# Bernam Bleachability Test Method for Palm Oil

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## ABSTRACT

A bleachability test method called the Bernam method is proposed for crude palm oil, based upon the percentage of color removed on the original color, as measured on a Lovibond 1 in. cell. The test uses absorption bleaching by 3% activated fuller's earth (Fulmont 237) at 150 C under inert gas blanket. It shows improved sensitivity over two existing bleachability methods and to both primary and secondary oxidation of palm oil, as measured by the peroxide, anisidine, and total oxidation values. It has been applied successfully to detect changes in the quality of palm oil during production, storage, and shipment to the consumer. In view of its encouraging response to oxidation, it should be possible for the consumer to relate the method to process conditions in the refining of palm oil.

## INTRODUCTION

The phenomenal growth of the palm oil industry in Malaysia, estimated to reach a record output of over 2.0 million tons in 1980, has been the result of major agronomical research since the dawn of the century, followed by important technological developments in processing during the past 2 decades (1).

The increasing volume of palm oil exports and the ever growing competition from other edible oils have spurred producers to a critical examination of the factors that govern its stability during production, storage, and shipment to the consumer (2).

In addition to the established analytical methods for determination of free fatty acids (FFA) moisture and insoluble impurities, the primary and secondary oxidation of crude palm oil has been evaluated in the prediction of its quality (3). The peroxide value, benzidine or anisidine value, and total oxidation value (2 PVe + AV) coupled with UV absorbance at 233 nm and 269 nm have proved useful methods in determination of the state of oxidation in palm oil, both for the producer and consumer.

However, the nature of oxidation in palm oil, as in other edible oils, is a complex phenomenon due to the interaction of oxidation products of carotene which are catalyzed by the presence of prooxidants. Hence, indirect methods for measurement of palm oil stability, like those of bleachability, which is influenced by all factors promoting oxidation, will continue to be required in any complete assessment of quality.

A number of bleachability tests has been developed for crude palm oil (4), mostly by the consumer, to grade palm oil from various sources. The notable among these are: (A) the earth bleaching and the combined heat and earth bleaching tests, developed primarily for Nigerian palm oil by the Tropical Products Institute, London, England (4); (B) the Edible Oil Refiners and Hardeners Association test in the United Kingdom (4); (C) the standard heat bleaching testing for special prime bleach (SPB) palm oil from the Congo developed in Belgium (B. Jacobsberg, Institut des Industries de Fermentation, Institut Meurice Chemie, unpublished data), which is popularly referred to as the Unilever method; and (D) the rapid bleachability heat test for crude palm oil developed at Hobum, Germany (J. Baltes, unpublished data), which is referred to as the Hobum method in this text.

Research was begun in 1968 at the United Plantations Quality Control Research Laboratory at Ulu Bernam, Malaysia, to develop a simple routine test method for bleachability that could be applied to the grades of palm oil produced at the United Plantations' factories. Initial trials with the existing methods mentioned above indicated that differences in bleachability were comparatively small within a limited quality interval. As a consequence, a new test method called the Bernam bleachability test was established, where crude palm oil is bleached with 3% activated fuller's earth (Fulmont 237) for 1 hr at 150 C under a carbon dioxide blanket. It is hoped that the method can be correlated with refining characteristics of crude palm oil in the near future.

## DETAILS OF APPARATUS AND DESCRIPTION OF METHOD

### Apparatus

*Bleaching flask:* Pyrex 250 ml flat-bottomed wide-mouthed conical flask with a Teflon cover is found suitable. (Pyrex catalog no. 1140/08). The cover has 2 holes, one for the stirrer and the other to hold a glass tube that extends to ca. 4 cm (Fig. 1).

*Heating bath*: HBIX Grant thermostatically controlled high temperature bath with UCEPAL-PON silicone fluid has been found suitable to maintain the temperature accurately at 150 C ± 0.5 C.

*Cooling bath*: This consists of a stainless steel container (17 cm in diameter and 7 cm high) with ca. 1 liter of suitable mineral oil, e.g. Voluta oil-45 marketed by Shell.

*Carbon dioxide*: Commercial grade carbon dioxide available in 40 or 60 lb cylinders is regulated using a $CO_2$ regulator (type SR-302 no. 0780-0354) before entering a calibrated gas flowmeter. The flowmeter is a glass U-tube manome' 'r across the top of which is connected a short length of precision bore capillary (Gallenkamp catalog no. FL 550). From the flowmeter, the gas flows through 2 gas wash bottles (each 250 ml) containing concentrated sulphuric acid to remove traces of water and then to another gas wash bottle (500 ml) with glass wool to prevent overflow of any acid vapors into the bleaching flask (Fig. 2). The flow rate of $CO_2$ is maintained at ca. 750 ml/min throughout the test.

*Bleaching earth*: Activiated bleaching earth, grade Fulmont 237, is used. Polythene packets (3 g) of this earth may be prepared in advance. The packets are heat-sealed and stored in airtight containers. Moisture in the earth should not exceed 10%.

*Stirrer*: Gallenkamp multispeed stirrer (catalog no. SS 564) is found suitable for stirring at 350-400 rpm.

*Filter funnel*: Glass filter funnel with a diameter of 12 cm holding a Whatman no. 1 filter paper (18.5 cm) has been found suitable.

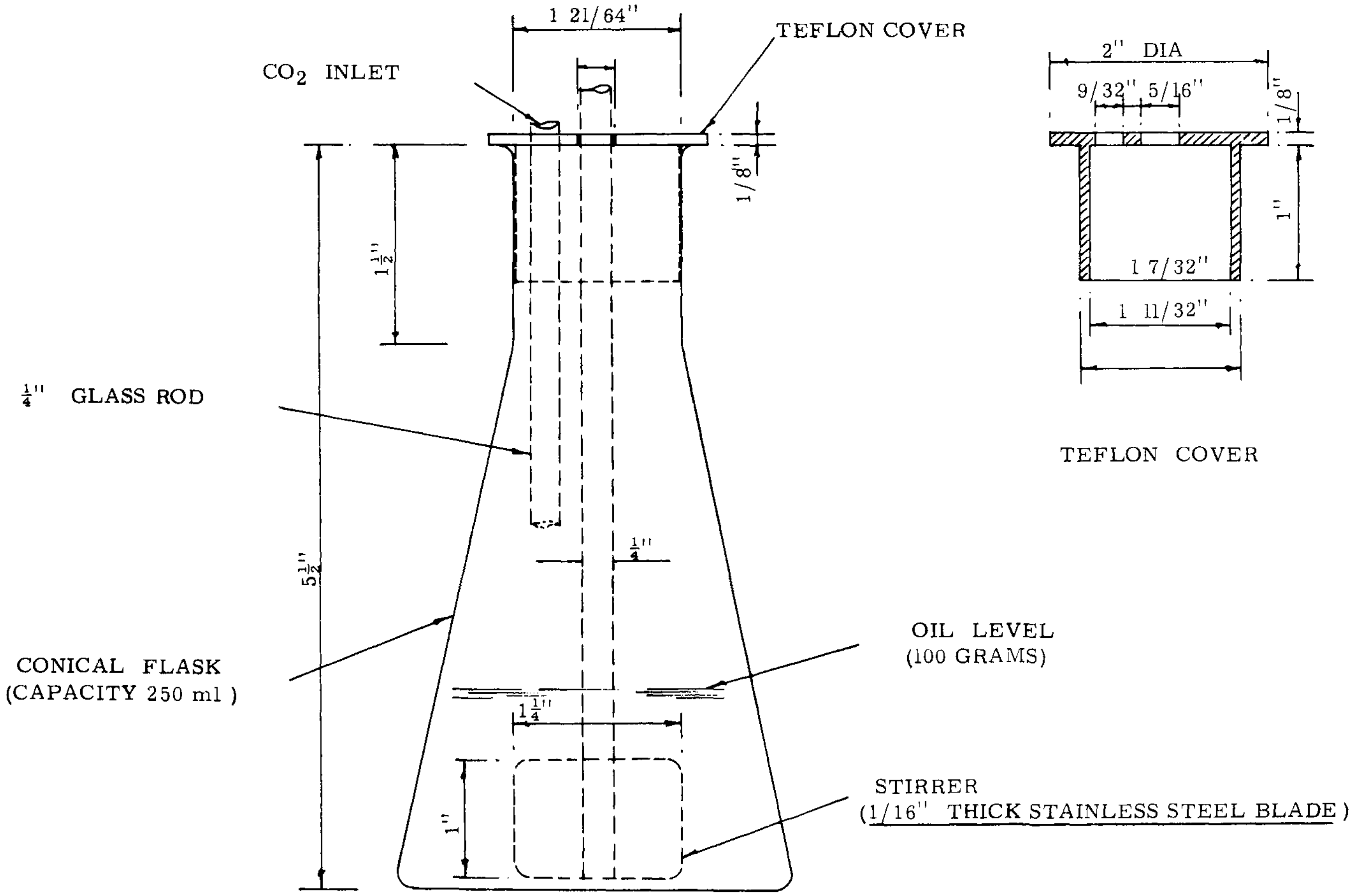


FIG. 1. Bleaching flask with Teflon cover.

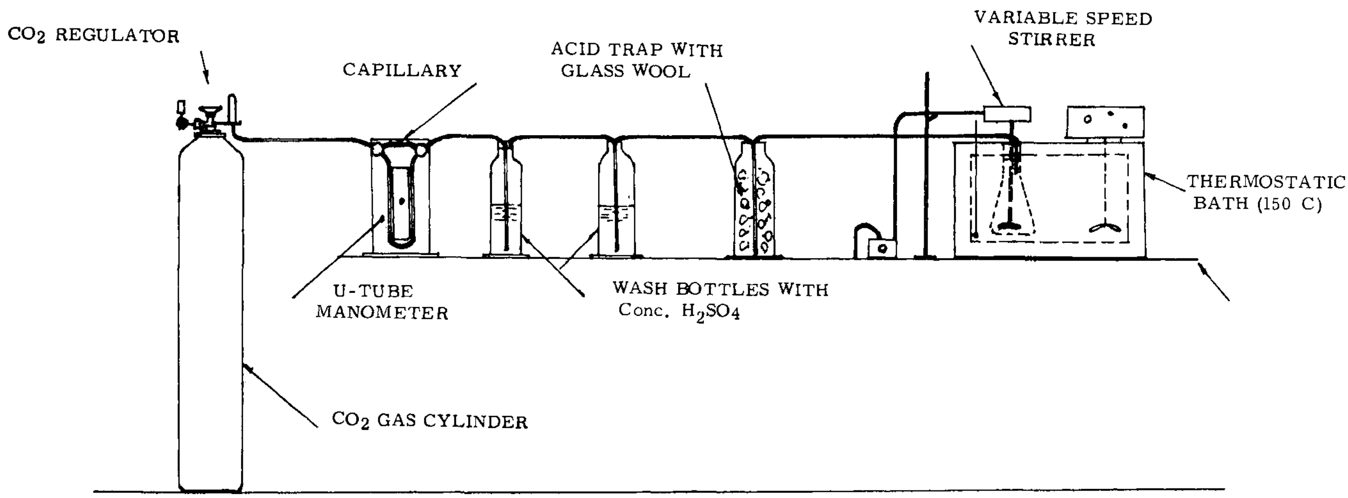


FIG. 2. Schematic diagram of Bernam bleachability test method (150 C/1 hr/3% earth).

*Air-oven*: An air-oven with accurate thermostatic controls, such as Memmert TV 30U has been found suitable.

### Procedure

The crude palm oil (100 ± 0.5 g) under test is weighed exactly into the bleaching flask. The flask then is fitted with the Teflon cover carrying the stirrer and gas inlet tube and bubbled with $CO_2$ for 5 min (flowrate, ca. 750 ml/min). The cover then is removed, and 3 g activated bleaching earth is added quickly. The closed flask is shaken gently until the earth is submerged and then immersed into the thermostatic bath maintained at 150 C; the stirrer is at 350-400 rpm. Care is taken to maintain the $CO_2$ flow throughout the test.

After exactly 1 hr, the stirring is stopped. The flask is removed with the cover, and the $CO_2$ flow is maintained while cooling in a stainless steel container with mineral oil. The flask, with contents, is swirled in the oil to accelerate cooling. After 5 min, the bleached oil is filtered at a temperature of 70 C using a filter funnel and Whatman no. 1 filter paper in a closed air-oven. When ca. 50 g oil has filtered, the residual color is observed on a 1 in. Lovibond cell at an oil temperature of 60 C.

### Residual Color

The residual color is reported in Lovibond units of 10 x red + yellow. Percentage color removed is as follows:

$$100\left[\frac{10\,(R+Y) - 10\,(R_1+Y_1)}{10\,(R+Y)}\right]$$

TABLE I

Comparative Analysis of Bernam, Unilever, and Hobum Bleachability Test Methods[a,b],

| Characteristics of crude oil | | | | | | | Bleached oil | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Lovibond color | | Lovibond color | | | Percent color removed | | |
| Free fatty acid (%) | Moisture (%) | Peroxide value (meq/kg) (AOCS) | Anisidine value (IUPAC)[c] | Total oxidation value (2 PVe + A V)[d] | 1 in. Cell | 5¼ in. Cell | Bernam method 1 in cell | Unilever method 5¼ in. cell | Hobum method 1 in. cell | Bernam method 1 in. cell | Unilever method 5¼ in. cell | Hobum method 1 in. cell |
| l.29 | 0.075 | 0.16 | 1.92 | 2.24 | 26R 24Y 1N | 66R 53Y | 0.2R 2Y | 1R 10Y | 1.0R 5Y | 98.59 | 97.19 | 94.72 |
| l.29 | 0.070 | 2.01 | 2.25 | 6.27 | 26R 24Y 1N | 66R 53Y | 0.3R 2Y | 1.1R 15Y | 1.1R 6Y | 97.88 | 96.35 | 94.06 |
| l.29 | 0.065 | 4.10 | 3.50 | 11.70 | 26R 24Y 1N | 66R 53Y | 0.5R 4Y | 1.4R 20Y | 1.2R 6Y | 96.83 | 95.25 | 93.66 |
| l.29 | 0.059 | 6.19 | 4.00 | 16.38 | 26R 24Y 1N | 66R 53Y | 0.8R 10Y | 1.8R 18Y | 1.2R 6Y | 93.66 | 94.95 | 93.66 |
| l.29 | 0.052 | 8.22 | 4.75 | 21.19 | 26R 24Y 1N | 66R 53Y | 1.2R 16Y | 2.0R 20Y | 1.4R 8Y | 90.14 | 94.38 | 92.25 |
| l.29 | 0.050 | 10.02 | 5.40 | 25.44 | 25R 24Y 1N | 64R 56Y | 1.5R 20Y | 2.2R 20Y | 1.3R 9Y | 87.22 | 93.96 | 91.96 |
| l.29 | 0.045 | 12.08 | 6.00 | 30.16 | 24R 23Y 1N | 62R 58Y | 1.8R 22Y | 2.4R 24Y | 1.3R 9Y | 84.79 | 92.92 | 91.63 |
| l.30 | 0.045 | 14.12 | 6.50 | 34.74 | 23R 23Y 1N | 60R 58Y | 2.1R 24Y | 2.6R 26Y | 1.5R 10Y | 82.21 | 92.09 | 90.12 |
| l.30 | 0.040 | 16.21 | 7.18 | 39.60 | 23R 23Y 1N | 58R 56Y | 2.3R 24Y | 2.7R 27Y | 1.7R 11Y | 81.42 | 91.50 | 89.33 |
| l.30 | 0.040 | 20.08 | 7.95 | 48.11 | 23R 22Y 1N | 56R 56Y | 2.3R 24Y | 3.4R 30Y | 1.8R 1.2Y | 81.34 | 89.61 | 88.10 |
| l.31 | 0.035 | 24.18 | 8.23 | 56.59 | 22R 22Y 1N | 54R 56Y | 2.4R 24Y | 3.5R 32Y | 1.9R 1.3Y | 80.16 | 88.75 | 86.81 |
| l.31 | 0.035 | 28.06 | 8.75 | 64.87 | 22R 20Y 1N | 54R 50Y | 2.5R 24Y | 3.8R 38Y | 2.0R 1.2Y | 79.58 | 87.11 | 86.67 |
| l.32 | 0.030 | 32.23 | 9.20 | 73.66 | 20R 20Y 1N | 50R 48Y | 2.6R 24Y | 4.7R 44Y | 2.0R 1.3Y | 77.27 | 83.39 | 85.00 |

[a]For the above table, correlation between peroxide value and bleachability: Bernam method (1 in. cell): $Y = 96.64 - 0.70 X$ and $r = -0.9379$; Unilever method (5¼ in. cell): $Y = 97.28 - 0.38 X$ and $r = 0.9873$; and Hobum method (1 in. cell): $Y = 94.90 - 0.314 X$ and $r = -0.9918$. Y = percent color removed, X = peroxide value (meq/Kg), and r = regression coefficient. Correlation between total oxidation value and bleachability: Bernam method (1 in. cell): $Y = 97.48 - 0.316 X_1$ and $r = -0.9372$; Unilever method (5¼ in. cell): $Y = 97.84 - 0.174 X_1$ and $r = -0.9763$; and Hobum method (1 in. cell): $Y = 95.27 - 0.1406 X_1$ and $r = -0.9919$. Y = percent color removed, $X_1$ = total oxidation value, and r = regression coefficient.

[b]Overall regression equations between peroxide value and bleachability: Bernam method (1 in. cell): $Y = 96.59 - 0.706 X$ and $r = -0.9389$; Unilever method (5¼ in. cell): $Y = 96.90 - 0.398 X$ and $r = 0.9831$; and Hobum method (1 in. cell): $Y = 94.78 - 0.3242 X$ and $r = -0.9895$. Y = percent color removed, X = peroxide value (meq/kg), and r = overall regression coefficient. Overall regression equations between total oxidation value and bleachability: Bernam method (1 in. cell): $Y = 97.54 - 0.319 X_1$ and $r = -0.9764$; Unilever method (5¼ in. cell): $Y = 97.42 - 0.179 X_1$ and $r = -0.9764$, and Hobum method (1 in. cell): $Y = 95.18 - 0.1447 X_1$ and $r = -0.9896$. Y = percent color removed, $X_1$ = total oxidation value = (2 PVe + A V), and r = overall regression coefficient.

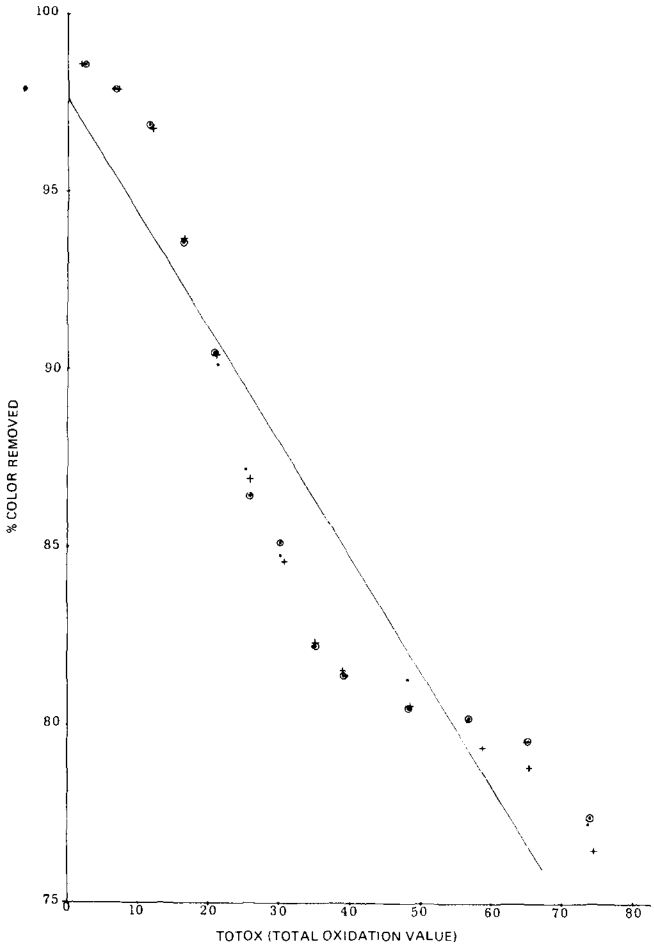


FIG. 3. Bernam bleachability test method (150 C/1 hr/3% earth). Relationship between total oxidation value and color removed (%). Overall regression equation: $Y = 97.48 - 0.319\ X_1$ and $r = -0.9764$. • = Free fatty acid, 1.29%; ⊛ = free fatty acid, 2.00%; and + = free fatty acid, 2.50%.

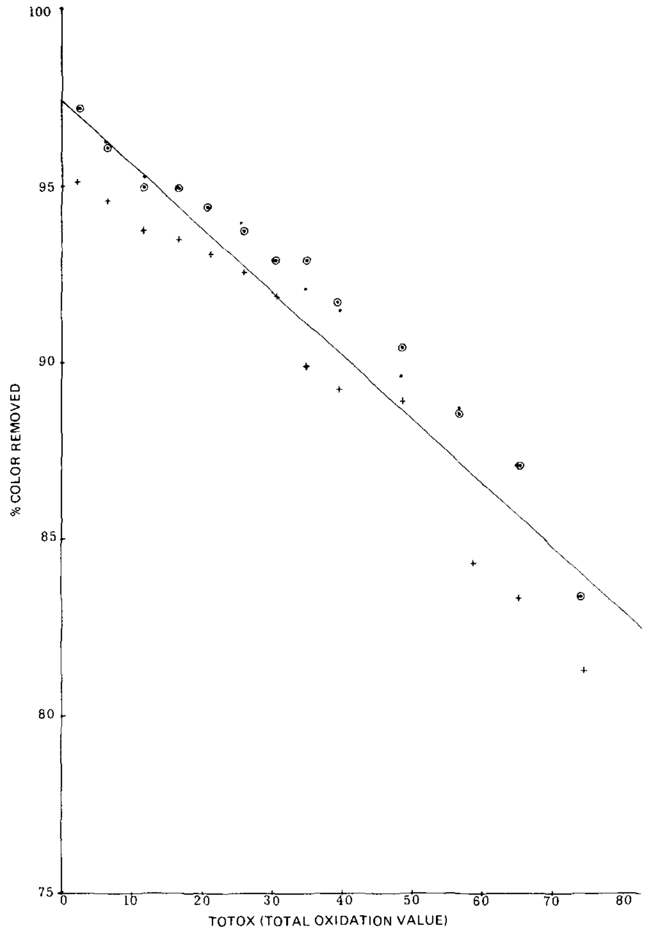


FIG. 4. Unilever bleachability test method. Relationship between total oxidation value and color removed (%). Overall regression equation: $Y = 97.42 - 0.179\ X_1$ and $r = -0.9764$. . = Free fatty acid, 1.29%; ⊛ = free fatty acid, 2.00%; and + = free fatty acid, 2.50%.

where 10 (R + Y) = total color of crude oil before bleaching, and 10 ($R_1$ + $Y_1$) = total color of bleached oil.

### Sensitivity to Oxidation

The sensitivity to oxidation of the Bernam bleachability test method was studied while comparing it under identical conditions for SPB oils with two other bleachability methods namely (A) the standard heat bleaching test for SPB oil (Unilever method) and (B) the rapid heat bleachability test for crude palm oil (Hobum method).

The Unilever method consists of heating crude palm oil for 1 hr at 240 C followed by addition 1% Tonsil standard bleaching earth and stirring at 110 C for 20 min. Both operations are conducted in a atmosphere of carbon dioxide. The residual color of the filtered oil is measured on a 5¼ in. Lovibond cell and should not exceed 2 red Lovibond. The yellow value should be ca. 10 times the red value.

The Hobum method subjects 50 cc filtered crude palm oil to 300 C for 45 min under a flow of carbon dioxide. The residual color is registered on Lovibond 1 in. cell (correction of color 1 blue = 5 red = 50 yellow).

The estimation of bleachability by the method is recorded as follows: below 2 red and 20 yellow, well bleachable; more than 2-3 red and 20-30 yellow, sufficiently bleachable; and more than 3 red and 30 yellow badly or very badly bleachable.

## EXPERIMENTAL DETAILS

Three samples of palm oil were drawn from the production line at the factory having an FFA of 1.29%, 2.00%, and 2.50%, respectively. Oxidation was induced artificially in the samples by controlled aeration at 90 C. Subsamples having varying peroxide and total oxidation values (2 PVe + AV) were withdrawn at regular intervals and tested for their bleachability by all three methods.

The color of the crude palm oil and the residual colors of the bleached oil were measured on a 1 in. or 5¼ in. Lovibond cell, as specified by the particular method.

To have an exact comparison of the methods, bleachability is expressed as percent of color removed over the total original color as measured in the specified Lovibond cell (1 or 5¼ in.).

The comparative results of bleachability between the three methods for the FFA of 1.29% are arranged in Table I. A similar relationship was obtained between the three methods for the other samples with FFA of 2.00% and 2.50%.

The regression equations correlating peroxide value and total oxidation value with percent color removed is given at the bottom of Table I. The correlation between total oxidation value and percent color removed also is illustrated graphically in Figures 3, 4, and 5.

## DISCUSSION OF RESULTS

All the methods were correlated significantly with peroxide value under conditions of the test.

However, if the slopes of the regression line are taken as an indication of sensitivity, the order would be as follows: Bernam slope, 0.706; Unilever slope, 0.398; and Hobum

TABLE II

Expected Changes in Bleachability for Bernam, Unilever, and Hobum Methods during Production, Storage, and Shipment of Palm Oil

| Stage | Peroxide value (meq/kg) (AOCS) | Anisidine value (IUPAC)[a] | Total oxidation value (2 PVe + AV)[b] | Bleachability (% color removed) Bernam method | Unilever method | Hobum method |
|---|---|---|---|---|---|---|
| Production and storage at factory | 0.20–2.00 | 1.50–2.50 | 1.90– 6.50 | 98.5–97.5 | 96.5–95.5 | 94.5–93.5 |
| Sotrage and ocean shipment from Bulking installation | 2.00–6.00 | 2.50–4.00 | 6.50–16.00 | 95.5–94.5 | 95.5–94.5 | 93.5–93.0 |
| Outturn on arrival overseas | 6.00–12.00 | 4.00–6.00 | 16.00–30.00 | 93.5–84.5 | 94.5–92.5 | 93.0–91.5 |

[a]IUPAC = International Union of Pure and Applied Chemists.
[b]PVe = peroxide value and AV = anisidine value.

slope, 0.324.

The sensitivity of the Bernam method was found to be higher than that of the Unilever and Hobum methods to changes in peroxide value. The overall regression equations correlating total oxidation value ($X_1$) with percent color removed ($Y_1$) for the three methods were as follows: Bernam method (1 in. Lovibond cell), Y = 97.48 – 0.319 $X_1$ and r = -0.9764; Unilever method (5¼ in. Lovibond cell), Y = 97.42 – 0.179 $X_1$ and r = -0.9764; and Hobum method (1 in. Lovibond cell), Y = 95.18 – 0.1447 $X_1$ and r = -0.9896. The three methods were correlated significantly with total oxidation value under conditions of the tests.

However, if the slopes of the regression line are taken as an indication of sensitivity, the order would be as follows: Bernam slope, 0.319; Unilver slope, 0.179; and Hobum slope, 0.145.

The sensitivity of the Bernam method is found to be higher than that of the Unilever and Hobum methods to changes in total oxidation value. However, all the methods do show reduced sensitivity to changes in total oxidation value when compared to that of peroxide value.

It is evident that the Bernam method is able to bring out quality differences in crude palm oil over a wide range of peroxide values and total oxidation values.

In contrast, the Unilever and, in particular, the Hobum methods show differences in bleachability only for a limited quality interval.

## PRACTICAL APPLICATION OF BLEACHABILITY

A bleachability test method to be of use to the producer should allow him to trace, without ambiguity, quality differences in palm oil during production at the factory, storage at the installation, and shipment by ocean tankers to the consumer. The time interval between production and outturn could vary from a few months to even a year, depending upon demand.

In Table II, an attempt has been made to predict oxidative changes in quality from production to outturn with bleachability using the three methods. The range of values recorded for peroxide, anisidine, and total oxidation values are estimated to be average figures for Malaysian palm oil.

It is seen from the table that the Bernam bleachability method clearly brings out the differences in quality of palm oil during production and shipment.

The differences in bleachability by the Bernam method are ca. 14.0% color removed between production and outturn, while those of Unilever and Hobum show marginal differences of 4.0 and 3.0%, respectively.

The encouraging response of the Bernam method to oxidation should make it possible for the consumer to relate it with the actual plant conditions during processing of the crude oil to various end products.

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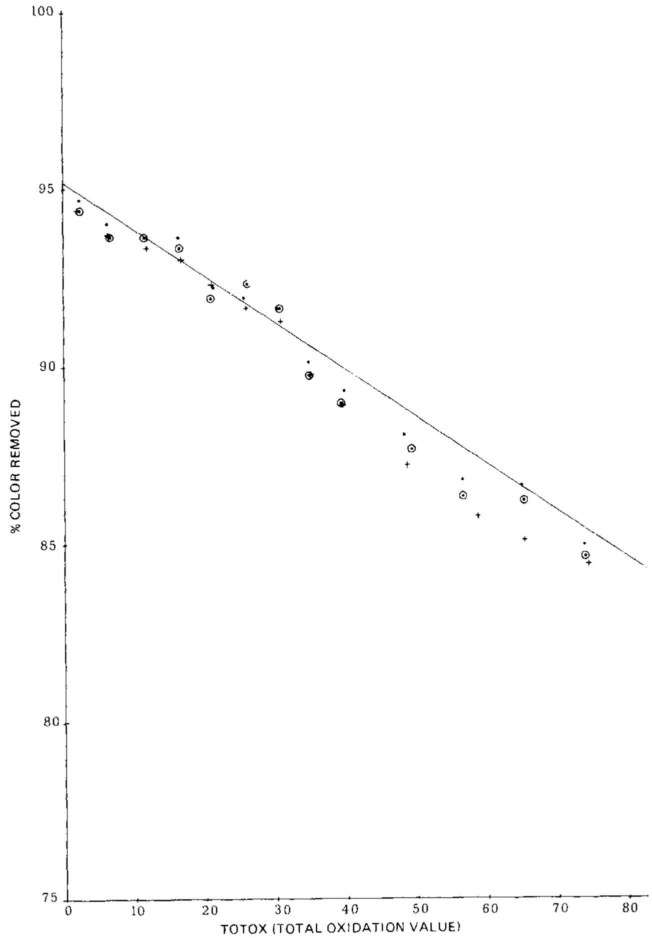


FIG. 5. Hobum bleachability test method. Relationship between total oxidation value and color removed (%). Overall regression equation: Y = 95.18 – 0.1447 $X_1$ and r = -0.9896. . = Free fatty acid, 1.29%; ⊙ = free fatty acid, 2.00%; and + = free fatty acid, 2.50%.

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