Kapok Oil and the Halphen Test*

Notes Describing the Extreme Activity of this Oriental Oil Toward the Sulfur-Bisulfide Reagent

By H. P. TREVITHICK *and* W. H. DICKHART

KAPOK OIL is the oil pressed from the seed of the kapok plant, a plant very similar to the cottonseed in general characteristics, common in the Orient, particularly in China. The seeds are round, about the size of a pea, with black hull, and white kernel. They are free from lint, being similar to "bald" cottonseed in that respect, and easy to crush.

A sample of such oil from imported seeds, crushed in an American crude oil mill, gave the following results on analysis:

Moisture & Volatile Matter	0.45%
Insoluble Impurities (meal)	0.36%
Specific Gravity (@ 15.5° C.)	0.9221
Iodine Value (Wijs)	94.9
Saponification Value	194.5
Index of Refraction	1.4710
Unsaponifiable Matter	0.66%
Free Fatty Acids (Oleic)	12.13%
Refining Loss	35. %
Color (Refined Oil)	35 Yellow 7.0 Red
Titre (Refined Oil)	28.1° C.
Titre (Soapstock Acids)	30.2° C.
Halphen Test	Strong—Immediate

Due to the fact that this oil and cottonseed oil are very similar, we made some comparative tests on the sensitivities of the two oils to the Halphen reaction. In these experiments, sesame oil, olive oil, palm kernel oil and coconut oil were used. After some preliminary experiments, a series of mixtures was prepared of each of these oils containing 0.05%, 0.1%, 0.5% and of 1% of kapok oils, and another series containing 0.5% and 1% of cottonseed oil in each of the above-mentioned oils. Each mixture was subjected to the Halphen reaction. In this test, 5 cc. of the oil mixture in a test tube is shaken with 5 cc. amyl alcohol, 5 cc. of a 1% solution of sulfur in carbon bisulfide is added, the whole heated cautiously in the steam bath until the bisulfide has evaporated; and the tube then heated in the boiling water bath for 5 hours, counting the time from the point at which the bulk of the carbon bisulfide is removed. The results were as follows:

COMPARATIVE HALPHEN TESTS

Oil Samples	Result of Test	Length of time to show color	Depth of red color in 5 hours
Sesame	Negative	5 hours	None
Olive	"	"	"
Coconut	"	"	"
Palm Kernel	"	"	"
Cottonseed	Positive	5 minutes	Very deep red
Kapok	"	2 minutes	" " "
Sesame plus 0.05% Kapok	Positive	20 minutes	Light pink
Olive " " "	"	30 "	" "
Coconut " " "	"	30 "	" "
Palm Kernel " " "	"	30 "	" "
Sesame plus 0.1% Kapok	Positive	20 minutes	Definite red
Olive " " "	"	" "	" "
Coconut " " "	"	" "	" "
Palm Kernel " " "	"	" "	" "
Sesame plus 0.5% Kapok	Positive	10 minutes	Deep Red
Olive " " "	"	" "	" "
Coconut " " "	"	" "	" "
Palm Kernel " " "	"	" "	" "
Sesame plus 0.5% C/S Oil	Positive	45 minutes	Light Red
Olive " " "	"	" "	" "
Coconut " " "	"	" "	" "
Palm Kernel " " "	"	" "	" "
Sesame plus 1% C/S Oil	Positive	40 minutes	Definite Red
Olive " " "	"	" "	" "
Coconut " " "	"	" "	" "
Palm Kernel " " "	"	" "	" "

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Kapok Oil

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In all these tests, the color developed by the kapok oil is much deeper than that developed by the same percentage contamination of cottonseed oil. Further, the color produced by any given mixture with kapok oil is equivalent to that given by mixtures containing cottonseed oil in quantities ten or more times as large as the particular kapok admixture. Mixtures of oils containing cottonseed oil in amounts of 5% or more give colors of such deep red, that the depth of color is of very little value, as indicating the percentage of contamination. This is also true of kapok mixtures containing 1% kapok oil, or more.

These tests, of course, were made on cottonseed oil, which had not been subjected to any heat that would vitiate the Halphen reaction.

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Solvent Specifications

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The fact that the temperature of extraction determines the amount of extracted material obtained leads to some interesting conclusions regarding the determination of oil in cottonseed products by different laboratories. In South Texas, cottonseed start moving in July. Therefore, the laboratories have to make analyses during the hottest part of the year. The petroleum ether which has been used contains a large amount of iso-pentane quickly lost when extracting in hot weather, necessitating the refilling of the extraction flask two and three times, thereby materially increasing the amount of hexane in the solvent, which would tend to give a higher percent of oil than that obtained by laboratories operating in cooler climates.

Complaint was made against the oil results obtained by one laboratory being materially higher than those obtained by another laboratory, both using the same petroleum ether and operating in the same city. On investigation, the fact was found that the laboratory obtaining the lower results was using a Smalley extraction tube, while the other laboratory was using a Butts tube. Both tubes are of the percolating type, but the Smalley tube has a side arm which carries the hot vapors of the solvent direct to the condenser without the vapors coming in contact with the material being extracted, which contact does occur in the Butts tube. On substituting a Butts tube for the Smalley tube, the results obtained by the laboratory obtaining the lower results checked the results obtained by the laboratory obtaining the higher results. The conclusion was reached, independent of the work of this committee, that the differences between the two laboratories was due to the difference in temperature at which extraction occurred.

A number of the samples of cottonseed which had been extracted at a temperature of 80° C., to obtain the results given in Table V, were re-extracted in the ordinary manner. The material extracted was solid and reddish-brown in color, apparently of a resinous nature.

Conclusions

THE solvent used should be of the pentane type, containing a minimum amount of iso-pentane, iso-hexane, and hexane. The following specifications would give a product meeting the above requirements:

Initial boiling temperature	—not less than 35° C.
" " "	— " over 40° C.
Dry flask end point	— " " 60° C.
" " " "	— " less than 50° C.
At least 95% distilling under	—55° C.
Not over 85% " "	—40° C.
Specific gravity at 60° F.	—.630 to .675
Color—water white	
Evaporation residue—not over .002% by weight	
Doctor test—sweet	
Copper strip corrosion test—non-corrosive	
Unsaturated compounds—trace only permitted	

This product would have approximately the following composition:

Iso-pentane	0% to 5%	or about	2%
Normal pentane	70% " 85%	" "	80%
Iso-hexane	5% " 20%	" "	9%
Normal hexane	5% " 20%	" "	9%

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Linseed Crush

According to preliminary figures of the Census Bureau there were 25 mills in the United States which crushed flaxseed during the quarter ending June 30, 1931, reporting a crush of 201,223 tons of flaxseed and a production of 130,324,839 pounds of linseed oil. These figures compared with 203,568 tons of seed crushed and 130,863,405 pounds of oil produced for the corresponding quarter in 1930, and 278,933 tons of seed and 187,018,538 pounds of oil in 1929.

Stocks of flaxseed at the mills on June 30, 1931, amounted to 33,535 tons, compared with 65,173 tons for the same date in 1930 and 100,928 tons in 1929. Stocks of linseed oil reported by the crushers were 47,156,191 pounds on June 30, 1931, compared with 71,222,982 pounds for the same date in 1930, and with 83,544,918 in 1929.