Comparative Evaluation of Palm Oil Color Measurement Using a Prototype Palm Oil Colorimeter

Yew-Ai Tana,*, Ainie Kuntom^a , Chak Khiam Leeb, and Kum Sang Low^c

^aMalaysian Palm Oil Board (MPOB), 43000 Kajang, Selangor, Malaysia, ^bSpektrum Saujana (M) Sdn. Bhd., 46000 Petaling Jaya, Selangor Darul Ehsan, Malaysia, and ^cLaser and Opto-electronics Laboratory, Institute for Post-Graduate Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

ABSTRACT: The color of refined palm oil and palm oil products is conventionally measured using the manually operated Lovibond[®] Tintometer. In the present study, one manual/visual and three automatic colorimeters for the measurement of vegetable oil color were used for color measurements of refined palm oil. All colorimeters used were commercially available instruments except for an automatic palm oil colorimeter developed specifically for the measurement of palm oil color. The color values obtained from all four instruments were compared using the visually obtained readings as reference values. Results showed that all three automatic instruments gave correlation coefficients of greater than 0.9300 for red color measurements. In addition, the Student t-test showed no difference between the analysis of red color using the visual method and the palm oil colorimeter. This investigation concludes that, although it is extremely difficult to reduce the lack of precision in color measurement of palm oil, a properly designed and calibrated automatic instrument may still be the better choice because reproducibility and repeatability are required in all standard test methods. The palm oil colorimeter offers a ready and relatively inexpensive solution to the problem of color matches based on visual observations.

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KEY WORDS: Color measurement, objective method, palm oil colorimeter, palm oil color, refined palm oil, visual method.

Color is an important indication of product composition, purity, and degree of deterioration. It is a quick check on degradation and the suitability and stability of the product for a particular use. In the case of vegetable oils, it is necessary to monitor each stage of the refining process to determine whether the required color has been obtained, as each type of oil will have its own "sell by color" specification. Frequent color measurements are often the key to considerable savings in time and bleaching earths that are used for refining vegetable oils. Hence, color measurement is used for quality monitoring, production control, and determination of final product conformance to predetermined color tolerance and of compliance with customer specifications.

Manually operated visual instruments have held the mo-

nopoly on color measurements for almost a century (1,2). Accordingly, conventional methods of color measurement are by visual comparative techniques. The product to be measured is compared against reference solutions (3) or colored glass (4,5). These methods tend to have poor reproducibility and are not useful for research purposes because the data are subjective and depend on the color vision and judgment of the observer.

Vegetable oil color is routinely measured using standard procedures set down in the early 1900s. The well-established subtractive colorimeter called the Lovibond® Tintometer (1) is used to measure oil color instrumentally, and results are reported as Lovibond values of red and yellow, plus blue or neutral if these are required for a good color match. This subjective method clearly depends on the analyst's judgment as well as on the type and model of the colorimeter used. As such, laboratory-to-laboratory variation in Lovibond color measurement can be rather large. Not withstanding this limitation, the Lovibond color standards are accepted throughout much of the vegetable oil trade as a proven means of assigning color values.

Visual methods are not always adequate for investigative purposes. For objective and unbiased assessment, automated colorimeters are called for. These instruments are usually designed to measure the transmitted color of optically clear liquids. For obvious reasons, the precision of color measurements with automatic colorimeters is better than that for manual operated instruments. However, the wide acceptance of visual methods is such that these automatic instruments require correlations with existing, visually obtained color data before they can be used confidently. Hence in 1988, Erickson conducted a color-in-oils study to compare color readings obtained using an automatic spectrophotometer (Tintometer Model AF 960) and the manual Lovibond Tintometer Model 710. Participants in the study were told to measure the color of oil samples in a 5 1/4-in. cell (13.3 cm) at a temperature of 60 ± 1 °C. At the time of the study, it was recommended to the Commercial Fats and Oils Analysis Committee of the American Oil Chemists' Society that the most appropriate action was to develop an AOCS Recommended Practice instructing laboratories how to form their own manual vs. automated studies and how to develop comparison charts (6).

Vegetable oil producers have long felt the need for simple and inexpensive instruments that are not dependent on visual

^{*}To whom correspondence should be addressed at Product Development and Advisory Services Unit, Malaysian Palm Oil Board, No. 6, Persiaran Institusi, Bandar Baru Bangi, 43000 Kajang, Selangor, Malaysia. E-mail: tan@mpob.gov.my

judgments for the determination of colors. Numerous proposals have been forwarded for methods to avoid the difficulties associated with visual methods of color measurement in vegetable oils and fats. Instruments based on light transmission for objective color measurement are available, but these are rarely used for edible oils and fats partly because of cost and the broad acceptance of manual color measurements. Based on results from a collaborative study in 1950, it appeared at the time that the AOCS spectrophotometric method Cc 13c-50 (7) was a potential alternative to the manual Lovibond method. However, to date, this method is not often used, probably because the manual method is still much easier to carry out. This paper discusses the evaluation of a palm oil colorimeter (8) for objective color measurement of refined palm oil and palm olein. A comparison was also made on the performance of the palm oil with commercially available automatic as well as manual colorimeters.

MATERIALS AND METHODS

Test samples. Refined palm oil (RPO) and refined palm olein (RPOo) were obtained from local refineries. To obtain RPO samples with a wide range of Lovibond red values, oils that had been stored for some time in an oven at $60 \pm 2^{\circ}$ C were used as test samples. In addition, freshly refined palm oil samples were also used, and these were obtained by laboratory refining (9) of crude palm oil.

Apparatus/instruments. Lovibond Tintometer Model F (The Tintometer Ltd., Salisbury, United Kingdom); Lovibond PFX 880 Series (The Tintometer Ltd.); LICO® 300 (Dr. Bruno Lange GmbH, Berlin, Germany) and the palm oil colorimeter designed and calibrated for palm oil color measurement. Glass cells, 5 1/4-in. and 1-in. (13.3 and 2.5 cm) path lengths, were used for containing the test samples.

Preparation of test samples. All test samples were homogenized to clear liquids by heating them in an oven at $60 \pm 2^{\circ}$ C.

Methods used for color measurement. The color of 30 samples of RPO and RPOo were analyzed using the Lovibond Tintometer Model F (4), Lovibond PFX 880 series, and the LICO 300 (according to the operator's instruction manual, respectively). Duplicate measurements were all carried out in using 5 1/4-in. glass cells.

Measurement of color using the palm oil colorimeter. The colorimeter is designed to determine the color intensity of palm oil by passing a beam of light from a light source through the oil and measuring the transmitted light with a silicon photo detector. The analogous output of the detector is then converted into digital mode in a 12-bit analog-to-digital converter. A microprocessor is used to process the digital data obtained against a predetermined color scale. The light source consists of red (R), green (G), and blue (B) light-emitting diodes. The microprocessor includes computer software that is capable of reading the digital signals and comparing them with a predetermined scale of color and path length. The processed data can then be displayed on a liquid crystal display (LCD) and/or recorded in the computer *via* an RS232

terminal. The predetermined scale is the Lovibond scale. The readings of the colorimeter must be calibrated, and appropriate software using an artificial neural network is used to calculate the red and yellow Lovibond value.

The process of estimating calibration values is termed "training." During the training process, the colorimeter is exposed to many oil samples (150) of known Lovibond values (as measured using the manual Lovibond Model F). During actual color measurement, the colorimeter will use the saved calibration values and measured red, green, and blue values (8) to calculate the Lovibond color value of the oil. Color readings of the RPO and RPOo samples were analyzed by the palm oil calorimeter after visual values were taken to ensure that the operator would not be influenced by the colorimeter results.

Repeatability of the palm oil colorimeter. The repeatability of the palm oil colorimeter was checked by measuring the oil color of two palm oil samples—A and B. The colors of these two oil samples were first determined visually by measurement using the Lovibond Tintometer Model F.

Comparison of commercial automatic and manual instruments. Color readings were taken on a wide range of oils using both the automatic and manual instruments. Results obtained from subjective manual measurement of oil color using Lovibond Tintometer Model F were compared against those from the objective and automatic readings with the Lovibond PFX 880 Series and LICO 300. All recommendations and calibration procedures as outlined in the operating manuals of the respective instruments were strictly adhered to. All color measurements taken were recorded for evaluation.

Comparison of palm oil colorimeter with commercial colorimeters. The commercial colorimeters referred to are the manually operated Lovibond Tintometer Model F, the automatic Lovibond PFX 880 Series, and LICO 300. The color values of RPO and RPOo were measured using all three instruments and the palm oil colorimeter.

Statistical analysis. All measurements were in duplicate. Results of color measurements including mean, SD, and linear regression analysis to establish correlations between readings obtained using different colorimeters were processed by the computer program Microsoft® Excel 2000 (Redmond, WA). The Student *t*-test was also used to determine differences, if any, in color measurements when using the automatic colorimeters and the manual colorimeter.

RESULTS AND DISCUSSION

Sample distribution. The samples used for the comparative evaluation and for calibration of the palm oil colorimeter had color values ranging from 1.8 to 17.3 Lovibond red units.

Comparison of automatic and manual instruments. The color of RPO and RPOo samples were measured using the manual instrument, Lovibond Tintometer Model F, and the automatic instruments Lovibond PFX 880 Series, the LICO 300, and the palm oil colorimeter. Least-squares linear regression analyses for the mean red and yellow values of the oils are shown in Tables 1 and 2. Correlations between the auto-

TABLE 1 Least Squares Linear Regression for Red Color: Y = Intercept + Slope (X) and Data for the Student t-test

Dependent variable Y	LICO 300	PFX 880	Palm oil colorimeter
Dependent variable X	Model F	Model F	Model F
R-squared	0.9339	0.8605	0.9236
Intercept	0.4476	-0.9974	0.4585
Slope	0.8919	1.6789	0.9101
Student <i>t</i> -critical value	1.578	4.600	0.908

TABLE 2

Least Squares Linear Regression for Yellow Color: Y = Intercept + Slope (X) and Data for the Student t-test

Dependent variable Y	LICO 300	PFX 880	Palm oil colorimeter
Dependent variable X	Model F	Model F	Model F
R-squared	0.4457	0.4758	0.7974
Intercept	15.781	27.811	0.8136
Slope	0.4651	0.6952	9.1603

matic colorimeters and the manual colorimeter for red colors of the test samples were acceptable for the LICO 300 and the palm oil colorimeter as shown by the values of r^2 , slopes close to 1, and intercept near zero (Table 1). The regression results for the LICO 300 and the palm oil colorimeter were acceptable, but that for the PFX 880 was not.

Correlations between the mean yellow values obtained between automatic and manual measurements were poor, with *r*² values of 0.4457, 0.4758, and 0.7974 for the LICO 300, PFX 880, and the palm oil colorimeter, respectively (Table 2). Intercepts also deviated considerably from zero for the LICO 300 and PFX 880, although that for the palm oil colorimeter was still quite close to zero.

The palm oil colorimeter. The practical advantage in retaining the expression of color in red and yellow Lovibond units in the development of the palm oil automatic colorimeter is that the whole palm oil industry is familiar with the Lovibond color scale. Both developed and developing nations in all parts of the world understand the color language of Lovibond red and yellow units. Retaining the use of the arbitrary Lovibond color scale will minimize changes in worldwide communication of color data. Therefore, the color scale used for measurement of color using our palm oil is also expressed in terms of Lovibond red and yellow units or values.

In this method, light discharged by three super-bright light-emitting diodes of red, green, and blue is first collimated before passing it through a 1-in. (2.5 cm) glass cell containing the oil sample. The transmitted light is detected by a silicone photodiode detector. Analog signals from the detector are then digitized before being fed to a microprocessor. An artificial neural network is used to obtain calibration factors to calculate values from the digitized signals and to correlate them with predetermined red and yellow Lovibond color units. The processed data can be displayed using an LCD or stored in the computer *via* an RS 232 port.

Table 3 shows that the repeatability of the palm oil colorimeter is extremely good with an SD of zero and a CV of zero for the red values. In comparison to the red values, the repeatability of the yellow units was less satisfactory. The SD and CV for Sample A were 0.209 and 1.07%, respectively, and for Sample B, the SD and CD were 0.103 and 0.62%, respectively.

Comparison of palm oil colorimeter with commercial colorimeters. Tables 1 and 2 show the relationship of the red and yellow units obtained using the palm oil colorimeter and the Lovibond Tintometer Model F. To ascertain whether there was a difference in the results obtained by using the automatic and manual instruments, a paired comparison using the Student *t*-test was determined. The test statistic for paired comparison of red color measurement between the palm oil colorimeter and the Lovibond Tintometer Model F was 0.91; between the Lovibond Model F visual Tintometer and the Lovibond automatic PFX 880 was 4.60; and between Lovibond Model F Tintometer and the LICO 300 was 1.58. Since the calculated value for that between the palm oil colorimeter and the Lovibond Tintometer Model F was less than the critical value, $t_{\text{cri}} = 2.04$ (DF of 29 at the 95% confidence level), there was therefore no difference between red color measurement obtained using these two instruments. Similarly, there was no difference in measurement of red color using the LICO 300 and the manual colorimeter. On the other hand, the calculated value for the visual Tintometer and the Lovibond automatic PFX 880 was higher than 2.04, indicating a difference between red color measurements using this automated colorimeter and the manual instrument.

The lack of precision in color measurement is extremely difficult to reduce and almost impossible to eliminate. Substitution of the eye with other receptors/detectors such as the

photodetector in our palm oil still gives less precise measurements than the equivalent made by a trained observer. Properly designed and operated instruments produce convenient, rapid, repeatable, and economical color determinations. Two instruments of the same make and model may give the same reading, whereas two observers are less likely to do so. This is denoted as reproducibility and repeatability, not precision. For most practical color measurements and purposes, only moderate precision is required, whereas repeatability and reproducibility are often required in all standard test methods. This is one of the main reasons for the need for an automatic colorimeter for vegetable oil determination.

Color measurement using the palm oil colorimeter is simple, involving three short steps: (i) inserting the glass cell containing the sample into the sample chamber, (ii) pressing a button to initiate the measurement, and (iii) taking the results in Lovibond red and yellow units displayed by the LCD. A large number of color determinations can be carried out in a short time, as less than 3 s is required per analysis. Unlike most bulky automatic colorimeters being marketed currently, the instrument is portable and compact, weighing a mere 1.1 kg (2.4 lb).

The correlations of yellow color units obtained using all three automatic colorimeters against those by the manual colorimeter were found to be poor. However, this limitation can be overlooked because it has been established that yellow values show large variations ($r^2 = 0.4761$, slope = 0.6744, intercept = 17.725 , $n = 24$), even when measured by two experienced operators on the same day and using the same instrument (10). In the manual method for measurement of color, the procedure involved determination of the approximate color initially by setting the ratio of 10.0 yellow to 1.0 red, followed by finding the best match without necessarily maintaining this recommended ratio. The dominant factor in this measurement is the red color; the yellow filters are used to enhance the color match. In the calibration of the palm oil colorimeter for measurement of oil color, the yellow values were determined independently of the red values, i.e., without prior knowledge of the red values. The best fit for yellow values was thus obtained with the use of yellow color alone. Therefore, the difference can be rather large compared with the manual method, where both red and yellow are determined interdependently. The correlation of the yellow units measured using the palm oil colorimeter against those obtained using the manual colorimeter can easily be improved by taking the red units into consideration when calibrating the yellow values.

The colorimeter is especially designed for color analysis of palm oil and palm oil products according to the Lovibond color scale. It replaces the conventional visual grading method with instrumental measurement that is user-friendly, speedy, and reproducible. The time and tedium taken to carry

out a color match are reduced while prolonged viewing periods that cause eye fatigue and erroneous results are eliminated. The benefits of automatic reading are tremendous, considering the time saved in a refinery where hourly or even half-hourly color measurements are required for feedback process control and optimization.

The palm oil colorimeter was specifically programmed for the color measurement of RPO and palm oil products, as it has been calibrated only for the use on these products. It must be appreciated that the whole measurement process of the colorimeter is generic and it can also be calibrated to measure other vegetable oils such as soybean oil and palm kernel oil.

The automatic colorimeter for palm oil color measurement was developed in response to the need to eliminate subjectivity associated with visual assessment. It is a more efficient instrument for routine color measurement and paves the way for the introduction of on-line monitoring of color to estimate the progress in refining and processing.

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