In using liquid extractors the official procedure is followed to the point of transferring the soap solution to the extraction cylinder. At this point the transfer is made to the liquid extractor and petroleum ether is added to just below the side arm. A 250-ml. Erlenmeyer flask, containing a small quantity of petroleum ether, is connected to the side arm and the extraction started. Extraction is carried on for one hour and then the contents of the extractor are transferred quantitatively to a 500-ml. separatory funnel, allowed to separate, and the soap solution drained. By gently swirling the solvent, any whitening agent, or other insoluble material originally present in the soap, can be forced to the bottom of the funnel and drawn off. Then the contents of the Erlenmeyer flask are transferred quantitatively to the separatory funnel. Approximately 10 ml. of a saturated solution of basic lead acetate [$Pb(C_2H_3O_2)_2 \cdot Pb(OH)_2 \cdot H_2O$] are added. If the immediate precipitate is heavy it is drawn off without shaking. This step is repeated until there is only a slight precipitate and then the funnel is shaken vigorously for 30 seconds, the water layer drained, and the process repeated with another 10-ml. portion, after which the petroleum ether extract is washed 3 times with about 25 ml. of 10 percent ethyl alcohol. The petroleum ether extract is then transferred to a 300-ml. Erlenmeyer flask containing about 15 grams anhydrous sodium sulfate to remove moisture, swirled 5 minutes, and filtered into a tared Soxhlet flask if the solvent is to be recovered (otherwise into a tared beaker), and evaporated to dryness.

It has been found in this laboratory that traces of solvent in contact with extracted material in a Soxhlet flask can be removed by connecting a two-hole rubber stopper, or a one-hole rubber stopper with a V-notch on a side, to the flask and connecting a vacuum line to a tube through one hole while the flask is kept warm. In this way while there is a partial vacuum over the material there is a constant circulation of air through the other opening. This method has been found more satisfactory than allowing vapors to evaporate spontaneously on a steam bath or in an oven. After a minute or so of this, the flask with its contents is dried in a 100° C. air oven for one-half hour or longer to constant weight. Care must be taken not to produce a static electric charge on the flask by rubbing with a towel, or significant errors in weight will occur.

Rosin

An easier and more rapid method than the official method for preparing the fatty acids for the rosin determination is to dissolve 10 grams of soap in about 300 ml. of boiling water and then to separate the fatty acids with about 5 ml. of 1:1 H_2SO_4 (until no more suds and/or turbidity exists). While the fatty acids are still liquid they are transferred to a 500-ml. separatory funnel and cooled to below 25° C. The fatty acids are extracted with 50 ml. of ethyl ether after first washing the residue in the beaker into the separatory funnel with the ether. The aqueous portion is drawn off into the original beaker and the ether extract transferred to another separatory funnel. Three more extractions are made with 30-ml. portions of ether and all the ether extracts are collected in one separatory funnel. The aqueous solution is reserved for chloride determinations as described previously. The combined ether extracts are washed with 25-ml. portions of distilled water containing methyl orange (3 ml. of 1 percent solution per liter) until neutral. The ether extract is transferred to a 250-ml. Erlenmeyer flask containing about 15 grams of anhydrous sodium sulfate to remove moisture, swirled 5 minutes, filtered into a tared Soxhlet flask, and the solvent recovered. Removal of the last traces of ether is accomplished by connecting the flask to a vacuum line as mentioned above, and then drying in a 100° C. oven for one-half hour. After cooling and weighing, 10 ml. of naphthalene-beta-sulfonic acid (an adequate amount for soaps having less than 25 percent rosin) and 15 ml. of methyl alcohol are added. A blank is run in the same way and the determination is continued as outlined in the official method.

Titer Test

The only variation made in this test is the development of a mechanical vertical stirrer having a windshield wiper motor as the source of power. This device is the subject of a brief article now being published elsewhere. The mechanical stirrer furnishes a precise and uniform stirring rate and frees the operator from the fatigue induced by hand stirring.

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Kenaf Seed Oil*

MARIO LEWY[1]

Introduction

KENAF (*Hibiscus cannabinus* L.) belongs to the Malvaceae family. It is an annual plant, native of India and is extensively cultivated for its fibers which are in many characteristics comparable to jute fiber. Under favorable conditions the plant reaches 12 to 14 feet in height and its characteristic large flowers are yellowish with crimson centers. Kenaf was introduced to El Salvador several years ago when a few seeds were brought in from Java. From this original source the plant has spread throughout Latin America.

When introduced to El Salvador, the plant was considered erroneously to be Roselle (*Hibiscus sabdariffa var. altissima*) (2) but it was later identified as *Hibiscus cannabinus* L. (7). Two varieties, vulgaris and viridis make up the plant material being cultivated in Latin America.

Kenaf is a quick growing plant. Usually the fiber may be extracted about 90 days after planting. It may be impracticable to obtain both a seed crop and

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a fiber crop from the same planting and, for this reason, slightly different cultivation methods are used in obtaining the two products. For fiber production the current practice in El Salvador is to plant at the beginning of the rainy season (April or May) and harvesting takes place when the plants start blooming. For seed production, planting is done in September and the seed is harvested 3 to 4 months later. Further information on the cultivation of kenaf in other regions may be found in the work of Crane (4) and Crane and Acuña (6).

Experimental plantings in El Salvador have yielded nearly 1,000 kgs. of seed per hectare. In Cuba, Crane (6) obtained 1,500 lbs. of seed per acre (approximately 1,680 kgs. per hectare) from a planting made late in the season, but he pointed out that yields of over a ton per acre could be produced by planting earlier in the season.

Kenaf seeds are more or less tetrahedral in form and approximately 5 mm. long by 3 mm. wide. The seeds are rather hard and contain a yellowish kernel strongly adherent to the seed coat or episperm, the latter is black in color. The separation of the kernel from the episperm is difficult.

Preliminary laboratory tests showed that seeds with 7% moisture content yielded 20% oil extracted with petroleum ether or 14% oil extracted by pressure. With a production of 1,000 kg. of seed per hectare, it is possible, therefore, to obtain 200 kg. of oil extracted by means of solvents, or 140 kg. of oil extracted by pressure.

In view of the fact that kenaf seed oil production appeared to be a promising new industry for El Salvador, oil extraction methods and oil characteristics were investigated. The results of these investigations are reported here.

Grinding

As the episperm cannot be easily separated from the kernel and decortication is difficult and wasteful, the seed must be ground in its natural state. Grinding by means of crushing roll grinders, similar to those used in cotton seed oil extraction, proved to be entirely satisfactory.

Pressing

Although extraction of the oil may be done by means of solvents, which yield higher percentages of oil than by the pressing method, the latter procedure was investigated here because of the existing commercial equipment in El Salvador.

In the laboratory investigation, a Carver Hydraulic Laboratory Press with a cage system capable of developing a pressure of 5,000 lbs. per square inch, was used. The ground seed was stirred constantly in a glass container while it was maintained at a temperature of 100° C. over a steam bath for 30 minutes. The calculated quantity of water previously heated to 100° C., necessary to give a certain moisture percentage, was then added during the last 5 minutes of heating. The heated material was immediately put in the press and the pressure applied.

In making the extraction with seed containing 7% moisture a production of 10% oil was obtained using a pressure of 5,000 lbs. per square inch. By gradually increasing the moisture content to 10%, a progressive increase in percentage of extractable oil resulted until a 14% production was obtained at a pressure of 5,000 pounds per square inch. Material having more than 10% moisture was too soft and spilled from the box of the press.

Investigations on a commercial scale were conducted in a factory where the seed was put through crushing rolls and then brought to a cooker in which the material was cooked at a temperature of 105° C., for 20 minutes. The heated mass, containing between 9 and 10% moisture, was then put under 5,000 lbs. pressure in a steel box frame hydraulic press, and a production of 13.1% oil was obtained. As a basis of comparison, similar material, but unheated before extraction, yielded only 10% oil.

Oil Characteristics

The oil obtained by pressing the unheated material is clear yellow in color, while oil obtained from previously heated material is reddish brown in color. The cold pressed oil is almost odorless while the hot pressed oil has a mild odor, similar to that of cotton seed oil.

Several investigations have been made on kenaf seed oil from different parts of the world. The oil characteristics have been published by Jamieson (8).

Table 1 lists the characteristics of kenaf seed oil produced in El Salvador together with the characteristics of Roselle (*Hibiscus sabdariffa var. altissima*), cotton, and Kapok seed oils, as a basis for comparison.

Oil extracted from kenaf seed shows the Halphen Reaction as do the oils extracted from Kapok, cotton seed, and other malvaceous plants.

Oil Refinement and Oil Uses

Oil obtained in the factory was so dark that its color classification could not be determined. In the laboratory, on the other hand, where the extraction was made with fresh seed, oil reddish in color but clearer than crude cotton seed oil was obtained. This difference in color was probably due to differences in condition between the two lots of seed, since an oil sample extracted in the laboratory from seed partially deteriorated was very dark red in color, and had an acid value of 7, all other factors being equal.

It appears that there should be no essential differences between Kenaf oil refinement and cotton seed oil refinement. Commercial factory trials were made with caustic soda, 16° Bé lye with 30% excess.

The initial ambient temperature was 25° C. and this was gradually raised to 60° C. There was a 20-minute mixing period. The emulsion produced broke with relative rapidity, the soap stock being separated in almost solid and compact form which facilitated its separation.

Kenaf seed oil is an edible oil, nonsiccative, and has the properties enabling it to be used as an excellent substitute for cotton seed oil in all its uses, with the advantage of having a somewhat milder odor. Oil obtained in the factory was used as salad oil and for cooking purposes. It may also be used in soap manufacture, particularly in the manufacture of hard soaps.

Discussion

The storage of Kenaf seed does not present the same problems which accompany the storage of cotton seed. The seed does not hold moisture at the surface as does cotton seed, and the seed coat is harder; furthermore, the absence of lint lessens the danger of fire.

TABLE 1

(*Hibiscus sabdariffa var. altissima*), Cotton, and Kapok Seed Oils, as a Basis for Comparison.

	Kenaf El Salvador	Kenaf (8)	Roselle (9)	Cotton crude oil El Salvador	Kapok (8)
Specific weight at 15° C	0.9175	.....	0.923	0.928	0.920-0.933
Refractive index	$N_D^{40°}$ 1.4657	.....	$N_D^{20°}$ 1.4715	$N_D^{20°}$ 1.4745	$N_D^{40°}$ 1.4605-1.4657
Acid value	4.7	.....	2.2% (in oleic acid)	.....	.....
Saponification value	189.8	.....	193.1	197.2	189-195
Insaponifiable %	1.7	.....	1.1	1	0.8-1.6
Iodine value	99.7 (Hanus)	.....	107.3 (Wijs)	108 (Hanus)	86-100
Reichert-Meissl value	0.5	.....	.....	.....	0.1-0.2
Hehner value	61.8	.....	.....	96.1	.....
Oleic acid %	.....	45.3	.....	.....	.....
Linoleic acid %	.....	23.4	.....	.....	.....
Palmitic acid %	.....	14.0	.....	.....	.....
Stearic acid %	.....	6.0	.....	.....	.....

[The methods followed are from the A.O.A.C.(1).]

A lot of about 4 tons of Kenaf seed was stored in an ordinary warehouse, in henequen bags, for more than one year with the only apparent deterioration being the complete loss of germination ability. However, the seed is attacked by the larvae of a moth *Plodia interpunctella* (3). Although the damage produced by the larvae is usually insignificant, it could, in time, become a serious problem. Under certain conditions fumigation of the seed might be necessary if it is to be stored for any length of time. Damaged seed yields less oil and it is darker in color. Likewise, this oil has a higher acidity which results in greater losses in the refinement.

The residual cake obtained from the extraction is gray in color due to the episperm which is not initially separated from the kernel. The cake has an agreeable odor and is very similar to the odor produced by the residual cake of cotton seed.

Preliminary experiments carried out with milk cows in El Salvador showed that the cows readily ate the residual cake of Kenaf seed. The cake, which is high in proteins, offers, therefore, a concentrate food for dairy cows.

Michote (10) listed the composition of whole kenaf seeds as follows:

Moisture	9.64%
Mineral matter	6.40%
Oil	20.37%
Nitrogenous matter	21.44%
Saccharifiable matter	15.66%
Crude cellulose	12.90%
Other matter	13.94%

Analysis made on the residual kenaf seed cake showed the following composition (on a dry weight basis):

Crude protein	33.0%
Oil	6.0%
Crude fiber	17.4%
Ash	6.0%
Nitrogen free extract (by difference)	37.6%

Although the residual cake is more valuable as a concentrate food for cattle, it may also be used as a fertilizer material as shown by the following composition.

Nitrogen (N)	5.25%
Phosphoric Acid (P_2O_5)	0.95%
Potash (K_2O)	3.74%

Summary

Kenaf oil is obtained from the seeds of *Hibiscus cannabinus* L. The procedure for extracting the oil is practically the same as is used in extracting cotton seed oil. The seed is ground and pressed without decortication because the episperm is strongly adherent to the kernel and also because of the peculiar shape of the seed. Up to 20% oil of the weight of the seed may be obtained, depending upon the extraction method used. The oil is nonsiccative and, when refined, may be used for salads and cooking purposes. Generally speaking, it has the same uses as cotton seed oil which it may substitute with the advantage of having a somewhat milder odor.

The residual cake, which is gray in color due to the presence of the episperm may be advantageously used as a concentrate food for cattle.

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