Simultaneous determination of moisture and oil contents in palm fruits by near infrared spectroscopy

(Penentuan serentak kandungan lembapan dan minyak dalam buah sawit dengan spektroskopi inframerah dekat)

O. Muhammad Nor* and C. Abu Bakar**

Key words: NIRS, moisture and oil contents, palm fruits

Abstrak

Teknik spektroskopi inframerah dekat (NIRS) telah dinilai bagi penentuan cepat dan serentak kandungan lembapan dan minyak dalam buah sawit. Hirisan mesokarpa dibekukan dan dikisar sebelum analisis kimia dan NIRS. Graf kalibrasi penentuan kandungan lembapan dan minyak dengan 44 sampel telah diperoleh. Didapati bahawa sisihan piawai (SD) dan pekali korelasi berganda (*R*) lembapan masing-masing 0.788 dan 0.96 manakala SD dan *R* bagi minyak masing-masing 0.277 dan 0.97. Ramalan dengan 10 sampel menunjukkan bahawa kandungan lembapan mempunyai sisihan piawai ramalan (SEP) dan *R* masing-masing 0.382 dan 0.97 manakala ramalan kandungan minyak menunjukkan SEP dan *R* yang bernilai 0.294 dan 0.96.

Abstract

Near infrared spectroscopic (NIRS) technique was assessed for rapid and simultaneous determination of moisture and oil contents in palm fruits. The sliced mesocarps were frozen and ground prior to analyses by wet chemistry and NIRS. Calibrations with 44 samples were developed for moisture and oil content respectively. Results showed that standard deviation (SD) for moisture content was 0.788 and multiple correlation coefficient (R) was 0.96, whereas those for oil content were 0.277 and 0.97 respectively. Prediction of moisture content with 10 samples showed that the standard error of prediction (SEP) was 0.382 and R was 0.97, whereas for oil content the SEP was 0.294 and R was 0.96.

Introduction

Rapid determination of the quality parameters of crude palm oil during production is important in order to control the extraction process. This can be achieved through the application of near infrared spectroscopic (NIRS) technique. NIRS has been identified as one of the most powerful techniques for the determination of quality parameters, i.e. moisture, protein and fat contents in various products (Williams and Norris 1987) after its introduction by Norris (1964). NIRS technique is based on the difference between the energy of the incident light and the energy of the light reflected from the surface of the samples in the region of 750–2 500 η m. Although the technique has been categorised as a

*Chemistry and Technology Division, Palm Oil Research Institute of Malaysia (PORIM), P.O. Box 10620, 50720 Kuala Lumpur, Malaysia

^{**}Livestock Research Centre, MARDI Headquarters, P.O. Box 12301, 50774 Kuala Lumpur, Malaysia Authors' full names: Muhammad Nor Omar and Abu Bakar Chik

[©]Malaysian Agricultural Research and Development Institute 1998

secondary measurement, it has been widely used for the determination of various components in foods, feeds, grain, oilseeds and pharmaceutical products (Williams 1975; Tkachuk 1981; Panford et al. 1988). Currently, NIRS is included as an official method by the American Official Analysts Association (AOAC 1990) and the American Association of Cereal Chemists (AACC 1983).

Due to the simplicity in sample preparation, NIRS has an economic impact on the routine analysis because it allows non-destructive, rapid and simultaneous multiple analyses for quality control purposes. On the environmental issue, NIRS can play an important role with respect to green technology since it does not use any hazardous chemicals.

Although many studies have been carried out on edible oils, information on the use of NIRS in palm oil and its products is limited. Panford et al. (1988) have indicated the use of NIRS in oilseeds, including palm kernel oil. Due to the great interest in NIRS, this study was carried out to establish a simultaneous and rapid method for palm oil analysis using NIRS in response to the demand of palm oil industry.

Materials and methods *Sample preparation*

Palm fruits were collected from the UKM-PORIM Experimental Station in Bangi, Selangor. Three fruits each from randomly selected bunches were taken and cleaned to rid of sand, dirt and other foreign materials. About 15 g of mesocarp slices were obtained from the fruits and subsequently frozen at -4 °C. Frozen samples were ground in a chopper-blender for 30 s just prior to analyses to minimise loss of oil and moisture.

Moisture determination

Analytical determination of moisture content was carried out using methods Ac 2-41 and Ai 2-75 of AOCS (AOCS 1987). Two grams of ground, frozen mesocarp was used to determine the moisture content by drying at 105 °C (± 2 °C) for 16 h in a vacuum oven.

Oil content measurement

The oil content was determined by using method Ab 3-49 of AOCS (AOCS 1987). Five grams of frozen and ground mesocarp was refluxed for 16 h using petroleum ether (analytical grade, BDH, UK) in a soxhlet extraction unit. The solvent extract was dehydrated over anhydrous sodium sulphate and then evaporated using a rotary evaporator at 40 °C under vacuum.

NIRS analysis

Forty-four samples were used for calibration and 10 samples were used for prediction of NIRS measurement. A near infrared spectroscopic instrument (Pacific Scientific Research Analyzer Model 6250, USA, scanning monochromator, 1 100–2 500 ηm) was used to scan the samples. The physical data, i.e. moisture and oil contents of palm mesocarps, were entered via IBM computer. Calibrations and predictions were done by the software provided with the instrument using multiple linear regression.

Results and discussion

In Figure 1 which shows the band differences between fresh and vacuum oven-dried palm mesocarps, a slight difference in absorption bands occurred at the wavelengths around 1 564, 1 502 and 1 138 mm. These absorption wavelengths seemed to be due to -OH stretching of C16:0 and overtone of unsaturated moieties of fatty acid (Sato et al. 1991; Sato 1994). Using these wavelengths, a linear regression line for the determination of oil content in palm mesocarp was drawn as shown in Figure 2. It was found that the standard deviation (SD) was 0.277 and the multiple correlation coefficient (R) was 0.97. This correlation is better than the results previously reported when analysing oil content in palm mesocarps (Muhammad Nor and Abu Bakar 1997a). The wide range in fatty acid composition could influence the wavelength selection. This is because palm



Figure 1. Typical NIRS spectrum of fresh and dried oil palm mesocarps



Figure 2. Calibration curve of oil content in palm fruits

oil contains a high proportion of saturated fatty acid, i.e. palmitic acid. The oil contents predicted using NIRS were within acceptable range compared to those obtained by chemical analysis (*Table 1*) with a standard error of prediction (SEP) of 0.294 and correlation coefficient of 0.96.

The NIR spectrum indicated that there were slight differences at around 2 120, 1 392, 1 310 and 1 838 mm probably due to -OH absorption wavelengths. Absorption

peaks at wavelength 1 838 and 1 392 η m were greatly suppressed after samples were subjected to vacuum drying. However, the absorption bands at the 1 310 and 2 120 η m regions remained the same before and after drying. Using these wavelengths, a linear regression line for moisture determination was drawn as shown in *Figure 3*. From this regression line, the SD was 0.788 and *R* was 0.96. This correlation is better than that reported previously when analysing the



Figure 3. Calibration curve of moisture content in palm fruits

Table 1.	Prediction	of oil	content	in p	alm fruits	
by NIRS	5					

Oil content by chemical analysis (%)	Predicted values (%)		
54.13	54.01		
55.23	55.03		
56.41	55.81		
54.69	54.69		
55.13	55.02		
54.80	54.62		
53.36	53.04		
54.82	54.23		
56.24	56.52		
53.89	53.62		

Standard error of prediction (SEP) = 0.294Multiple correlation coefficient = 0.96

moisture content of palm mesocarps (Muhammad Nor and Abu Bakar 1997a). Prediction of 10 unknown samples showed that the SEP was 0.382 and *R* was 0.97 (*Table 2*).

Selection of wavelength was based on verification with correlation charts and other information in the literature (Osborne and Fearn 1986; Panford et al. 1988). It was found that the more wavelengths selected, the better the correlation obtained. This was also observed by the authors in their earlier studies (Muhammad Nor and Abu Bakar 1997a, b). Although the software allowed for

Table 2. Prediction of moisture content in palm fruits by NIRS

Moisture content by chemical analysis (%)	Predicted values (%)		
24.91	24.60		
24.41	24.56		
23.42	23.03		
22.00	22.41		
24.33	24.99		
24.00	23.86		
22.21	22.45		
20.70	20.56		
23.28	23.04		
24.91	24.48		

Standard error of prediction (SEP) = 0.382 Multiple correlation coefficient = 0.97

many wavelengths, this study was confined to not more than four wavelengths only.

Conclusion

The NIRS technique could be used for rapid and simultaneous determination of moisture and oil contents in palm fruits. This was because the technique employed simple methods for sample preparation and no chemical treatments were involved. The prediction results also showed that there were no significant differences between the mean values of the results obtained by NIRS and chemical analysis. References

- AACC (1983). Methods 39-10 and 39-11. In Approved methods of the American Association of Cereal Chemists 8th ed., Vol. 1. St. Paul, Minn.: AACC
- AOAC (1990). Methods 989.03 and 967.19. In Official methods of analysis of the Association of Official Analytical Chemists 15th ed., Vol. 1 and 2 (Helrich, K., ed.). Arlington, VA.: AOAC
- AOCS (1987). Methods Ac 2-41, Ai 2-75 and Ab 3-49. In *Official methods and* recommended practices of the American Oil Chemists Society 3rd ed., Vol. 1 (Walker, R. C., ed.). Champaign, IL.: AOCS
- Muhammad Nor, O. and Abu Bakar, C. (1997a). A preliminary study on near infrared spectroscopic method in the determination of moisture and oil content in palm mesocarp. PORIM viva report CT 249
- (1997b). The use of near infrared spectroscopic analysis to determine the moisture content in palm fruit. PORIM viva report CT 249
- Norris, K. H. (1964). Reports on the design and development of a new moisture meter. *Agric. Eng.* **45:** 370–2
- Osborne, T. B. and Fearn, T. (1986). *Near-infrared spectroscopy in food analysis* 200 p. Harlow, Essex: Longman Scientific & Technical
- Panford, J. A., Williams, P. C. and deMan, J. M. (1988). Analysis of oilseeds for protein, oil, fiber and moisture by near-infrared reflectance spectroscopy. JAOCS 65: 1627–34
- Sato, T. (1994). Application of principal-component analysis on near-infrared spectroscopic data of vegetable oils for their classification. *Ibid.* 71: 293–8
- Sato, T., Kawano, S. and Iwamoto, M. (1991). NIR spectral patterns of fatty acid analysis. *Ibid.* 68: 827–33
- Tkachuk, R. (1981). Protein analysis of whole wheat kernel by near infrared reflectance. *Cereal Foods World* **26**: 584–7
- Williams, P. C. (1975). Application of near infrared reflectance spectroscopy to the analysis of cereal grains and oilseeds. *Cereal Chem.* 52: 561–76