CENTRAL PLANTATION CROPS RESEARCH INSTITUTE

ARECANUT FAT, FATTY ACIDS AND [illegible]

A REPORT OF WORK

By the

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I. INTRODUCTION

About 150,000 tonnes of arecanut are produced annually in the country, mainly in the states of Karnataka and Kerala. While good quality arecanut finds use in the preparation of betelnut powder, inferior quality nuts do not find a ready market. Arecanut contains 10-12 per cent of fat, rich in myristic and lauric acids. This fat can easily substitute high-priced coconut fat in the manufacture of soaps, fatty alcohols, fatty acids and a variety of surfactants. The defatted meal contains about 15 per cent tannins which can be separated and used in the leather industry while the defatted and detanned meal can be used in the preparation of compound feed for cattle. Good quality defatted meal can also be used in the preparation of betelnut powder (supari). Thus processing of arecanut on these lines will open up an attractive market.

The Central Plantation Crops Research Institute, Vittal approached the Regional Research Laboratory, Hyderabad (RRL-H) for taking up studies on solvent extraction and other processing methods with a view to utilise the low grade nuts. The laboratory with its extensive experience in processing of oilseeds, fat splitting and fatty acids processing agreed to undertake this

work. After discussion, the following programme of work was agreed upon:

- pretreatment of arecanut for solvent extraction;
- solvent extraction of pretreated nut;
- extraction of tannin from decoiled nut;
- splitting of arecanut fat;
- distillation of fatty acids; and
- analysis of fatty acids

Since the fat content in nut is low, expelling is not feasible. Solvent extraction with hexane was therefore considered as the only method suitable for economic recovery of fat.

2. PRETREATMENT AND EXTRACTION OF FAT AND TANNINS

2.1 Laboratory-scale investigations

The whole nuts were reduced in size by hand pounding followed by crushing in a laboratory hammer mill. The sample had 8.8 per cent moisture and 11.8 per cent fat. The ground material was extracted in a Soxhlet extractor using food-grade hexane as a solvent. The recovered fat was analysed (AOCS methods) to give the following characteristics:

Free fatty acid	:	Trace
Saponification no.	:	222.5
Iodine no.	:	26.6
Melting point, ^{o}C	:	42-43
Unsaponifiable matter, %	:	1.6

The run-of-the-mill sample was found to have a fat content of 9.9 per cent (dry basis). On sieving the sample into several fractions, it was found that the finer the material, the higher was the fat content (details are given in Table 1 at the end of this section).

For the analysis of arecanut, arecanut fat and split arecanut fat samples, the methods of the American Oil Chemists' Society were followed. GLC analysis of the distilled fatty acid

fractions was carried out on the corresponding methyl esters in a Toshniwal unit equipped with flame ionisation detector using a column of EGSS-X on Chromosorb W (80-100 mesh) programmed from 110-200°C (6°/min) and nitrogen (30 ml/min) as carrier gas.

2.2 Bench-scale studies

2.2.1 Pretreatment: For efficient solvent extraction, the surface area of the material for contact should be increased consistent with necessary mechanical strength to avoid crumbling during solvent extraction. The arecanut was processed as follows:

2.2.1.1 Size reduction: The nut as received was of a size range 18 to 25 mm and has to be broken to a size acceptable to the rolls of the flaking machine and corresponding to the range of nip angle of the rolls. The disc huller was used for this purpose with different gap settings of the disc.

The disc huller with one stationary and one rotating disc is similar to an attrition mill except that the plate on one side is stationary. There are cast bars or ridges with cutting edges radiating from the centre on the face of both discs. The face of each disc is concave to feeding the material

at the centre. The feed material thus travels towards the periphery through a gradually narrowing space between the discs. The distance between the discs is least at the periphery where the seed is cut between the stationary and rotating sharp edges.

The arecanut was fed to the disc huller with a maximum gap setting whereby it was crushed into two or three pieces (6-8 mm). The cracked nuts were then fed into the huller twice with the minimum gap setting when a size reduction to 3 mm or less took place.

2.2.1.2 Cooking and flaking: Arecanut crushed to less than 3 mm size was then cooked for 10 minutes in a cooker mounted on a baby-oil expeller after adding 10 per cent water (in addition to the 8.8 per cent moisture already present). The cooked material was flaked in a five-high crushing rolls, consisting of five chilled cast iron rolls arranged vertically one over the other. The assembly is so erected that each roll supports the weight of all the rolls above it, with the result that the particles are subjected to progressively increasing pressure as they pass from one pair of rolls to another. As the material is fed, it passed back and forth between adjoining pairs of rolls as it travels from the top to the bottom. Thus it is rolled four times. The cooked arecanut flakes (0.25-0.5 mm thick) were kept overnight to adjust the moisture to equilibrium level and the structure became firm to withstand mechanical handling in the succeeding operations.

2.2.2 Extraction of fat: Preliminary studies on solvent extraction were carried in a small unit using 3 kg. of uncooked flaked material using food-grade normal hexane. The yield of fat was 9.3% (based on the weight of the meal). The fat had a light cream colour.

Bench scale experiments on solvent extraction of cooked arecanut flakes were carried out in a cylindrical stainless steel batch extractor, 60 cm diameter and 120 cm height. Food-grade normal hexane in a ratio of 1:2 (solid:solvent) was circulated in the extractor charged with arecanut cooked flakes (11.7% moisture) for 6 hours and kept overnight. The miscella was drained and again the same quantity of fresh solvent was added and the extraction repeated. The second stage miscella was removed and the resulting residue (marc) was air dried and kept aside for tannin extraction. The first and second stage miscellas were pooled, and ~~and~~ the solvent was recovered by distillation. The residual fat was further stripped off the remaining solvent under a reduced pressure of 40-50 mm/Hg. When 83 kg. of material was thus extracted (solid:solvent ratio, 1:2) for 6 hours, 7.9 kg of fat (yield 9.6%) and 72 kg of meal (yield 86.7%) were obtained. The defatted meal had a residual fat content of 1.2 per cent and moisture 11.0 per cent. The fat had the following characteristics:

Acid no.	:	11.9
Iodine no.	:	25.9
Saponification no.	:	222.0
Unsaponifiable matter, %	:	1.6

2.2.3 Extraction of tannins: Experiments were carried out on the extraction of tannin from defatted arecanut meal in the extractor (used for fat extraction). Since extraction with water resulted in fermentation and the meal got spoiled, a mixed solvent, namely, aqueous acetone (acetone:water 40:60) was tried to recover tannins.

Defatted meal (35 kg) was taken in the extractor and extracted 4 times, each time with 50 litres of aqueous acetone for 6 hours. The solvent was recovered by distillation and the tannins were recovered from the residual liquor in a spray drier (Lurgi type), a direct dispersion type drier operating on the principle of atomising a fluid feed to form a spray of droplets which mix with hot gas to evaporate the liquid and produce a dispersed dry tannin powder. The yield of tannins was 3.5 kg (10%).

Table 1 Fat content in different sieve fractions of arecanut
(on dry basis)

Fraction	Fraction	Fat extracted in 1st extraction for 6 hrs	Extracted after regrinding to -35 microns	Total
	%	%	%	%
A. (Finest) -35 mesh (-351 microns)	17.7	14.2	0.1	14.3
B. -50+35 mesh (-500 microns +351 microns)	17.7	9.9	0.7	10.6
C. -80+50 mesh (-853 microns +500 microns)	23.2	8.7	2.3	11.0
D. +80 mesh (+853 microns coarse)	41.4	7.1	3.1	10.2
Whole arecanut ground to -35 mesh (to the size of finest fraction i.e.A)	-	-	-	12.9
Run of the mill (before sieve analysis i.e. fractions A+B+C+D)	-	-	-	9.9

Note: Harder portions of arecanut give coarser particles with lesser fat content

3. ARECANUT FATTY ACIDS

3.1 Splitting of arecanut fat

Arecanut fat collected from the extraction experiments in the bench scale unit was pooled, dried and analysed for acid number, iodine number, saponification number and content of unsaponifiable matter (see 2.2.2).

Studies on splitting were carried out in a 1-litre capacity autoclave provided with a mechanical stirrer, sampler withdrawal system and suitable controls for regulating pressure and temperature, and a safety valve. Splitting was done keeping the fat to water ratios at 1:0.4, 1:0.5 and 1:0.6 and pressures at 20, 25 and 30 kg/cm^2. To achieve a better split in lesser time, and for keeping the glycerine concentration in the sweet water high (at 14.0%), two stage splitting was tried, using the sweet water from second stage for splitting in the first stage of a subsequent batch. This was followed by fresh water in the second stage. Single stage splitting was carried out for 4 hours, in each case drawing samples at intervals of half an hour. Split fat where split was 90 per cent and more was pooled, washed and dried for carrying out distillation studies. Three successful runs were carried out in the two stage splitting to study repro-

ducibility. Per cent splits were calculated on the basis of acid values of samples extracted with ether which excludes ether-insoluble foreign matter. The total quantity of charge viz. fat + water taken in each case was kept constant at 525 ± 10 g (except in the case of residue splitting) to keep the space for steam and rate of stirring constant through out. Detailed conditions of splitting and results thereof afe presented in Table 2 for straight splitting and Table 3 for two stage splitting. The residue pooled from different distillations was also analysed (acid no. 18.9; ether insolubles 15%) and resplit for complete recovery of fatty acids (Sl. No.8, Table 2) at a pressure of 25 kg/cm^2 with a fat to water ratio of 1:0.6.

3.2 Distillation of arecanut fatty acids

Split arecanut fatty acids were pooled, washed free of glycerol and dried in vacuum. Per cent split was determined on the ether extracted sample (90.0%). From this, 200 g lots were taken for distillation, first in a laboratory distillation unit and then in a unit with 30 cm high extended surface glass column. Finally further 200 g lots were taken and fractionated in an electrically heated Tower's fractionating column packed with glass beads (1 meter). A vacuum of 3 to 5 mm Hg was maintained during

these distillations and fractionations. Fractions based on their boiling range were separately collected and the corresponding methyl ester samples prepared. These methyl ester fractions were analysed by gas liquid chromatography along with a standard mixture of methyl esters. Details of these distillations/fractionations are tabulated in Table 4 while the fatty acid composition of the fat as well as these fractions are presented in Table 5.

Table 2 Splitting of arecanut fat (single stage)

S.No.	Conditions of split			Percent split in hours								
	Fat:Water ratio W/v	Pressure ± 1 kg/cm^2	Temperature ± 5°C	0*	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
1	1:0.4	25	220	21.9	-	82.3	-	83.6	-	84.1	-	84.3
2	1:0.5	25	220	21.2	54.2	84.2	86.7	88.5	88.7	88.6	88.5	88.5
3	1:0.6	25	220	23.0	66.7	87.7	90.2	90.5	90.4	90.9	91.1	91.2
4	1:0.5	30	230	29.8	67.6	88.7	85.7	89.0	89.6	89.4	89.6	89.7
5	1:0.6	30	230	38.6	69.8	90.8	91.2	91.8	92.1	92.3	92.3	92.4
6	1:0.5	20	205	17.4	-	80.5	-	85.2	-	86.9	-	87.2
7	1:0.6	20	205	21.7	-	84.8	-	88.7	-	89.1	-	89.7
Resplitting of arecanut fatty acid distillation residue												
8	1:0.6	25	220	44.4	-	67.6	-	79.4	-	81.0	-	-

* Zero hr sample is the first sample drawn on attaining the desired pressure

Table 3 Splitting of arecanut fat
(in two stages, 1 hr in each stage)

S.No.	Conditions of splitting				1st stage percent split in 1st hr	2nd stage percent split in 2nd hr	Remarks
	Fat:water ratio	Pressure ± 1 kg/cm^2	Temperature ± 5°C	Time of split in each stage in hrs			
9	1:0.4	25	220	1	81.2	96.0	
10	1:0.4	25	220	1	80.7	95.2	Sweet water from 2nd stage of run No.9 was used in the 1st stage of run No.10. Fresh water was used in the 2nd stage of run No.10 after separating 1st stage sweet water.
11	1:0.4	25	220	1	81.0	95.5	Sweet water from 2nd stage of run No.10 was used in the 1st stage of run 11. Fresh water was used in the 2nd stage of run No.11 after separating 1st stage sweet water

Note: 1. Sweet water of Run No.10 after 1st stage and Run No.11 after 1st stage was pooled together to determine percent glycerine which was 14.0% as against 12.5% obtained in sweet water analysed in an optimum run in direct splitting (single stage).

2. Shortage in quantity of sweet water from 2nd stage used in the first stage of the following batch was made up with fresh water which was about 25 ml in each case.

Table 4 Distillation of arecanut fatty acids and split arecanut residue

S.No.	Particulars of apparatus	Fraction I Temp. range °C	Fraction I DFA %	Fraction II Temp. range °C	Fraction II DFA %	Fraction III Temp. range °C	Fraction III DFA %	Fraction IV Temp. range °C	Fraction IV DFA %	Product recovery DFA %	Product recovery Residue %	Column hold up loss %
1	Laboratory distillation unit without any fractionation	below 206	80.0	-	-	-	-	-	-	80.0	18.0	2.0
2	Laboratory distillation unit with 12" high extended surface glass column	below 160	60.0	160-200	15.0	-	-	-	-	75.0	22.5	2.5
3	Laboratory distillation unit with 12" high extended surface glass column	below 140	18.0	140-170	40.0	170-180	10.0	180-196	7.0	75.0	22.0	3.0
4	Electrically heated Tower's fractionation unit 1 m high packed with glass beads	below 80	5.0	80-120	12.5	120-140	41.0	140-200	6.5	65.0	28.0	7.0
5	Arecanut fat split in two stages											
5	As in S.No. 4	below 100	10.0	100-180	59.0	180-200	6.5	-	-	75.5	18.0	6.5
	Pooled and split arecanut fatty acid distillation residue											
6	Laboratory distillation unit without fractionation	below 180	22.5	above 180	37.5	-	-	-	-	60.0	39.0	1.0

Notes:
1. Percentages reported above are on the wt. of acids taken
2. DFA = Distilled fatty acids
3. Actual column hold up in Tower's unit on extraction was found to be 11 g i.e. 5.5%, the rest being losses.
4. A reduced pressure of 3-5 mm Hg was maintained throughout in all distillations
5. See Table 5 for fatty acid composition

Table 5 Fatty acid composition of arecanut fat and distilled arecanut fatty acid fractions*

Fatty acid	Arecanut fat wt.%	Run No.1 Distilled fatty acids, wt.%	Run No.2 Fraction I, wt.%	Run No.2 Fraction II, wt.%	Run No.3 Fraction I, wt.%	Run No.3 Fraction II, wt.%	Run No.3 Fraction III, wt.%	Run No.3 Fraction IV, wt.%	Run No.4 Fraction I, wt.%	Run No.4 Fraction II, wt.%	Run No.4 Fraction III, wt.%	Run No.4 Fraction IV, wt.%	Run No.5 Fraction I, wt.%	Run No.5 Fraction II, wt.%	Run No.5 Fraction III, wt.%	Run No.5 Fraction I, wt.%	Run No.5 Fraction wt.%
8:0	0.8	0.8	-	-	-	-	-	-	-	-	-	-	0.5	0.9	-	-	-
10:0	2.2	3.5	-	-	-	-	-	-	Trace	-	-	-	2.0	1.2	-	-	-
12:0	14.1	24.0	22.4	15.0	53.6	-	3.9	5.1	90.7	17.1	25.5	11.2	66.1	10.4	33.3	10.6	1.3
14:0	51.2	52.0	66.3	48.7	46.4	82.9	12.3	16.1	9.3	82.9	66.5	14.6	31.4	79.4	23.1	57.3	22.3
16:0	13.5	12.0	9.9	23.9	-	17.1	47.3	20.3	-	-	8.0	27.8	-	3.0	22.8	20.6	32.6
18:0	1.1	0.5	1.4	Trace	-	-	6.7	5.3	-	-	-	4.1	-	1.5	3.7	1.1	2.6
18:1	10.3	5.0	-	12.4	-	-	28.8	42.3	-	-	-	35.6	-	3.6	17.1	10.0	37.6
18:2	6.8	2.2	-	-	-	-	1.0	10.9	-	-	-	6.7	-	-	-	0.7	3.1

* See Table 3 for distillation and fractionation conditions

4. DISCUSSION AND CONCLUSIONS

4.1 Fat content of different size fractions

Soxhlet extraction of different sieve fractions (Table 1) indicates that the harder portions of the arecanut containing coarser particles contained lesser amount of fat compared to the finer fractions. The fractions containing biggest particle (853 micron) contained 10.2% while the finest fraction contained 14.3% fat. This indicates the need for grinding the nuts without however causing problems due to fines.

It may be necessary to analyse arecanuts grown in different regions for the yield and quality of fat before the overall economics for extraction of fat can be assessed. It is also desirable to study the concentration of oil-cell locations and develop methods to concentrate these oil-cell-rich fractions.

4.2 Pretreatment

Difficulty was faced during flaking of arecanut in five-high crushing rolls due to wide variations in particle size distribution of the pieces obtained during cracking operation. Whenever a large particle is drawn into the first pair of rolls, more quantity of smaller sized material slips into the second roll space. The unit was overloaded and tripped. Further exhaustive studies are needed to evolve suitable cracking, grinding and flaking units so that meal with uniform size and minimum amount of fines can be obtained.

4.3 Solvent Extraction

Extraction (Table 5) showed that good quality arecanut fat could be obtained even from low quality arecanuts. The yield was 9.6% on the weight of cooked arecanut flakes with 11.0% moisture (10.8% on moisture-free basis). Residual fat content in the defatted meal (11.0% moisture) was 1.2% (1.35% on moisture-free basis). This is equivalent to 1.08% on arecanut basis after accounting for 10% tannins and 10% fat extracted. This is comparable to the calculated value of 1% for residual fat on dry arecanut basis (11.8% in Soxhlet extraction minus 10.8% extracted in pilot plant). This residual fat content could be brought down to less than 1.0% in commercial plants where continuous and counter current solvent extraction is carried out using hexane at elevated temperatures (around 45°C).

No difficulty is envisaged in alkali refining of arecanut fat, bleaching with activated earth and/or carbon and deodorisation, if necessary. It is expected that arecanut fat will be a very good substitute for ghee and vanaspati and can also be used in margarine manufacture. It can also be blended perhaps with other fats to give nutritious oil/fat blends.

4.4 Extraction of arecanut tannins

Since aqueous extraction of defatted arecanut meal resulted in fermentation, a mixed solvent of acetone and water in the ratio of 40:60 (v/v) was tried for extracting tannins. The yield of spray dried tannins was about 10% based on the weight of defatted meal with 11.0% moisture. The yield on dry basis is 11.2%. The content of tannins in arecanuts may vary from region to region and may depend on other factors. This needs further study. It is necessary to get the samples of the tannins evaluated with regard to their suitability in leather industry by the Central Leather Research Institute, Madras.

4.5 Arecanut fat splitting

From the data on single stage splitting, a pressure of 25 kg/cm^2, a fat to water ratio of 1:0.6 and a duration of 1.5 hours are recommended. Under these conditions a split of 90.2% was achieved (Table 2, Run No.3). When a higher pressure of 30 kg/cm^2 is employed a split of 90.8% was achieved in 1 hr (Table 2, Run No.5).

Two stage splitting is desirable since higher concentration of sweet water is obtained and is better for economic recovery of glycerol. Simultaneously 95-96% split (i.e. 5% high

than in single stage splitting) is attained. Higher recovery of fatty acids in the first distillation step leaving lesser residue for resplitting is also achieved. This slightly increases the handling capacity of the autoclave. Glycerol content in sweet water in a single stage splitting was 12.1% as against 14.0% for two stage splitting.

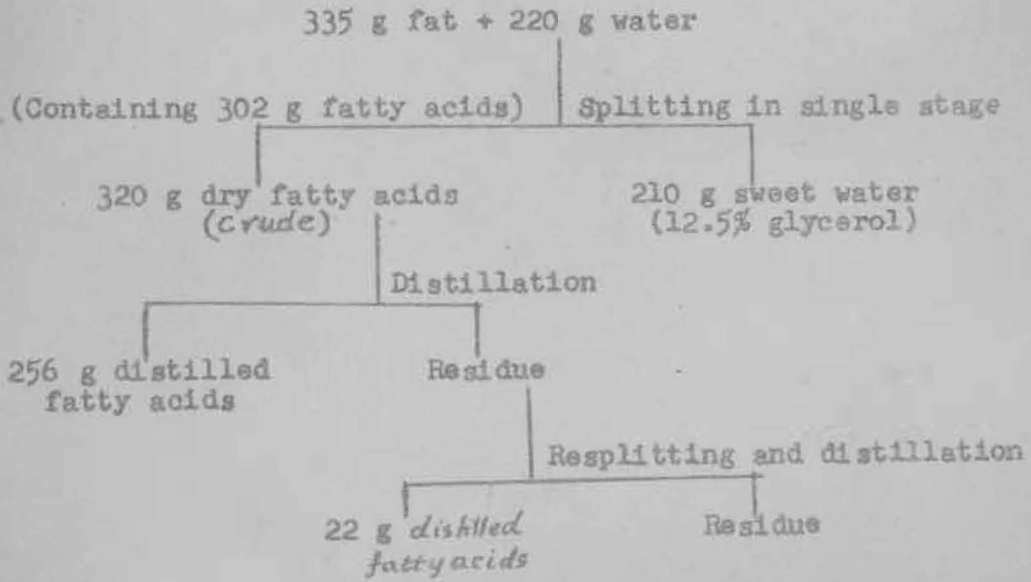
4.6 Distillation and fractionation of arecanut fatty acids

In the laboratory distillation unit, a yield of 80% of distilled fatty acids was obtained on the weight of dried crude fatty acids taken for distillation in the first stage. An additional yield of 11% of fatty acids was recovered by resplitting with residue followed by distillation (Runs 1 and 6 of Table 4). When fractionation was carried out in an extended surface glass column and in the Tower's fractionating unit packed with glass beads, the yields were 75% and 65% respectively, on the weight of crude fatty acids taken (Table 4). However, with arecanut fat split in two stages a yield of 75.5% of distilled fatty acids was obtained on the weight of crude fatty acids in the Tower's column as compared to 65% in the case of single stage splitting. It was found by extracting the material held in Tower's column with hexane that the hold-up was 5.5% (on the weight of crude fatty acids). Hence, the yield can be expected to be higher by 5.5% when a series of distillations are carried out (as the column hold up will be almost constant). Higher yields can also be expected

in large scale commercial distillation units which are provided with open steam arrangement.

4.7 Recovery of fatty acids

The recovery of fatty acids is shown below:



Thus from the available 302 g of fatty acids, 278 g (in the first distillation and 22 g from the split residue) were recovered on direct distillation accounting for 92.0% of the available fatty acids. This recovery, however, excludes hold up and the relatively lower boiling fractions which could be condensed and recovered in the commercial operations but were not done in the present investigation.

It will be seen from the composition of the fatty acid distillate fractions (Table 5) that fractions containing over 90% of lauric and myristic acids could be obtained which will be useful in replacing costly coconut fatty acids. These fractions could be used in the manufacture of soaps, fatty alcohols, esters (e.g., isopropyl myristate) and various surfactants needed in cosmetic, pharmaceutical, detergent and pesticide industries. Lauric and myristic acids are in great demand in the country but are in short supply for the manufacture of various derivatives. These acids and their derivatives have also excellent export potential.

4.8 Byproduct Utilization

4.8.1 Defatted and detanned arecanut meal: The use of defatted and detanned arecanut meal in compound feed formulations and fertilizers may be investigated at veterinary and agricultural research institutes.

4.8.2 Sweet water: Glycerol can be recovered from the sweet water containing 12-14% glycerol obtained during the splitting of arecanut fat. Quantities obtained in laboratory scale experiments are not sufficient for carrying out studies on glycerol recovery. However, no problem is anticipated in the recovery of glycerol from sweet water obtained from splitting of arecanut.

4.8.3 Supari manufacture: Good quality defatted but not detanned meal can also be used in scented supari manufacture. Such evaluation may be carried out by supari manufacturers.