WIDELINE NUCLEAR MAGNETIC RESONANCE FOR MEASURING THE OIL CONTENT OF PALM MESOCARP

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Wideline nuclear magnetic resonance (WLNMR) has been used to measure the oil content of palm mesocarp. The WLNMR was calibrated using crude palm oil and a factor to correct the signal readings due to non-oil components of mesocarp was introduced into the calibration equation. Mesocarp from Tenera *palm fruits was cut and dried in either a conventional drying oven or in a microwave oven. The dried mesocarp was then ground and the NMR signal of a known amount of sample was taken after tempering at 70°C for 30 minutes. The percentage of oil in the mesocarp was then calculated and this value was compared with that obtained by Soxhlet extraction. The results showed that WLNMR could be used to measure the oil content in the mesocarp rapidly, and that the measuring time was much shortened with microwave drying.*

INTRODUCTION

The oil content of palm fruit is commonly determined by solvent extraction, and the Soxhlet method is widely used. An attempt was made to develop an alternative and more rapid method of measuring the oil content using low resolution nuclear magnetic resonance (NMR) spectrometry.

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NMR spectrometry has been used to determine the oil content of seeds such as sunflower (Robertson and Morrison, 1979; Robertson and Windham, 1981; Jones, undated), mustard and soya bean (Tiwari, 1980). This study aimed at testing the feasibility of measuring the oil content of palm mesocarp using our existing low resolution wideline NMR, which is already used for routine measurement of solid fat content (SFC).

The mesocarp samples were dried in the conventional drying oven used in connection with solvent extraction. In addition, samples were dried by using a microwave oven in order to reduce the drying time. Microwave heating has been available for many years and the depth of penetration of heating is a function of frequency (Copson, 1975). Both microwave and infra-red have been used in drying oil seeds such as soya beans (Short, 1982). Microwave heating has been applied to moisture analysis in food, including cereals (Steele, 1974) and it has also been used in the drying of foods (Rosenberg and Bogl, 1987).

EXPERIMENTAL

Material and Sampling

Tenera fruits were used in this study. The fruits were individually removed from the spikelets and the mesocarp was then cut into small pieces, which were dried before grinding with a Moulinex blender (Type 2.41). Two NMR tubes were prepared for each sample.

Drying in a Conventional Heating Oven

The samples were dried in a conventional heating oven (Gallenkamp Moisture Extractor Oven) at 105°C for 24 hr (Rao *et al.*, 1983). The dried samples were ground and used for both Soxhlet extraction and NMR determination.

Microwave Drying

Microwave-dried samples were used for NMR determination only.

A National microwave oven (Model Dimension 3) which has a power range of between 60 and 600 Watts was used.

Both the power and the heating time were studied so that an optimal procedure for microwave drying could be established. The drying was carried out with intervals of heating followed by intervals of cooling to prevent overheating and charring of the mesocarp.

NMR Spectrometer

A Newport Wideline NMR model IIIA was used. This instrument has a 2 ml sampling coil. It was set with the following parameters:

Gatewidth	:	1.5 Gauss
RF Level	:	45 micro Amp
AF Gain	:	375 units
Integration Time	:	32 seconds

Calibration

The instrument was calibrated by taking the signals of crude palm oil samples of known weight at 70°C after tempering them for 30 min at the same temperature. The readings of dried oil-free mesocarp samples obtained after Soxhlet extraction were also taken similarly.

NMR Measurement

A dried sample of known weight (about 0.9 g) was prepared in the NMR tube (2 ml capacity) and the sample height was kept below 1.8 cm to avoid saturation. After the samples had been tempered at 70°C for 30 min, two signal readings were taken for each NMR tube and the average signal was used for calculating the oil content. The mean oil content from the two sample tubes was recorded.

Soxhlet Extraction

About 5 g of dried and ground mesocarp were weighed into the extraction thimble and a filter paper was placed on top of the thimble to prevent the mesocarp from floating away during the extraction. The sample was extracted for about 16 hr (Rao *et al.*, 1983) till the solvent turned colourless. The solvent in the flask was then removed by using a rotary evaporator and the residue was dried in an oven at 105°C for 2 hr and weighed after cooling. The percentage oil content was calculated from the weight of oil extracted.

RESULTS AND DISCUSSION

Microwave Oven Drying

The criterion chosen in this work was to dry the sample until two constant NMR signals were obtained. A heating time of 3 min at Medium setting (420 Watts) was used at first, but some of the mesocarp fibres were found to have charred. The charring was not observed, however, when intermittent 2 min heating steps were used. Moisture and volatiles and NMR signals determined in the experiments are given in *Table 1*.

Based on the various observations made, the following drying procedure was adopted. The cut sample was dried in the microwave oven set at Medium power (420 Watts) for 6 cycles of 2 min heating, each followed by 2 min of cooling. The details of the drying conditions were given in an earlier report (Oh, *et al.*, 1989).

TABLE 1. MOISTURE AND VOLATILES (%) AND NMR SIGNAL READING OF PALM MESOCARP AT VARIOUS STAGES OF DRYING[a]

Heating time (min)	Moisture and volatiles (%)	Signal reading/g at 70°C
3 min heating steps		
0	N.A.	42.5
3	18.6	42.0
6	23.7	40.9
9	24.6	40.0
12	24.9	39.5
15	25.0	40.1
18	25.1	39.4
21	25.2	39.9
24	25.1	40.4
27	25.2	40.6
30	25.2	40.0
2 min heating steps		
0	N.A.	41.4
2	15.1	40.9
4	19.6	41.0
6	21.6	40.2
8	22.2	39.6
10	22.7	39.8
12	22.1	39.8
14	22.1	39.8
16	22.7	39.3
18	23.0	39.4
20	23.0	39.6

N.A. Not applicable

[a]The values became steady after certain heating steps indicating that all moisture and volatiles have been removed.

Sample Height

Table 2 shows that NMR signal saturation did not occur for samples less than 2.0 cm in height, which is completely within the measuring coil. In this study the sample height was kept below 1.8 cm and the sample weight was about 0.9 grams.

Calibration and Principle of Calculation

The NMR signals of 10 crude palm oil samples at 70°C were taken, this temperature being chosen so that all the oil present would be in the liquid state.

A linear calibration equation of the following form is obtained:-

$$W = aR - b \quad (1)$$

where

W = weight of crude palm oil in grams;
R = reading of NMR signal;

a and b are empirical constants with values of 0.02040 and 0.00848 respectively as obtained experimentally.

Equation (1) may be used to calculate the weight of the oil in the mesocarp. However it was observed that the non-oil components of the mesocarp also gave rise to small NMR signals. A correction term for the signal, R_N, due to the non-oil components was therefore introduced.

The signals of dried residual mesocarps after the Soxhlet oil extraction were measured after tempering at 70°C for 30 min and a linear regression equation of the form below was obtained :-

$$R_N = kW_N + n \quad (2)$$

where

R_N = NMR signal reading of non-oil components.
W_N = weight of non-oil components in grams.

The values of the empirical constants k and n obtained experimentally were 3.7972 and 0.3042 respectively.

W_N of the sample can be obtained from the weight of the sample (M) and the weight of crude palm oil(W) as given by equation (1).

Thus, we have

$$W_N = M - W \quad (3)$$

The corrected NMR signal, R_C, of palm oil in the sample may be expressed as

$$R_C = R - R_N \quad (4)$$

TABLE 2. NMR SIGNALS OF MESOCARP SAMPLES OF VARIOUS WEIGHTS AND HEIGHTS AT 70°C

Sample Weight (g)	Sample Height (cm)	NMR Signal
0.1	0.2	4.2
0.2	0.4	7.9
0.3	0.6	12.3
0.4	0.8	15.0
0.5	1.0	18.8
0.6	1.2	23.0
0.7	1.4	27.3
0.8	1.6	29.8
0.9	1.8	35.4
1.0	2.0	38.5
1.1	2.2	42.4
1.2	2.4	44.1
1.3	2.6	45.9
1.4	2.8	46.3
1.5	3.0	46.1

and the corresponding corrected weight, W_C, of palm oil in the sample is therefore

$$W_C = aR_C - b \quad (5)$$

On substitutions

$$W_C = a(R - R_N) - b$$
$$= aR - a(kW_N + n) - b$$
$$= aR - ak(M - W) - an - b$$
$$= aR + akW - akM - an - b$$
$$= aR + ak(aR - b) - akM - an - b$$

On rearranging, we have

$$W_C = aR(1 + ak) - b(1 + ak) - an - akM \quad (6)$$

The corrected percentage oil content of the mesocarp is then given by

$$P_C(\%) = 100W_C/M \quad (7)$$

On substituting the values of a, b, k and n, the following equation was obtained :

$$P_C(\%) = (2.198R - 1.53)/M - 7.75 \quad (8)$$

TABLE 3. MESOCARP OIL CONTENT (%) BY THREE METHODS

Method[a]	Y	Z	X	Y	Z	X
Sample				Sample		
1.	70.7	69.0	69.9	31.	74.1	73.1	73.5
2.	68.5	64.5	67.7	32.	71.3	72.1	72.7
3.	71.4	70.0	69.9	33.	74.9	72.8	73.7
4.	65.1	64.1	64.0	34.	66.4	64.0	64.8
5.	65.6	64.4	64.7	35.	67.8	65.4	66.3
6.	68.7	67.1	68.5	36.	68.5	68.3	66.9
7.	81.5	80.9	81.8	37.	76.7	74.8	75.7
8.	82.2	81.1	82.0	38.	76.4	74.8	75.0
9.	81.1	81.1	82.3	39.	76.2	73.0	75.4
10.	74.8	73.0	74.8	40.	72.5	68.7	70.2
11.	76.8	76.6	77.2	41.	68.4	66.6	67.3
12.	76.0	77.3	77.8	42.	69.4	65.7	66.8
13.	77.2	73.1	75.8	43.	76.8	77.0	76.9
14.	75.9	75.5	77.2	44.	77.9	75.7	77.0
15.	73.4	73.8	73.6	45.	78.1	76.8	76.4
16.	70.4	66.7	68.5	46.	71.8	72.0	71.2
17.	71.3	71.3	70.4	47.	72.7	74.3	72.6
18.	71.5	70.9	70.4	48.	70.4	69.4	69.6
19.	80.8	77.8	78.1	49.	74.0	74.4	74.5
20.	78.7	78.7	77.7	50.	74.5	74.2	73.9
21.	79.4	77.9	78.0	51.	73.4	72.7	73.3
22.	77.6	78.1	77.5	52.	72.0	71.4	71.9
23.	78.4	77.7	79.7	53.	69.4	68.1	68.9
24.	79.5	78.7	79.0	54.	72.2	71.4	72.2
25.	85.1	82.7	81.7	55.	77.7	77.1	78.6
26.	82.8	82.9	83.0	56.	77.1	75.6	76.0
27.	85.2	82.2	81.9	57.	75.8	75.5	75.1
28.	83.2	81.2	80.7	58.	75.1	75.2	73.4
29.	79.5	77.7	77.5	59.	72.8	71.3	70.2
30.	81.5	80.5	80.5	60.	71.2	69.5	70.4

[a]Y = by Soxhlet Extraction
Z = by NMR on Conventional oven-dried sample
X = by NMR on Microwave oven-dried sample

Equation (8) was used to calculate the oil content of palm mesocarp samples. If a different instrument is used a new set of constants a, b, k and n has to be established.

Comparison of Results and Regression Equations

Table 3 gives the oil contents obtained by both extraction (Method Y) and NMR methods. For the NMR measurements, samples were dried in both the conventional drying oven (Method Z) and the microwave oven (Method X).

The following linear regression equations between Methods Y and X and between Methods Y and Z were obtained:

$$X = 0.991Y - 0.09 \quad (r^2 = 0.9482) \qquad (9)$$

$$Z = 1.014Y - 2.21 \quad (r^2 = 0.9353) \qquad (10)$$

where

X = percentage oil content by NMR measurement on mesocarp dried in the microwave oven.

Y = percentage oil content by Soxhlet extraction

Z = Percentage oil content by NMR measurement on mesocarp dried in the conventional drying oven.

r^2 = coefficient of regression

Method X is preferred since it has a slightly greater precision and is faster than Method Z.

CONCLUSION

The oil content of palm mesocarp can therefore be quickly determined by NMR instead of the routine Soxhlet extraction. The mesocarp oil content of individual fruits may be determined by the NMR method described above if required.

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