BRJ BASIC RESEARCH JOURNALS

Basic Research Journal of Agricultural Science and Review ISSN 2315-6880 Vol. 7(6) pp. 53-57 August 2019
Available online http//www.basicresearchjournals.org

Full Length Research Paper

Qualitative analysis of palm kernel cake using near-infrared reflectance spectroscopy and proximate analysis

Norlindawati, A. P.[1]* and Shariff, S.[2],

[1]Malaysia Veterinary Institute, KM13 Jalan Batu Pahat, Beg Berkunci 520, 86009 Kluang, Johor, Malaysia.
[2]Veterinary Research Institute, 59 Jalan Sultan Azlan Shah, 31400 Ipoh, Perak, Malaysia.

*Corresponding author email: norlindawati.dvs@1govuc.gov.my

Acceptance 10 August 2019

ABSTRACT

The purpose of this study was to determine whether near-infrared (NIR) reflectance technology is reliable to be used as a rapid method for qualitative analysis of palm kernel cake. Six parameters were analyzed; moisture, ash, crude protein (CP), ether extracts (EE), crude fiber (CF) and nitrogen free extract (NFE). Palm kernel cake (PKC) samples (n = 48) were randomly selected from Feed Analysis Laboratory, Malaysia Veterinary Institute, Johor in year 2009, 2010 and 2012. CP was determined by the Kjeldahl method, CF was measured using Fibertec methods, and EE was measured using Soxtec methods. Each sample was also scanned with NIRFlex Model N-500. The data was analyzed using the SAS statistical program. Statistical significance of differences between means was tested by paired t-test analysis. A p-value of less than 0.05 ($p<0.05$) was considered statistically significant. There was no significant difference in the results from reference and test method for CP, EE, CF and NFE. These statistical analyses suggested that both methods can produce the same results for PKC sample. However, there are significant differences for moisture and ash result.

Keywords: palm kernel cake, near-infrared reflectance technology, proximate analysis

INTRODUCTION

Palm kernel cake (PKC) is already known as a high-fiber, medium grade protein feed that is best suited to ruminants. Based on the chemicals composition, PKC can be classified as an energy-feed (Alimon and Wan Zahari, 2012). According to Alshelmani et al. (2014) PKC can be a promising feedstuff of animal feed because of its content of a moderate level of crude protein 14.5% to 19.24% with 4% to 5% ash, 0.70% to 0.90% ether extract and 10% to 17% crude fiber. However, according to the study done by Shariff et al. (2012) nutritive value of PKC varies and some samples did not achieve the proper specification values. To obtain a balanced diet for livestock feeding, qualitative analysis of PKC should be done regularly.

Conventionally, proximate analysis is well established and approved by Association of Official Analytical Chemists (AOAC) as a reference method to determine the nutritional values in feedstuffs. However, the procedure is time-consuming, expensive and not environmental friendly. Simpler, less expensive technology with shorter turnover time was needed for feed testing (Corson et al., 1999). Near-infrared (NIR) has the benefit of being low cost and less time-consuming compared to conventional method so there is

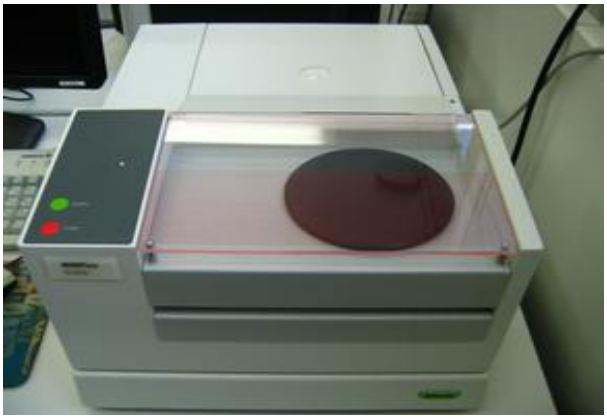

Figure 1. NIRFlex Model N-500

a great potential for adopting this rapid technology (Edney et al., 1994). Proximate analysis is a quantitative method to determine different macronutrient in feed; it is the partition of feed into six categories, which are moisture, ash, crude protein (CP), ether extracts (EE) or crude fat, crude fiber (CF) and finally nitrogen-free extract (NFE). Nitrogen-free extract estimates non-fibrous carbohydrate, such as sugars and starches. The nitrogen-free extract determination is the only estimate of proximate analysis determined by calculation of a difference versus chemical analysis followed by appropriate calculations. The calculation for nitrogen-free extract is:

%NFE = 100 - (%CP + %CF + %Ash + %EE) (equation 1)

As nitrogen-free extract is calculated by difference, all the errors associated with proximate analysis are additive in the estimate of nitrogen-free extract. To determine the percentage of dry matter for the feed samples is subtract the moisture content from 100% as below:

% Dry Matter = 100 - % moisture (equation 2)

Near-infrared reflectance spectroscopy (NIRS) technology now is a common quality control tool in the feed industry. This technology is already approved by Association of Official Analytical Chemists (AOAC), for use in determining moisture, Kjeldahl nitrogen and acid detergent fiber for feed and forage analysis (Undersander, 2006). The application of this instrument is extensive and covers medical, agricultural, chemical industries and pharmaceuticals analyses. Present conventional method need to be replaced with a rapid technology such as NIR because conventional methods require longer time of analysis as well as laboratory expertise.

The objective of the present study is to determine whether near-infrared reflectance technology is reliable to be used as a rapid method for qualitative analysis of palm kernel cake. Parameters studied were moisture, total ash, crude protein, ether extracts, crude fiber and nitrogen free extract.

MATERIALS AND METHODS

Palm kernel cake samples

The palm kernel cake samples used in this study were samples received by Feed Analysis Laboratory, IVM Kluang in year 2009, 2010 and 2012. A total of 15 samples were received in 2009, 6 samples in 2010 and 27 samples in 2012. During the 3 years, only 48 samples PKC has been accepted and analyzed. Samples received were ground and used to carry out proximate analysis and subsequently the same samples were tested using NIR equipment.

Proximate analyses

The ground samples were weighed and put in forced-air drying oven at 103 ± 2˚C overnight (AOAC 2000) to determine the dry matter. Ash content was determined by incineration at 550 ± 20˚C for 4 hours (FAO 2011). The ground PKC samples also been used to determine the chemical composition of samples. The CP content (*N x 6.25*) was determined after digestion in sulphuric acid by the Kjeldahl method using *Kjeltec*™ methods (FOSS, 2003). CF was measured after treated with boiling dilute sulphuric acid and with boiling sodium hydroxide solution using *Fibertec™* methods (FOSS, 2010). Ether extract was determined using Soxtec Extraction System (FOSS, 2008) and finally, the nitrogen free extract was calculated as mention in equation 1.

NIR Spectroscopy

The Near Infra-Red equipment used is a NIRFlex Model N-500 (Buchi) (Figure 1), in conjunction with NIRCal® software version 5.1 and NIRWare®Management software (Buchi). Reflectance NIR spectra were recorded

Table 1. Summary of descriptive statistics for the reference values

Parameter	Calibration set				Validation set			
	n	**Range**	**Mean**	***SD***	***n***	**Range**	**Mean**	***SD***
Moisture	183	4.11 – 8.75	6.67	0.99	62	4.02 – 10.59	6.69	1.15
Ash	229	2.77 – 16.3	8.32	3.81	72	3.36 – 16.1	8.22	3.76
CP	238	8.49 – 18.26	14.39	2.94	80	8.72 – 18.18	14.39	2.99
EE	221	1.49 – 9.63	4.01	1.89	73	1.48 – 9.45	3.79	1.69
CF	172	11.34 – 27.59	19.59	3.65	54	11.43 – 25.99	19.82	3.37
TDN	94	48.76 – 78.32	62.04	8.10	32	49.00 – 77.80	61.78	7.89
NFE	125	41.92 – 61.91	51.51	5.36	41	42.11 – 61.74	51.93	5.28
ME	182	5.21 – 12.17	9.56	1.71	61	5.57 – 12.13	9.58	1.68
P	178	0.15 – 0.77	0.43	0.14	60	0.15 – 0.72	0.43	0.14
Ca	163	0.09 – 2.47	0.85	0.57	55	0.24 – 2.29	0.89	0.61

SD =Standard deviation
CP: crude protein, EE: ether extract, CF: crude fiber, TDN: total digestible nutrients, NFE: nitrogen free extract, ME: metabolizable energy, P: phosphorus, Ca: calcium

Table 2. Prediction results for the optimized PLS regression models

Parameter	***SEP***	r^2	***Bias***	***RPD***
Moisture	0.57	0.89	0.0627	2.01
Ash	0.84	0.96	0.0274	4.48
Crude Protein	0.57	0.98	0.0369	5.23
Crude Fat	0.51	0.95	0.0326	3.31
Crude Fiber	0.99	0.96	0.0923	3.40
NFE	0.72	0.99	-0.0231	7.33

SEP (Standard error of prediction); RPD (Ratio Performance Deviation)= $SD_{validation}SEP^{-1}$

over the range 4000-10000 cm^{-1} (400-1000 nm) at 4 cm^{-1} intervals. Calibrations for NIR prediction of qualitative palm kernel cake qualitative was carried out by using NIRCal® software version 5.1. Partial least squares (PLS) regression was used to build the calibration models. Two-thirds of the samples were randomly selected as the calibration set and the rest one-third of the samples were used as the validation set (Norlindawati et al., 2013). Ground PKC samples were placed into a glass petri dish and spread to cover the surface of the petri dish. Moisture, total ash, crude protein, ether extracts and crude fiber were then determined by NIR equipment using NIRWare®Management software.

Statistical analysis

The comparison between reference method (proximate analysis) and test method (NIR) results was made using SAS statistical program. For the comparison of the means of the two methods, *paired t-test* was applied, with assumption that there is no difference in the mean values (μ_1 and μ_2, respectively) for each parameter. A p-value of less than 0.05 ($p<0.05$) was considered statistically significant.

RESULTS AND DISCUSSION

Reference data

The descriptive statistics of the sample sets for each respective parameter used for Partial Least Squares (PLS) regression model development are summarized in Table 1 and PLS regression model prediction results are shown in Table 2 (Norlindawati et al., 2013).

In developing calibrations for moisture, ash, crude protein, crude fat, crude fiber, total digestible nutrients, nitrogen free extract, metabolizable energy, calcium and phosphorus, PLS regression was employed. PLS regression model prediction results are shown in Table 2. The prediction results of this study included extremely low and high reference values. The calibration should be improved further if all the extreme reference values are excluded.

The final evaluation of the calibration models was based on the suggestions given by Sohn et al. (2007). The study suggests that prediction models with RPD value of 5.0 – 6.4 are adequate for quality control. RPD values from 3.1 – 4.9 are good and adequate for standard screening purposes. RPD values of 2.4–3.0 are considered adequate for rough screening purposes.

Unsuccessful predictions have RPD values lower than

Table 3. Mean, $\mu \pm S.D.$ and *p*-value of proximate analysis and NIR for each parameter

Parameter	Method	Mean, $\mu \pm S.D.$	*p*-value
Moisture	Proximate analysis	7.63 ± 1.69	0.00
	NIR	6.58 ± 1.20	
Ash	Proximate analysis	7.50 ± 3.61	0.00
	NIR	6.65 ± 2.73	
Crude Protein	Proximate analysis	16.17 ± 2.57	0.15*
	NIR	15.89 ± 1.94	
Ether Extract	Proximate analysis	4.70 ± 2.37	0.71*
	NIR	4.59 ± 1.78	
Crude Fiber	Proximate analysis	19.29 ± 5.66	0.12*
	NIR	18.34 ± 3.03	
Nitrogen Free Extract	Proximate analysis	52.13 ± 6.76	0.15*
	NIR	51.21 ± 5.90	

n = 48 samples for each parameter
**means between reference method and test method are not significantly different (p>0.05)*

1.5 or 0.65, respectively.

According to the Ratio Performance Deviation (*RPD)* values ($SD_{validation}SEP^{-1}$), the models for the NFE and crude protein value model is suitable for quality control purposes. *RPD* value for ash, crude fiber, and crude fat can be classified as fair are good and adequate for standard screening purposes. The moisture, value models performed poorly with these instruments are considered adequate for rough screening purposes.

A paired t-test was conducted to compare the means of reference method (proximate analysis) and test method (NIR) for each parameter (Table 3). There were no significant differences in the results between proximate analysis and NIR for CP, EE, CF and NFE. These statistical analyses suggest that both methods can produce the same results for PKC samples. But there were significant differences for moisture and ash result ($p<.05$).

Overall, there was a strong correlation between proximate analysis and NIRs results, with moisture and ash measures showing the weakest relationship. NIRs can be considered as a secondary method, as it is based on reference evaluations in comparison to proximate analysis and therefore cannot be more accurate than the methods on which it is based (Harris et al., 2018). However, calibration of moisture and ash from this instrument should be improved and further study need to be carried out to determine the *RPD* value for both parameters. This is because the accuracy of NIR prediction is totally dependent upon instrument calibration and supported by good quality assurance.

CONCLUSION

Based on this study, NIR reading has been shown to be a viable alternative to proximate analysis for the qualitative determination of PKC in our laboratory. The analysis takes only a few minutes for each sample once the calibration model for PKC has been set up. However, improvement of calibration of moisture and total ash for our instrument is necessary before it can be applied as a rapid tool for determining PKC nutritional composition.

ACKNOWLEDGMENT

The authors would like to thank the Director General of Veterinary Services, Dato' Dr Quaza Nizamuddin Bin Hassan Nizam for his permission to publish this paper, the Director of Malaysia Veterinary Institute, Kluang, Mrs. Sabariah Bt. Basir, all Feed Analysis Laboratory staff, and Tuan Haj. Syed Hussein Bin Syed Abdullah for publication of this paper.

REFERENCES

Association of Official Analytical Chemists (A.O.A.C) (2000). Official Methods of Analysis. 17th edition. Association of Official Analytical Chemists. Washington D. C.

Alimon AR, Wan Zahari WM (2012). Recent advances in the utilization of oil palm by-products as animal feed. In: International Conference on Livestock Production and Veterinary Technology. (ICARD), 1 - 4 Oct. 2012, Ciawi, Bogor, Indonesia. (Unpublished). [Online, accessed March 25 2014] http://umkeprints.umk.edu.my/1148/1/Paper%203.pdf.

Alshelmani MI, Loh TC, Foo HL, Lau WH, Sazili AQ (2014). Biodegradation of Palm Kernel Cake by Cellulolytic and Hemicellulolytic Bacterial Cultures through Solid State Fermentation. The Scientific World Journal. 2014: 1 - 8

Corson DC, Waghorn GC, Ulyatt MJ, Lee J (1999). NIRS: Forage analysis and livestock feeding. Proceedings of the New Zealand Grassland Association 61: 127–132.

Edney MJ, Morgan JE, Williams PC, Campbell LD (1994). Analysis of feed barley by near-infrared reflectance technology. Journal of Near-infrared Spectroscopy. 2 (1): 33-41.

Food of Agriculture Organization (FAO) (2011). Quality Assurance for Animal Feed Analysis Laboratories. FAO Animal Production and Health Manual No. 12. Jim Balthrop, Benedikt Brand, Richard A. Cowie,

Jurgen Danier, Johan De Boever, Leon de Jonge, Felicity Jackson, Harinder P. S. Makkar and Chris Piotrowski. Rome, Italy.
FOSS, (2003). Application note: AN 3001 (2003). Determination of Crude Protein (Kjeldahl Nitrogen) in Animal Feed, Forage (Plant Tissue), Grain and Oilseeds Using Block Digestion with Copper Catalyst and Steam Distillation into Boric Acid. Sweden: FOSS Analytical.
FOSS (2010). Application note 3414. Rev. 5.0 (2008). Extraction of Crude or total fat in Animal feed and forage according to ISO 6492 Using Soxtec Extraction System. Sweden: FOSS Analytical.
FOSS (2008), Application note 3437 (2010). Determination of Crude Fiber (CF) in feed using *Fibertec*™ or M6 according to AOAC 978.10, AACC 32-10 and AOCS Ba 6-84.
Harris PA, Nelson S, Carslake HB, Argo C Mc G, Wolf R, Fabri FB, Brolsma KM, van Oostrum MJ, Ellis AD (2018). Comparison of NIRS and wet chemistry methods for the nutritional analysis of haylages for horses. Journal of Equine Vet. Sci. Volume 71:13-20.
Norlindawati AP, Shariff S, Noormah Miw A, Sabariah B, Omar R, Samijah A, Sufi J, Jamnah O (2013). Analysis of palm kernel cake by near-infrared reflectance technology. Malaysian J. Vet. Res. Volume 4, No.1: 1 – 4.
Saeys W, Mouazen AM, Ramon H (2005) Potential for onsite and online analysis of pig manure using visible and near infrared reflectance spectroscopy. Biosystems Engineering. 91: 393–402.
Sharif S A, Norlindawati A P, Noormah Miw A, Sabariah B, Omar R, Samijah A, Supie J, Jamnah O (2012). Nutritive value of palm kernel meal since 1999 to 2010 based on samples analyzed by animal feed laboratory, IVM**.** Proceeding of the Department of Veterinary Services 2nd Research Convention 2012, 23-24 October 2012, Bankuet Hall UPM, Serdang. Pg 48 – 50.
Sohn M, Himmelsbach DS, Barton FE II, Griffey CA, Brooks W, Hicks KB. (2007) Near-Infrared Analysis of Ground Barley for Use as a Feedstock for Fuel Ethanol Production. Appl Spectrosc. 61(11):1178-83.
Undersander D (2006), "Uses and Abuses of NIR for Feed Analysis", Florida Ruminant Nutrition Symposium, Gainseville. [Online, accessed July 13, 2011] URL:http://dairy.ifas.ufl.edu/rns/2006/Undersander.pdf.
van Kempen T, Simmins PH (1997). Near-infrared reflectance spectroscopy in precision feed formulation. J. Appl. Poultry. Res. 6: 471 – 477.