Copra Quality Under New Rules—Importance Of Sampling and Analysis

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SPECIAL rules for controlling copra quality by means of chemical analysis have been adopted by the National Institute of Oilseed Products effective February 1, 1940, in part as follows:

> "The free fatty acids contents should not exceed 5%, and the resultant oil shall in no case be of a deeper color than 50 yellow and 9 red Lovibond Scale."

This is a striking departure from the older trade custom of evaluating this commodity on its physical appearances, and, as with any standards based on analysis, the ultimate value of the system depends on the representative character of the samples taken and their preparation and analysis. In fact, the entire subject of copra inspection assumes far greater importance now than formerly, and it becomes doubly important to adhere closely to proven methods that have been demonstrated to give results comparable with practical crushing. Without this thoroughness and accuracy all else that follows in the transaction fails in its purpose and will not provide a trustworthy basis for settlement.

In the past, analyses to reflect the quality of shipments had no accepted trade recognition, and were employed in commercial transactions simply to emphasize or identify disputed points of quality that were or were not indicated by appearances. Copra analyses, however, were recognized in cases of insurance and similar claims to reveal differences between the sound portion of a shipment and that damaged in transit, usually due to sweat, water leakage, etc. Copra analyses in more recent years have also been used by crushers as an advance guide before deliveries reached plants, and for factory control purposes.

The possible use and value of chemical analyses to determine quality of copra shipments were first investigated by us some thirty-two years ago and as a result of subsequent work in this field, various articles on copra and allied subjects were published (1919 to 1927) in columns of the American Oil Chemists' Society Journals (see 1 to 10 footnotes). The variability of component parts normally comprising shipments of this commodity presented a real problem in determining the best method of obtaining laboratory samples that would truly reflect the character of lots sometimes running into thousands of tons.

A further stage of this development was finding a method best suited to reduce large bulk samples amounting to 250 to 1000 lbs. to a state that would truly preserve the law of averages down to the final small amounts needed in the laboratory.

There was also the consideration of how best to obtain the oil for fatty acid determination, whether by pressing or extraction. If by pressing, it had to be as uniformly complete as practical crushing permitted and leave no more oil in the laboratory press cake than the approximate average of 7% remaining in factory press cake, a difficult accomplishment with laboratory facilities. If based on total extraction, then it required the application of some correction corresponding to the average oil left in the factory press cake, otherwise the results would not be comparable with factory runs, and they would not fully serve the purposes for which they were intended.

Most differences arising between factory crushing and laboratory results are due to not following proper and uniform methods of drawing and preparing samples and procuring and testing the oil. Steps necessary to this end were extensively reviewed some thirteen years ago in an article "COPRA SAMPLING, INSPECTION & ANALYSIS" (9), representing a revision of several earlier papers on related subjects (1 to 7 footnotes), and will not now require such lengthy discussion here.

Later research and changed conditions made it desirable to modify certain details of procedure not incorporated in former methods, but all basic requirements remain the same, and during these many years of practical demonstration in comparison with factory runs, it has not been found necessary to alter any fundamental practice. Before referring to present methods of inspection, however, a few facts about the characteristics of copra may be reviewed to advantage and will afford a better understanding of the subject as a whole.

We must keep in mind the physical state of any given copra shipment. It may consist of pieces varying from the size of a large hand to fines that will pass ⅛" screen and altogether will contain material reflecting all the variations that existed during the harvest and curing periods. Shipments usually represent copra collected from numerous plantations and may vary more or less widely from one lot to the other in the character of the oil which it will produce. These conditions are naturally responsible for an equally variable free fatty acid content of the various parts. From the largest to the smallest pieces and from the best to the poorest, a range of 3% to 60% free fatty acid in the oil may be encountered, yet the lot as a whole might average anywhere from 3 to 15% free fatty acid. On the other hand, there are shipments from different localities or countries of origin where the material is quite uniform in appearance and composition and consequently does not present the same difficulties in inspection. In other words, copra types and characteristics are an inseparable reflection of the producing district and the environments surrounding its harvesting and curing.

The conditions above outlined are probably more clearly indicated in tables A and B below, based on a "Manila Mixed Copra" consisting roughly of 75% Smoke-dried and 25% Sun-dried. This lot was selected for demonstrations, primarily on account of its abnormally large proportion of fines, the part always highest in fatty acids, as it illustrates the variable nature of the material that may comprise any given shipment, and no two deliveries are alike. The difficulty of drawing and preparing truly representative samples

of such a dissimilar mixture is best demonstrated by reviewing the following figures:

TABLE A

Sample partly separated on basis of appearances, into the following portions:

	% of Total Material	Total FFA in Oil
1. Best appearing pieces	2.3%	3.6%
2. Worst appearing pieces	2.7%	12.1%
3. Fines passing ¼" screen	6.4%*	38.2%
4. Balance of sample (average appearance)	88.6%	5.1%
	100.0%	
Algebraic average		6.9%
Algebraic composite of 1, 2 and 4 (excluding fines)		5.2%

* Abnormally large amount—responsible in this case for approximately ¼ of the total Free Fatty Acids.

TABLE B

Part of the same sample separated according to size of pieces regardless of quality appearance:

Size of Material	% of Total Material	Moisture	Ash,(Sand, Dirt,etc.)	Total Oil Recovered	Total FFA in Oil	% Total FFA Derived from Respective Portions
	%	%	%	%	%	%
1 Remaining on 1" screen (everything larger than 1")	49.1	3.88	2.02	66.89	4.4	31.3
2 Between 1" & ¾"	27.3	3.94	2.02	67.04	5.2	20.6
3 Between ¾" & ⅜"	15.5	3.66	2.18	67.23	8.0	18.0
4 Between ⅜" & ¼"	1.8	3.82	2.71	66.03	14.2	3.6
5 Between ¼" & ⅛"	0.9*	4.74	5.14	61.48	22.8	2.7
6 Passing ⅛"	5.4*	5.90	6.44	48.73	41.5	23.8
	100.0					100.0
Algebraic average		3.98	2.32	65.94	7.0	

* Abnormally large amount—responsible in this case for approximately ¼ of the total Free Fatty Acids.

Mainly to illustrate an extreme case in contrast to the above mixed copra, we cite a recent shipment of South Sea Sundried where the free fatty acids were only 2.6% as shown in Table C below. The fines passing a ¼" screen in this particular lot was only 0.47%, but had a free fatty acid content of 34.7%. If the fines had been omitted, however, the free fatty acids in the oil would have been 2.51% as against 2.60%, a marked contrast to the effect of the high percentage of fines indicated in Table A. While the difference is negligible in this case, it illustrates that even in the best grade of copra the oil in the fines is exceedingly high in fatty acid. If the percentage of fines had been high, they would obviously have had a much greater influence on the ultimate result. For each 1% of fines in this sample, the free fatty acid would be increased approximately 0.2%.

TABLE C

	% of Total Material	Total Oil Extracted	Total FFA in Oil
Coarse Material	99.53%	67.33%	2.51%
Fine Material (passing 4 mesh)	0.47%	35.29%	34.70%
Total	100.00%		
Algebraic average		67.19%	2.60%

The necessity for thoroughness throughout becomes more apparent when it is recognized that the free fatty acid and color in a partly expressed copra is usually higher than in one fully pressed. The difference is less pronounced with copra lower in free fatty acid and greater where it is higher. This is readily explained by the fact that in the exposed parts the tissues are softened and oxidized by elements of attack, and when in this state yield oil more freely, and are the portions highest in fatty acid and color. The harder and more resistant part adjacent to the shell, having better protection is lower in fatty acids and color and requires greater pressure to dislodge the oil, and is also the part most resistant to extraction as will be shown later.

Differences in the amount of oil pressed *from* copra have a marked influence on the results of analyses, as is demonstrated in the following table, where four copra grades have been expressed in three successive portions leaving roughly about 10% oil in the final cake of each:

TABLE D

Approximate Portions of Expressed Oil Tested (About 10% Left in Cake)

	First Third	Second Third	Last Third	Maximum Difference	Average of All Three
	% FFA	% FFA	% FFA	% FFA	% FFA
Sample 1	22.0	17.8	16.6	5.4	18.8
Sample 2	13.6	11.1	10.0	3.6	11.6
Sample 3	10.1	8.6	8.1	2.0	8.9
Sample 4	5.6	5.3	4.9	0.7	5.3

After many experiments made in an effort to express copra to the same practical limits obtained in factory crushing, we arrived at the conclusion that pressing to such a uniformly low oil content in the cake is not practical for routine laboratory procedure. The most consistent and dependable method of determining free fatty acids in the oil, as already pointed out in former articles on the subject, was found to be by extracting the copra with petroleum ether distilling below 40°C and correcting the free fatty acids to an average basis corresponding to approximately 7% oil left in the press cake, as will be referred to later in discussing methods.

To establish a suitable extraction system that would give results comparable to practical crushing, it had to be determined, among other things, whether it was necessary to dry the copra beforehand. Also the minimum time required for extraction; whether it could be adequately extracted without subsequent grinding with sand and further extracting; whether extraction could be so regulated as to permit leaving the equivalent of around 7% oil in the cake, as well as many other angles that would influence results and conclusions.

As a sequel to all this, it was determined that if 10 grams of a properly prepared copra sample were extracted for three hours, then thoroughly ground with sand and re-extracted one hour with fresh ether into a second flask, results were obtained that permitted for all practical purposes the necessary correction for oil normally left in press cake. Even after the second extraction there is usually around 0.2% to 0.5% oil still remaining, which, however, has no practical affect on the results. Figures in Table "E" show the uniformly low acidity of the oil recovered from the second extraction and the close agreement between the corrected laboratory result (corresponding to the normal 7% oil left in press cake) and free fatty acids found in the oil recovered from actual factory crushing.

TABLE E

	1st Extraction 3 Hours		2nd Extraction 1 Hour		Total of 1st & 2nd Extractions			
	% Oil Recovered	% FFA in Oil	% Oil Recovered	% FFA in Oil	% Oil Recovered	% FFA in Oil	% Corrected FFA	% FFA Determined on Factory Crushed Oil
1	59.97	5.88	5.92	0.95	65.89	5.44	5.6	5.8
2	65.40	9.08	3.43	1.65	68.83	8.71	8.9	8.9
3	66.39	7.60	3.42	1.65	69.81	7.31	7.5	7.4
4	65.24	7.05	2.87	1.97	68.11	6.83	7.0	7.2
5	62.84	5.11	3.89	1.46	66.73	4.90	5.0	5.1

It is obvious that comparisons are only possible when the entire lot is crushed in the factory at about the same time the laboratory sample representing the shipment

as a whole is examined. The opportunity for such comparisons is often lacking, as some part of the shipment may be crushed at one time and the balance at another. The present manner of discharging copra by the suction system creates more fines than are present at the time of shipment, and this is the part that increases in free fatty acids most rapidly. Furthermore, the copra may go to bins and remain for a considerable period before crushing takes place and even though later crushed as a whole, the result is apt to be somewhat higher in free fatty acids and color than if crushed shortly after discharge when the laboratory tests are made. More often, however, comparison is not possible because various lots of copra are mixed in bins and not crushed separately, but in Table "E" above the circumstances, both as to time, and crushing each lot in its entirety, enabled direct comparison of several recent shipments.

Methods of Sampling

Samples must be taken in uniform amounts at regular intervals during the entire discharge and in a manner that will preserve the true proportionate representation of all undamaged parts of the cargo as shipped. Portions found damaged due to conditions occurring in transit must be sampled and otherwise handled separately as far as practical discharge methods will permit.

The general method of sampling copra must naturally be adjusted to fit the manner or system of discharge prevailing at the several ports and plants, but in each case a uniform sample of 2 to 4 lbs. is taken at regular intervals according to the size and character of the shipment. If sampling is started with scoop or shovel (flat bottom and straight sides) arranged to hold about 2 lbs. of copra, this amount must be adhered to throughout, or if a scoop of 3 or 4 lbs. capacity is the desired amount, then the same capacity scoop or shovel must be used throughout the entire discharge for that particular lot.

a. If the method of discharge makes sampling necessary in the hold of the vessel, the sample should be taken near the suction intake at regular intervals during the entire discharge period.

b. If cargo is transferred by suction to dump trucks for delivery to plants, then each or every other truck may be sampled in equal amounts at time of discharge at plant rather than in the hold of the vessel.

c. If the suction line delivers from ship to hopper, belt conveyor, or bins, etc. then some convenient point in this system may be selected for a uniform sample taken at regular intervals.

The bulk sample taken as above described representing any particular lot, should be not less than 300 lbs. for small deliveries, to around 400 to 800 or even 1,000 lbs. for larger shipments. The bulk samples for purposes of meeting requirements of distribution are mixed and quartered down by shoveling over quartering irons until a final mathematical subdivision of two, three, and sometimes four, equal parts of 80 to 100 lbs. each have been procured, the balance being discarded. Each sample is placed in a paper-lined sack or large paper bag, or in a fine-grain burlap bag similar to a coffee sack. Lots between 1,000 and 3,000 tons should be represented by two distinct sets of samples. In other words, three thoroughly mixed and divided samples representing half the shipment and three sacks for the second half. One sack each goes to buyer, one each to Referee Chemist in case of claim, and the third of each set is held in reserve.

The fine material in copra has the lowest oil content and highest free fatty acid and color values, and is the most difficult portion to incorporate equally and uniformly in samples while in the crude state before cutting. The difficulties attached to preparing these samples, and the necessity for obtaining equal amounts of fines in each of the 80 to 100 lbs. subdivided samples, *cannot be overestimated* and without this accomplishment concordance and representative results will not be obtained.

Preparing Laboratory Samples

In order to maintain the law of averages throughout, so difficultly accomplished with small laboratory samples that are intended to represent correctly shipments of hundreds and sometimes thousands of tons of a variable material, three stages of preparation are necessary and equipment is required that *cuts* the copra but does not grind, crush or dislodge the oil which could not be uniformly reincorporated in the sample.

A. The entire 80 to 100 lbs. sample is put through a large copra cutter which conforms to the above requirements, or if two sacks represent the shipment, then each sack is put through separately. It is cut over the 1" screen, which reduces it to a state for easy and accurate mixing and subdivision over a "quartering iron" or "ripler." In reality the major part of the material passing the 1" screen is reduced to a much finer state than the mesh would indicate.

B. Two halves (40 to 50 lbs. from each sample or sackful) resulting from the first cutting (paragraph A), are then recut in the large cutter over the ¼" screen and thoroughly mixed and subdivided to any desired amount for the still finer and final cutting in the small copra shredder. For check purposes it is best to run each half (40 to 50 lbs.) as a separate unit.

C. The hopper of the small copra shredder, fitted with No. 10 disc (1/16" opening), is filled to near the level of the dividing cross-plate with the well-mixed and subdivided material from the ¼" screen referred to in paragraph B. The sample passing No. 10 disc after mixing is then ready for extraction or pressing.

Throughout these three cutting operations in two separate machines, the copra at no time becomes hot, nor is the oil dislodged, as the equipment cuts and does not crush or grind. After each cutting operation, material adhering to the machine should be removed and incorporated in the sample to which it belongs.

NOTE: *All shells, rocks, or metal pieces* have to be removed from any portion of the sample which is going to be used in either machine. This can be accomplished only by spreading the entire material on a well-lighted table and laboriously picking it over by hand. If this is not done, the knife edges of the copra cutter and more particularly the shredder discs will be damaged if not ruined completely. Information about satisfactory equipment can be obtained from the writer.

Methods of Analysis

EXTRACTION METHOD—The following extraction method for determining free fatty acids in the oil is the most consistent and dependable laboratory procedure we have been able to develop and it gives results agreeing closely with practical factory crushing:

10 grams of the *freshly* prepared sample are weighed into a paper thimble (22x80mm) and placed in a Smalley type extraction tube using a tared 100cc wide-mouth extraction flask containing a few porcelain chips. The copra is weighed and extracted without oven or desiccator drying. It is extracted with 40cc petroleum ether (Skellysolve F) at a rapid rate for three hours. Thimble

is then removed and contents finely ground with about 10 grams of treated fat-free sand (16 to 30 mesh) in a porcelain mortar. Mortar and pestle are rinsed with petroleum ether into the thimble after the ground material has been returned and the thimble is re-extracted for one hour using a *second* tared flask to recover the final oil. A fat-free cotton plug is used to cover copra in thimble during both extractions. Ether is evaporated from the two flasks on the steam bath, and the oils dried for 60 minutes and 30 minutes respectively with the bottoms of the flasks exposed directly to the live steam on the steam bath. At the end of their respective drying periods, the two flasks are removed from the steam bath, wiped dry and allowed to cool, and reweighed. 25cc of neutral alcohol is added, the flasks warmed and titrated with N/10 NaOH.

It is to be noted in the above connection that there is generally about 0.3% oil still left in the cake after the second extraction, corresponding to about 1% of oil in a factory press cake, as the cake is roughly one-third of the original copra. This amount of residual oil (0.3%) or its fatty acid content has no practical bearing on the fatty acid determination, but should be determined if the total oil content of the copra is to be reported.

The free fatty acid in each portion of extracted oil is calculated. It is pertinent to note here that the F.F.A. of the second portion is always far less than that of the first, corresponding to the known fact that the first oil pressed from a copra is always more acid than the following portions. If the total F.F.A. in the copra is desired, the results are averaged algebraically. To correct the results to correspond with average oil content remaining in factory press cake, it is assumed that 2.3% of the original oil (representing about 7% of oil in the press cake) is left in the cake. In calculating the corrected F.F.A., the F.F.A. of all the first oil extracted and the F.F.A. of the second oil less 2% (see example below) are averaged algebraically. (This 2% plus the 0.3% still remaining in the cake making up the 2.3% assumed to be left in an average factory pressed copra). Results are reported to the nearest 0.1%.

It should be observed in the above connection, that the finely ground sample prepared for laboratory use is not stable. Analyses will show increasing F.F.A. as time goes on, often showing considerable increase even after the lapse of only one day, thus making laboratory rechecks on the prepared sample unreliable unless undertaken promptly. Therefore, the first and second extractions should always be completed and weighed the same day the sample is prepared.

EXAMPLE

Results from 10 Gram Sample

	Oil Recovery	F.F.A. in Oil
1st extraction	6.150 grams (61.50%)	6.45%
2nd extraction	0.539 grams (5.39%)	1.05%
Total	6.689 grams (66.89%)	6.02% (Algebraic average)

$$\text{Corrected F.F.A. to compare with 7\% oil in cake} = \frac{0.615 + 6.45\% + (0.0539 - 0.02 \times 1.05\%}{(0.6689 - 0.02)} = 6.17\%$$

(Reported as 6.2%)

LABORATORY PRESSING—Oil pressed on a laboratory scale involves difficulties hard to overcome when compared with factory runs, as the oil left in laboratory press cake must not be much over factory averages of around 7%, something not easily or uniformly accomplished with laboratory equipment. Furthermore, if the oil is not adequately removed from the cake the resulting free fatty acids and color of the oil will not be representative, as the first oil pressed is the highest in free fatty acids and color. The color of course, is determined on the pressed oil and while influenced by not pressing to the usual practical factory limits, there is no penalty provided when the color exceeds the 50 yellow and 9 red referred to in the present rules.

If any of the final sample passing the No. 10 disc of the small copra shredder is used for pressing, the sample should be handled somewhat as follows:

About half fill a friction-top can of sufficient capacity and place in some air oven at not over 150°F and heat a few minutes with occasional mixing, rolling and turning. When the copra is fully and uniformly heated all the way through, place in suitable press equipment, the cage and ram, etc. of which have also previously been heated.

Unless one is familiar with the variety and types of copra shipments entering the market, some of the foregoing may appear superfluous, but where practical operations and results have been observed over a long period, the necessity for carrying out all the above suggested provisions becomes much more apparent.

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