

L104577



PATENT SPECIFICATION

DRAWINGS ATTACHED

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COMPLETE SPECIFICATION

Process for Extracting Oil from Palm Fruit and Olives

I, RAYMOND ARTHUR COUCHE, Australian citizen, of 168 Adelaide Terrace, Perth, State of Western Australia, Commonwealth of Australia, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for extracting oil from palm fruit and olives.

The present practice of oil extraction consists of pulping the fruit and pressing the oil and juice from the solid material with hydraulic or mechanical presses.

In the case of palm fruit the fruit are harvested in large bunches. Early sterilization is necessary to destroy enzyme activity which otherwise would degrade the oil. After sterilization, which also loosens the fruit in the bunch, the fruit is separated from the stalk by threshing, digested and pressed. The oil is recovered by settling from the press liquor or by centrifugation. The fruit pulp residue from the press is dried and the kernels are recovered as a separate product. The residual fibrous solid is either burnt as fuel or used as a plantation mulch.

In the case of olives the fruit are crushed lightly and oil and juice pressed therefrom, this pressing yielding the prime grade oil. The cake is broken up often mixed with some water and pressed again at a higher pressure to yield a second grade oil. Sometimes yet a third pressing at very high pressure yields a low grade oil. The remaining press cake may be dried and a low grade oil recovered by solvent extraction. The oil, other than that recovered by final solvent extraction, is recovered by settling or centrifugation. The solid material is either burnt or used as a plantation mulch.

The object of the present invention is to provide for direct solvent extraction of the fruit pulp and the substantially complete recovery of oil as prime grade from palm fruit and olives.

[Price 4s. 6d.]

Broadly the invention resides in a process for extracting oil from palm fruit or olives which comprises passing the fruit into a multi-stage counter-current flow with an organic solvent which is miscible with water and in which oil is soluble, the fruit pulp being disintegrated in at least the first stage with medium to high intensity mixing, sufficient to cause disintegration of the pericarp (fruit pulp) without significant breaking of the kernels, the temperature being such to maintain the oil being extracted in a liquid condition.

High intensity mixing is only essential for palm fruit. Olives can be processed in accordance with the invention with medium intensity mixing.

The organic solvents which are suitable for the purposes of the present invention comprise acetone or suitable mixtures of solvents such as ethyl alcohol, ethyl acetate and acetone in the volume ratio of 1:1:1, or ethyl alcohol, ethyl acetate and isopropyl ether in the volume ratio of 4:2:1, or other alternative mixtures all of which preferably have a boiling point or boiling range lower than 100°C.

It is generally understood that in the case of palm fruit the enzyme which causes rapid deterioration of the palm oil is destroyed when heated to 50°—55°C for about 30 minutes and for the purposes of the present invention it is generally accepted that extraction should be carried out at a temperature of between 50° and 55°C. In the case of olives no such enzymatic deterioration of oil is encountered but a similar operating temperature has advantages in more rapid extraction of oil from the pulp. The extraction should also be carried out at a temperature sufficient to maintain the oil being extracted in a liquid condition.

The mixing is carried out preferably in a cylindrical tank fitted with four vertical baffles spaced evenly around the tank wall and using an open turbine impeller of six to eight flat or curved blades placed vertically without pitch and located centrally in the tank. The mixing



centrifuges

5 impeller is driven at a suitable speed to pro-
 10 duce intense agitation in the case of palm fruit
 15 and medium agitation in the case of olives to
 20 cause disintegration of the pericarp or fruit
 25 pulp without breaking the kernels. The mixing
 30 tanks may be operated full with continuous
 35 feed and overflow. The sizes of the tank and
 40 mixer will depend upon the raw material input
 45 and retention time essential for effective dis-
 50 integration of the fruit. For fresh palm fruit
 55 a period of 15 minutes appears to be satis-
 60 factory while for sterilized palm fruit and
 olives a period of about 5 minutes should be
 suitable. The slurry passing out of the mixing
 tank consists of fruit pulp, oil, water, solvent
 and kernels; this slurry is passed over a screen
 to separate kernels and coarse fibrous solid, the
 undersized material passing to a centrifuge
 or alternative equipment for separation of
 solid and liquid. In the case of ~~centrifuges~~ these
 may be either basket type continuous feed
 units or alternatively solid bowl decanter units.
 The solid discharge passes to the second stage
 whilst the outgoing liquor is passed to the oil
 and solvent recovery section. The oil is separ-
 ated, preferably by centrifuge, from the
 aqueous-solvent liquor and stripped of solvent.
 The aqueous-solvent is distilled for recovery of
 the solvent and the aqueous solution discharged
 from the still utilized on the plantation. In the
 second and subsequent stages the mixing re-
 quired is not intense and while the general
 pattern of the mixer unit is retained a smaller
 impeller consuming less power is used. The
 solvent flows back to the second stage from
 the third and before entering the mixing tank
 it is used to spray wash the kernels and coarse
 fibre from the first stage screen. After a suit-
 able period of mixing the solid is again separ-
 ated from the liquid and passed to the third
 stage while the liquid passes to the first stage.
 The coarse solid and kernels are rinsed with
 each solvent liquor before it is passed into the
 mixing tank with the solids passing forward.
 The stage 4 liquor washes stage 2 coarse solids
 and kernels and mixes with stage 2 fine solids
 to form stage 3 whilst fresh solvent washes
 stage 3 coarse solid and kernels and mixes with
 stage 3 fine solids to produce stage 4. The
 stage 4 solids discharge to a solvent dryer
 for recovery of solvent. Additional stages may,
 if necessary be added to obtain the optimum
 recovery of oil.

55 The process is illustrated in the flow sheet
 shown in the accompanying drawing.

60 The various aspects of the present invention
 will be better understood by reference to the
 following description of several specific em-
 bodiments thereof as applied to a sample of
 fresh palm fruit and two samples of sterilized
 palm fruit.

A. Equipment.

A.1. *Disintegrator - Extractor.* This unit
 was a high intensity mixing vessel

65 which generated high shear forces in
 the region of the impeller. It was a
 70 7 inch diameter by 10 inch high
 vessel fitted with four vertical baffles
 of 0.75 inches width and spaced
 75 evenly round the tank wall. The mix-
 ing impeller was an open turbine
 with six flat blades all placed verti-
 cally and the impeller was placed
 centrally in the tank one inch above
 the tank bottom. The outside dia-
 80 meter of the turbine was 4.5 inches
 which allowed a clearance of 0.5
 inches between the turbine tip and
 baffles. The material of construction
 was mild steel.

The impeller was driven by a 0.5
 H.P. flameproof motor with speed
 control by means of a variable voltage
 control. The general operating speed
 was 800 r.p.m.

A.2. *Basket Centrifuge.* A 12 inch dia-
 meter by 4 inch stainless steel basket
 type bowl was fitted to an MSE Super
 Medium centrifuge and operated at
 2000 r.p.m. The basket had 2 mm
 90 diameter holes.

A.3. *Miscellaneous Equipment.* An electro-
 thermal heating tape was used to
 maintain temperature in the mixing
 vessel.

95 Several extraction stages where
 mixing was not required were carried
 out in a large stainless steel beaker
 and solvent was separated from the
 solid with a Buchner filter.

B. Procedure.

B.1. *Fresh Fruit.* One kilogram of fruit
 was extracted with one liter of solvent
 (acetone) with water added to bring
 the total water/solvent mass ratio to
 105 1/2, e.g.

1 kg. fruit contained an estimated
 200 g. water 200 g. water were
 added to the batch, and
 800 g. acetone were added, hence
 total water/acetone = 400/800 g.
 110 i.e. a ratio of 1/2.

This solution was placed in the
 mixing vessel and the temperature
 was adjusted to 50°C. With the
 mixer in operation the fruit was
 added and the mixing speed adjusted
 to be just not too violent. Mixing
 was continued for 15 minutes.

120 After the reaction period the liquid
 and solid were separated using the
 basket centrifuge which was pre-
 heated with boiling water. The solids

recovered were further extracted with one liter of acetone in the mixing vessel in order to complete the shredding of the fruit pulp and produce "clean" kernels. The solids and liquids were separated using a Buchner filter.

The solid residues were extracted twice more with fresh quantities of acetone using the stainless steel beaker method. The final solids were dried in an air oven at 90°C.

The liquors obtained were used in subsequent extractions in counter-current operation, the first stage liquor in each case being processed for oil recovery. The system was operated for a sufficient time to be nearly in equilibrium, and oil was recovered from stage 2 liquor prior to stage 1 extraction to evaluate the potential of such recovery. A smaller percentage was recovered from stage three liquors by cooling the liquor between stage 2 and stage 3. In practice the oil could all be passed forward into stage 1 or separated.

The liquors recovered by centrifugation were washed from the centrifuge using boiling water and the oil recovered from these liquors by settling at 80–90°C. for some hours. After cooling the aqueous layer was poured off leaving the solid layer of palm oil and fine solids. Clean water was added and the oil mixture melted and stirred in the clean water. Several hot water washes recovered a clean palm oil which was dried and stripped of residual solvent at 120°C., under 755 mm vacuum.

The oils were analysed for free fatty acid and compared with values obtained for the oil extracted from the original sample by means of boiling water. The technique adopted for these comparative oils was disintegration of the palm fruit pulp in boiling water, evaporation of the water on a steam bath and extraction of the dry solids with hexane. The hexane was evaporated and stripped under vacuum.

B2. Sterilized Fruit.

B2.1. *First sample.* The procedure outlined for fresh fruit in B.1. was used with the exception that reaction time in stage one was reduced to 5 minutes and stage 2 was carried out by the beaker technique.

The residual solids were analysed for oil content and the fine solids which passed 22 mesh screen were analysed for nitrogen.

In order to obtain the total oil yield for stage 1 the palm oil was settled hot and cooled. The solid palm oil was removed and the remaining sludge and aqueous layer was extracted several times with hexane until the hexane layer appeared to be free of oil. The hexane extracts were combined and the oil recovered by evaporation of the hexane and stripping under vacuum at 120°C.

The "dry sludge" was recovered and analysed for residual oil.

The aqueous liquor was evaporated to dryness to determine soluble solids.

B2.2. *Second Sample.* The procedure adopted with this sample was similar to the previous method but the water/solvent ratio in Stage 1 was adjusted to 1:1.

The re-cycle liquors were retained from the operation outlined in B2.1. and used directly at the commencement of operation with this sample. The stage 1 liquor was heated to boiling and re-centrifuged through the residual fibre bed already in the centrifuge basket. A light rinse with boiling water was used to displace oil from the fibre. This technique was adopted in an attempt to reduce the percentage of solids-not-oil which seemed to be high in the previous operation.

The stage 2 liquor was diluted with water to produce the ratio required for stage 1 and heated to 50°C. This dilution caused oil to precipitate due to reduced solubility. The oil was separated by gravity.

The stage 3 liquor from the last batch processed was cooled to precipitate oil insoluble at atmospheric temperatures (about 15°C). With continuous operation without cooling all the oil stayed in solution and passed into stage 2. This oil crystallised from solution and was separated by draining. The residual solvent content was evaporated and the oil was dried and stripped under vacuum.

The process was also varied as follows:—

The kernels were physically separated from the centrifuged solids from stage 1. Before passing the liquor into the residual solids for each stage, the kernels were washed with the re-cycle solvent of the particular stage.

The process also was extended to



outlined

5 six stages for more effective recovery of oil from the residual solids. The clean kernels and residual solids were de-solventised separately.

10 The oils were analysed for free fatty acid and the oil content of the residual solids was determined by Soxhlet extraction. Nitrogen was also determined on the -22 mesh solids. The solid content of the aqueous solvent ex stage 1 was not determined for this sample, nor was the oil content of sludge from stage 1.

C. Results.

C.1. *Fresh Palm Fruit.* After the first trial of fruit in the bunch (cut into small portions and including stalk cut into 2—3 cm lengths) all tests were restricted to fruit because of difficulty in disintegrating the stalk fibre.

The oil recovered contained a high percentage of free fatty acid and because this was unsatisfactory, detailed study was not completed.

The FFA value of oil samples obtained are shown in Table 1:

TABLE 1

F.F.A. as percent palmitic acid, in PALM OIL extracted from fresh palm fruit.

Sample	% FFA as palmitic acid
Fresh fruit, stage 1 oil extracted by solvent extraction as described above immediately after receipt (about 6 days after harvesting)	43.6
Fresh fruit extracted with boiling water. One day after solvent extraction as described above (fruit stored under refrigeration)	50.9
Fresh fruit 2 days after receipt sterilised at 60 psig for 30 min. then oil extracted	54.1

C.2. Sterilised fruit.

30 C2.1. *First sample.* A mass balance is shown in Table 2, together with head

sample analysis free fatty acid values on recovered oils are shown in Table 3.

TABLE 2

Process Mass Balance and Head Sample Analysis for Sterilised Palm Fruit

Mass Balance — Input — 1 kg. fruit.

Output (g)	Oil	Solids	Kernels	Water (by difference)
Stage 1	136.5			
„ 1 (Hexane extracted)	54.0			
„ 2	106.0			
„ 3	50.0			
„ 1 sludge	100.0	20.0		
+ 22 mesh residue	0.4	37.6	308.0	
— 22 mesh residue	1.9	28.1		
Totals	448.8	85.7	308.0	157.5



	Oil	Solids	Kernels	Water (by difference)
<u>Head Samples (Percent)</u>				
1.	46.8	12.4	23.0	17.8
2.	46 $\frac{1}{5}$	13.5	22.0	18.0

TABLE 3

Free Fatty Acid Content of Oils from Sterilised Palm Fruit

Sample	% FFA as Palmitic Acid
Head (before solvent extraction)	2.25
Stage 1	2.28
Stage 2	2.00
Stage 3	0.42
Head (after completion of Solvent extraction)	2.70

The nitrogen content of the fine solids (minus 22 mesh) was 2.17 percent as determined by Kjeldahl analysis. Other possible constituents such as phosphate were not determined.

The oil content of residual solids was:

+ 22 mesh 1.1%
- 22 mesh 6.3%
dry sludge 83.4%

C.2.1. *Second Sample.* A detailed mass balance was not determined with this sample. Yield data and an analysis of a head sample are shown in Table 4. Free fatty acid determinations are shown in Table 5.

TABLE 4

Approximate Product Yields and Head Analysis for Sterilised Fresh Fruit (Percent)

	Yields	Head
Kernels	35.0	29.0
Fibre and fine solids	5.0	11.0
Solids in Sludge (estimated)	1.0	
Oil — recovered Stage 1	20.0	45.5 (total oil)
Stage 2	2.0	
Stage 3	3.0	
Residual solids (oil)	0.1	
Balance (by difference)	33.9	14.5 (water)
	100.0	100.0

{ oil
sludge
water



TABLE 5

Free Fatty Acid content of oil from sterilised fresh palm fruit

Sample	Percent Palmitic Acid
Head	1.02
Stage 1	0.63
„ 2	0.75
„ 3	0.05

The nitrogen value of the fine solids was 2.18 percent by Kjeldahl analysis.

The oil in the residual solids was:—

+ 22 mesh : 1.0%
— 22 mesh : 1.5%

D. Discussion of Results.

D.1. *Fresh Fruit.* The high value obtained from fruit sterilised prior to analysing for FFA indicated that the fresh fruit sample was in a severe state of deterioration when received.

Disintegration of the stalk was difficult in the system of fluid mixing described above. While this may have been partly due to the small scale of operation, it is certain that to be effective in practice, reasonable chipping with cutting knives or alternative size reduction may be essential to ensure suspension in the slurry.

The fruit disintegrated reasonably easily but it was necessary to use two stages of fairly intense mixing to adequately strip the pericarp from the kernel. The dry solid residues and kernels were separated readily by screening.

D.2. Sterilised Fruit.

D2.1. *Digestion-Extraction.* In the case of sterilised fruit, disintegration was easy and one stage of mixing was sufficient to adequately strip the pericarp from the kernel. At the same time lower agitation intensity was used indicating low power requirements for this operation. Separation of the kernels from the stage 1 slurry was simple by wet screening.

Immediate separation and separate processing of kernels and fibre solids in the subsequent stages is desirable.

It may be desirable to separate coarse fibre from the fine solid at

this stage because the majority of residual oil in the solids appeared to be in the fine solids. The practical advantage with this technique is the ready removal of residual oil from kernel and fibre by a simple washing system on a screen deck while the fine solids require longer reaction time and positive separation from the solvent.

D2.2. *Oil Recovery.* The first stage liquor required separation from fibrous solids to enable a ready separation of the oil. In commercial operation separation would be carried out using a solid bowl centrifuge similar to the Westfalia decanter or a vibrating screen as used at present to remove fibre from the oil. The solids would be returned into the second stage of extraction. More efficient separation of the hot liquor could be achieved using an automatic desludging centrifuge, the solids again being returned to stage 2, the oil passing to a solvent stripping column and the aqueous solvent passing to solvent recovery.

The second stage oil seemed to separate satisfactorily and rapidly under gravity and use of a settling tank is possible. Addition of the optimum water requirement for stage 1 to the liquor prior to entering stage 1 would ensure a maximum yield of stage 2 oil. If oil is required from stage 3 it is desirable to cool the outgoing liquor from this stage to precipitate oil readily and remove it by settling.

Variation in the percentage recovery between the three stages may be obtained by variation of the process conditions. As shown in Table 4 the yield of oil from both stages 2 and 3 was lower than that shown in Table 2. This may have been



caused by the boiling water rinse adopted to displace oil from the "bed" of fibre in the centrifuge. Similarly a re-cycle liquor rinse may be used to reduce or control the quantity of oil carried forward with the solids.

The only loss from the process is that retained in the solid residue leaving the final extraction stage.

It may be expedient to recover all the oil in one stage only because separate recoveries require separate stripping units and there may be little advantage to be gained by separate recovery. The most notable improvement however was in the third stage oil, but the overall percentage yield in this stage was low.

All samples of oil bleached readily.

E. Olives. The following example illustrates the application of the invention to olives:

E.1. Material. Fresh olives were processed for the recovery of olive oil.

E.2. Equipment. The equipment used was similar to that described for palm fruit.

E.3. Procedure. 1 kg. of fruit was mixed for 5 minutes under mixing conditions imposing moderate fluid shear on the system. The solvent mass was determined by the solvent/water mass ratio of 2/1 and the water content of the fruit thus 1240 g. solvent was added for the water content of 62%.

The temperature of the reaction was above 25°C.

The liquor was separated from the solids and the solids further processed in a countercurrent system at 45—50°C to recover the oil retained in these solids. A total of four stages of extraction were used.

Oil was recovered by two techniques.

(i) by separation from the solvent by settling, decanting and stripping of solvent from the oil by steam;

(ii) by separation from the aqueous liquor remaining after solvent recovery.

The solids were dried and recovered.

E.4. Results.

E.4.1. Process Yields compared with Head.

	Process	Head
Oil	13.6%	11.9%
Pips	12.0	26.1 (pips and solids)
Solids	11.0	
Oil-sludge	0.7	
Water (by difference)	62.7	62.0 (direct evaluation)

E.4.2. Oil.

(i) *Recovered by separation and stripping of solvent.*

The oil was very pale yellow and similar to first quality virgin oil in appearance, taste and bouquet.

It contained free fatty acids equal to 0.11% expressed as oleic acid and possessed a refractive index at 15°C of 1.4701.

(ii) *Recovered after distillation of solvent.*

This oil was darker than the oil which was separated from the aqueous solvent. It was similar to commercial olive oil in appearance and taste and it retained a distinct fruity bouquet.

It gave the following analysis:

Free fatty acid (as oleic acid)	: 0.48%
Refractive index at 15°C	: 1.4701
S.G. 15.5/15.5	: 0.918
Weight per ml at 20°C	= 0.914
Iodine Value (Hanus)	= 83
Saponification	= 193

WHAT I CLAIM IS:—

(1) A process for extracting oil from palm fruit or olives which comprises passing the fruit into a multi-stage counter-current flow with an organic solvent which is miscible with water and in which oil is soluble the fruit pulp being disintegrated in at least the first stage with medium to high intensity mixing sufficient to cause disintegration of the pericarp (fruit pulp) without significant breaking of the kernels, the temperature being such to maintain the oil being extracted in a liquid condition.



gave 75



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- (2) A process as claimed in Claim 1 wherein the solid material is separated from the extraction liquor before being passed to the next stage. 25
- 5 (3) A process as claimed in Claim 2 wherein the separated extraction liquor flowing from one stage to the preceding stage is used to wash the solid material passing from said preceding stage to said one stage. 30
- 10 (4) A process as claimed in any one of the preceding claims wherein the outgoing extraction liquor from the first stage is treated to separate the oil from the aqueous solvent liquor and said separated oil is stripped of solvent. 35
- 15 (5) A process as claimed in Claim 2, 3 or 4, wherein the slurry of extraction liquor and solid material from the first stage is treated to separate the kernels and coarse fibrous solid material and the fine or undersized solid material is separated from the extraction liquor before being passed to the next stage. 40
- 20 (6) A process as claimed in any one of the preceding claims wherein the liquor passing out of at least one stage is treated to remove free oil which separates therefrom before said liquor is passed to the preceding stage.
- (7) A process as claimed in any one of the preceding claims wherein the outgoing liquor from the first stage is distilled to remove the solvent to produce an aqueous liquor and oil from which the oil is subsequently separated.
- (8) A process as claimed in any one of the preceding claims wherein the solvent is acetone or a mixture of ethyl alcohol, ethyl acetate and acetone in the ratio of 1:1:1, by volume or a mixture of ethyl alcohol, ethyl acetate and isopropyl ether in the ratio of 4:2:1 by volume.
- (9) A process as claimed in any one of the preceding claims wherein the extraction is carried out at a temperature of between 50° and 55°C.
- (10) A process for extracting oil from palm fruit and olives substantially as herein described.

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