower oil showed no difference in speed of drying, hardness, and water resistance, as com-

pared with similar linseed oil varnishes, but they were outstandingly superior in non-yellowing properties.

This was particularly pronounced in white enamels made by pigmenting alkyd resin varnishes (30% phthalic) with zinc oxide and titanium dioxide. They were painted on tinned steel panels and allowed to age for 6 months in a dark cupboard. The linseed enamel became distinctly yellow whereas the safflower enamel was practically unchanged in colour. This difference is demonstrated in Figure 2 by the increased absorption by the aged linseed enamel film in the 400-600 m μ . region.

Durability

The resistance to outdoor weathering of paints made from safflower oil and mixtures of safflower and linseed oil have been examined on variable angle and vertical exposure racks facing north at Maribyrnong, Victoria. The formulations, details of which have been reported elsewhere (3), included white lead/zinc oxide paints (white), iron oxide paints (red), and yellow ochre paints (light brown). In the first series of trials which were commenced three years ago, the safflower oils were obtained by solvent extraction in the laboratory from seeds of two varieties (U.S.A. and Niphad) grown on experimental plots at the Waite Agricultural Research Institute, Adelaide. A second more extended series is being prepared, using commercially produced safflower oil.

The exposure panels were examined periodically by the method of Rischbieth (13) for general appearance, discolouration, dirt collection, gloss, chalking, erosion, checking, and flaking and blistering. The individual ratings for each panel were summed up, using a nomograph as recommended by Rischbieth, Bussell, and Laurie (14). Individual ratings for each property are not reported here, but the aggregates of the ratings, termed "utility value," are shown in Table III.

The outdoor performance of paints based on safflower oil or safflower oil/linseed oil mixtures is essentially similar to that of linseed oil paints. With the exception of the red oxide paint series the paints

TABLE III
Durability of Safflower Paints

Paint	Method of Exposure	"Utility Value"				
		Safflower U. S. A. Strain	$\frac{1}{2}$ Safflower U. S. A. Strain $\frac{1}{2}$ Linseed	Safflower Niphad. Strain	$\frac{1}{2}$ Safflower Niphad. Strain $\frac{1}{2}$ Linseed	Linseed
White White Lead-Zinc Oxide	Variable angle	8	7 $\frac{1}{2}$	7 $\frac{1}{2}$	7	7 $\frac{1}{2}$
	Vertical	8	8	8	8	8
White Zinc Oxide-Anatase Titanium Dioxide	Variable angle	7 $\frac{1}{2}$	8	8 $\frac{1}{2}$	7 $\frac{1}{2}$	7 $\frac{1}{2}$
	Vertical	7 $\frac{1}{2}$	8	8	7 $\frac{1}{2}$	7 $\frac{1}{2}$
Cream White Lead-Zinc Oxide	Variable angle	6 $\frac{1}{2}$	6 $\frac{1}{2}$	6 $\frac{1}{2}$	7 $\frac{1}{2}$	7
	Vertical	8	6 $\frac{1}{2}$	8	8	8
Red Red Oxide	Variable angle	7	7	8	8	8
	Vertical	7	7	8	8	8
Light Brown Yellow Ochre	Variable angle	7	7	7	6	7
	Vertical	8 $\frac{1}{2}$	8	8	8	8 $\frac{1}{2}$

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.

REFERENCES

1. Rabak, U. S. Agr. Cir. No. 366 (1935). Revised.
2. Claassen, Nebr. Agr. Exp. Sta. Cir. No. 87 (1949).
3. Pugsley and Winter, Munitions Supply Laboratories (Australia), Report 171 (1947).
4. Godbole and Amarendva, *Allgem. Ol. u. Fettztg.*, 33, 7 (1936).
5. Juchnovski, *Chem. Zentr.*, 1111 (1933).
6. Kaufmann and Fiedler, *Fette u. Seifen*, 44, 420 (1937).
7. Bickford, Mann, and Markley, *Oil and Soap*, 20, 85 (1943).
8. Hatt and Troyahn, *J.C.S.I.R.* (Australia), 19, 86 (1946).
9. Winter, *Paint Notes*, 2, 160 (1947).
10. Farmer, *J. Oil and Colour Chem. Assoc.*, 31, 393 (1948).
11. Gunstone and Hilditch, *J. Soc. Chem. Ind.*, 66, 205 (1947).
12. Hilditch, *J. Oil and Colour Chem. Assoc.*, 32, 5 (1949).
13. Rischbieth, *Munitions Supply Laboratories Circular No. 10* (1947).
14. Rischbieth, Bussel, and Laurie, *Paint Notes* 4, 34 (1949).

[Received October 25, 1949]

Utilization of the Seed of the Chinese Tallow Tree

DON S. BOLLEY and RALPH H. McCORMACK, National Lead Company, Brooklyn, N. Y.

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 al-

kali 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