Usage of Near Infrared Spectrometer as An Analyzing Tool for Nutrients in Leaf, Fertilizer and Soil in Oil Palm Industry

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Abstract. In oil palm industry, a huge money is used for chemical items to carry out examination on samples especially for leaf (foliar), fertilizer and soil. However, with advent of FOSS NIRS DS2500 technology, there is a huge reduction in chemical usage in laboratories. Results were attained in less than a minute while covering many parameters in a single scanning. This tool uses the technology of Near Infra Red Spectroscopy (NIRS) to scan the particles of the substance and thus, elements are detected within NIRS wavelength region. FOSS NIRS DS2500 uses a broad wavelength range of 400 to 2500nm, covering a highest possible performance across on any chemical elements/molecules. Furthermore, there is accurate analysis results for a wide range of parameters, such as oil content, moisture content, N, P, K, Mg, Ca, B, Zn, and Cu. The objective of this paper is to discuss the '*hands-on*' experience on the usage of FOSS NIRS DS2500 as an analyzing tool for determining the nutrient values for the above-mentioned samples. FOSS NIRS DS2500 is a 'secondary method' as this tool need to be calibrated periodically with database gathered from traditional laboratory (primer method/conventional method). Statistics is used to verify the accuracy and acceptance level of data, for example standard error of prediction, standard error of cross validation and standard error lab. Unification usage of applied science of statistics, biochemistry of plant tissues, appropriate sampling methods in the fields had resulted in a practice where huge use of chemicals are eliminated while accuracy of data are retained.

Keywords: foliar samples, FOSS NIRS DS2500, statistics, standard error of prediction, standard error lab

1 Introduction

The success of an oil palm plantation business is inseparable from the efficiency factor. Increasing efficiency can be done by reducing cost per unit of output as low as possible, without a reduction in yield or quality achieved. One of the alternative fertilization cost efficiency measures that can be taken is to increase the effectiveness in the formulation of fertilizer application rates [1] [2]. For effective formulation of fertilizer application rates, it is imperative to have well-interpreted results of analysis especially of those leaf, soil and fertiliser samples. There were well established standard operation

procedures for oil palm plantations to carry out leaf, soil and fertilizer samplings as well as for respective chemical/nutrient analysis [3]. Furthermore, nutrient analysis for leaf, soil and fertiliser samplings are generally carried out by ISO-accredited chemical laboratories.

Apart from these, there are also opportunities for analysing leaf, soil and fertiliser samples while maintaining the accuracy of results by using Near Infrared Spectroscopy (NIRS) technology [4][5][6]. NIRS technology is not a new to agriculture as this NIRS technology and similar concepts were used in other agricultural products such as sugarcane and orange. A summary on the usage of NIRS in the agricultural industry with selected references is given in **Table 1**. Measurement by using NIRS technology is actually an 'indirect method' that requires the development of a multivariate calibration model against a suitable reference method. Validation and prediction models for NIRS technology especially for soils, plant tissues and fertiliser were already carried out by numerous researchers in spectroscopical fields [7][8][6]. [8], had concluded that spectroradiometer able to predict nutrient deficiencies in oil palm with reference to frond 17, using partial least square (PLS) analysis. [9] also had confirmed the use of the spectrometer in oil palm. They reported that a correlation of NIR spectral absorbance data and chlorophyll can be achieved, using a PLS regression model with Savitzky-Golay Smoothing (SGS). The overall results show, SGS has the best performance for pre-processing method with the results, the coefficient of determination (R^2) values of 0.9998 and root mean square error (RMSE) values of 0.0639. Thus, they concluded that NIR spectroscopy method can be used to identify chlorophyll content in oil palm leaves.

This paper discusses on the usage of NIRS in determining nutrients of leaf, soil and fertilizer samples at a commercial scale, in oil palm industry. The objective of this paper is also to reconfirm the accuracy of data obtained by NIRS technology in Sumber Tani Agung Resources Tbk (STAR) and the relationship of analytical results from chemical laboratories that uses both traditional/conventional/standard methods and NIRS technology.

1.1 What is NIRS?

Near Infrared Spectroscopy (NIRS) is the study of the absorption of near infrared light (energy) by molecules. Changes in the absorbance of an element are interpreted into data. Wavelength area for NIRS generally falls in between 800 to 2500 nm as illustrated in **Figure 1**. In STAR, 4 units of FOSS NIRS DS2500 model is employed for performing NIRS technology in the laboratories. This FOSS NIRS DS2500 is capable of using a broader wavelength ranging from 400 to 2500 nm.

Figure 1: An illustration depicting the position of NIR in the electromagnetic spectrum

Table 1: Summary on the use of NIRS in the agricultural industry with selected references

2 Materials and Methods

2.1 Study Area

All leaf, soil and fertilizers were sampled periodically for analysing purposes from 19 estates, owned by Sumber Tani Agung Resources Tbk (STAR), Indonesia. All the calibration and validation of data against the conventional/standard method/reference were done with the master unit. The master unit plays an important role as a primary storage of all validated data. The client units were standardized with this master unit. In STAR, there are one master unit and 3 client units as shown in **Table 2.**

N ₀	with FOSS NIRS Laboratory DS2500	Coordinates	Remarks
	KSJA Lab, Tebing Tinggi, North Sumatra, Indonesia	N 3.345428 E 99.220680	Master unit
$\mathcal{D}_{\mathcal{L}}$	PMKS TPAI Lab, Palembang, South Sumatra, Indonesia	N 2.803126 E 104.8976	Client unit
3	PMKS TPA Lab, Palangkaraya, Central Kalimantan, Indonesia	N 1.423716 E 113.400252	Client unit
4	PMKS KSUP Lab, Sambas, West Kalimantan, Indonesia	N 1.038844 E 109.308216	Client unit

 Table 2: Location of Laboratories in STAR and Details on Master and Client units

2.2 Sample Preparation

There are three main types of samples tested for its nutrient contents, namely leaf, fertilizer and soils. Instrument that used for analysing these three types of samples was FOSS NIRS DS2500 which was designed and made by FOSS. The preparations of samples for analysing in FOSS NIRS DS2500 are summarized below (*2.2.1-2.2.3*). All samples divided into two portions; first portion for analysis with FOSS NIRS DS2500 while second portion was sent to traditional/conventional chemical laboratories for reference method.

2.2.1 Sample Preparation for Leaf Analysis. Oil palm leaves were sampled at 17th frond as per STA Resources's policy. Leaf samples were collected within a block of sizes that varies from 25-40 ha. Leaf samples collected from fields were oven-dried on the same day of sampling. Leaf samples were oven-dried for 6-8 hours at the temperature about $70 \pm 10^{\circ}$ C [3]. The time of drying varies depending on the size and nature of the material. The sample is considered dry when it is crisp to the touch, and quite brittle; and stored in a closed clean polythene bag. The dried sample was passed through a stainless steel grinding mill (RT-N04 model) fitted with a 2mm sieve and the grounded sample stored in closed wrap polythene bag.

2.2.2 Sample Preparation for Soil Analysis. Thoroughly remove all foreign matters from the soil. Soil samples were grinded then crushed into powder (<2mm) similarly in step *2.2.1*.

2.2.3 Sample Preparation for Fertilizer Analysis. The sample was homogenously mixed with tablespoon and 1 to 3 tablespoon of fertilizer sample was scooped from each bag into composite sample bag. The quantity of composited sample was not less than 50g. The composited sample was grinded to a fine homogenous powder using the grinder. The grounded sample was transferred into a plastic bag and tied it with rubber band to provide airtight closure. The composited sample is ready for analysis. The remaining sample shall be kept in the store for retention and will be disposed after the testing of composited sub-sample is completed.

The above *2.2.3* procedures are valid and same for compound/granular as well as for straight fertilizer.

2.3 Preparation for Scanning

Sample preparation for scanning by the NIRS Technology /FOSS NIRS DS2500 is one of the most significant steps in ensuring the accuracy of the results. The powdered sample is stired well so that it is homogeneous.

Samples were scanned on NIRS using small cup (**Figure 2(a), (b) & (c)**). The labelled sample would be filled into the small cup, flatten the sample/compact it so that there are no translucent cavities when the small cup is exposed to the light from below. Powdered materials \langle <2 mm) of leaf samples, fertilizer samples and soils should be placed in the small cup and inserted into small cup holder in FOSS NIRS DS2500 model for scanning. The type of product for analysis is selected by selecting the estate menu, (e.g. for leaf or select the leaf sample analysis) and start the analysis. An average from three readings would be taken after scanning. The analysis results are automatically saved/stored in a software or a computer in real time. Flow-chart for operating NIRS technology /FOSS NIRS DS2500 for leaf, soil and fertilizer samples are illustrated in **Appendix 1.**

Figure 2: Powdered in a special small cup for scanning by FOSS NIRS DS2500: (a) leaf samples (b) soil samples (c) fertilizer samples

3 Results and Discussion

A simple correlation analysis was conducted to estimate the relationship between the results obtained from standard methods in a conventional chemical laboratory and the NIRS technology by FOSS NIRS DS2500. The results for leaf, soil and fertiliser parameters are shown in **Table 3,4** and **5** respectively.

3.1 Leaf

A total of 45 samples were used for determination of leaf parameters such as Ash, N, P, K & Mg, while for leaf Ca and B, a total of 36 samples were analysed. Only 8 leaf samples were used for analysis for Cu, Fe, Zn and Mn determination as shown in **Table 3**. The value of coefficient of determination (R^2) values from 0.2849 (leaf B) to 0.9639 (leaf Ash) were obtained for the leaf samples.

[4] had confirmed that leaf samples tested with FOSS NIRS DS2500 instrument gave no significant difference at P=0.01 level when compared with those tested with conventional chemical laboratories (using standard methods). This means all the readings from FOSS NIRS DS2500 had only a minimal variation from those of conventional chemical laboratories. R^2 values as shown in **Table 3** also in line with findings of [4], where R^2 for N, K, Mg, Ca, Cu and Fe recorded more than 0.5.

However, among the leaf N, P, K and Mg values which considered as main/major nutrients in oil palm nutrition studies, leaf P recorded with lower range of \mathbb{R}^2 values (0.0936). [7] also reported lower \mathbb{R}^2 values for soil P content.

\mathbf{u} character chemical necessary, that \mathbf{u} such an \mathbf{u} method						
Parameter	n		\mathbf{R}^2			
Leaf Ash	45	$y = 0.8266x + 0.9747$	0.9639			
Leaf N	45	$y = 0.7284x + 0.5633$	0.7510			
Leaf P	45	$y = 0.1768x + 0.1243$	0.0936			
Leaf K	45	$y = 0.8469x + 0.0679$	0.8544			
Leaf Mg	45	$y = 0.9656x + 0.0476$	0.8399			
Leaf Ca	36	$y = 0.6031x + 0.193$	0.6316			
Leaf B	36	$y = 0.4541x + 14.209$	0.2849			
Leaf Cu	8	$y = 1.0212x - 0.5169$	0.6085			
Leaf Fe	8	$y = 0.4062x + 26.506$	0.5265			
Leaf Zn	8	$y = -0.0212x + 20.367$	0.3300			
Leaf Mn	8	$y = 0.3099x + 140.45$	0.4809			

Table 3: Relationship on leaf parameters determined by NIRS technology (FOSS NIRS DS2500) and conventional chemical laboratory that using a standard method

3.2 Soil

The value of coefficient of determination (R^2) for soil chemical properties such as soil nitrogen, average phosphorus, total phosphorus, potassium and magnesium had registered of 0.8839, 0.8836, 0.9357, 0.7721 and 0.2785 respectively.

Soil pH (H₂O) was also highly correlated between measured and reference readings with R^2 value at 0.8768. Results obtained for soil chemical properties of soil nitrogen, average phosphorus, total phosphorus, potassium were highly correlated with $R^2 > 0.75$.

Parameter	n		\mathbf{R}^2
Soil CEC	20	$y = 0.853x + 0.3961$	0.9260
Soil N	20	$y = 0.7957x + 0.0134$	0.8839
Soil Av P	20	$y = 0.7243x + 0.2574$	0.8836
Soil Total P	20	$y = 0.9868x + 21.94$	0.9357
Soil K	20	$y = 0.4325x + 0.0437$	0.7721
Soil Mg	20	$y = 0.5116x + 0.0487$	0.2785
Soil Ca	20	$y = 0.7741x + 0.0562$	0.4267
Soil B	20	$y = 0.4155x + 26.126$	0.2883
Soil Cu	20	$y = 0.4075x + 1.1886$	0.3326
Soil Zn	20	$y = 0.7423x + 2.0869$	0.9425
Soil Mn	20	$y = 0.0279x + 15.045$	0.0030
Soil Fe	20	$y = 3.0207x + 10.026$	0.0067
Soil Na	20	$y = 0.629x + 0.0053$	0.5392
Soil $pH H2O$	20	$y = 0.8825x + 0.5882$	0.8768

Table 4: Relationship on soil parameters determined by NIRS technology (FOSS NIRS DS2500) and conventional chemical laboratory that using a standard method

3.3 Fertilizer

Analysis results of fertilizer samples shown in **Table 5** are from those of compound NPK 13:6:27:4+.0.65B. Except for fertiliser B, all other fertiliser parameters are generally having higher or satisfactory \mathbb{R}^2 values. Fertiliser K value in Compound NPK 13:6:27:4+0.65B is having high correlation between measured and reference readings with \mathbb{R}^2 value at 0.9015. \mathbb{R}^2 values for the fertiliser samples are ranging from 0.2910 (fertilizer B) to 0.9015 (fertilizer K). Results observed for fertiliser properties of nitrogen, potassium and magnesium were highly correlated with conventional

chemical laboratory. From the **Table 5**, it is observed that the calibration performance as indicated by $R²$ were very good for all three nutrients except for fertiliser P with low $R²$ value of 0.3558. Similar recording of low \mathbb{R}^2 value for P also registered in other works as observed by [14].

Table 5: Relationship on fertilizer parameters determined by NIRS technology (FOSS NIRS DS2500) and conventional chemical laboratory that using a standard method

Among the three types of samples, the value of coefficient of determination (R^2) for soil parameters are relatively higher than those from leaf and fertilizer parameters. There was a lower R^2 especially among the fertiliser parameters such as moisture content, P and B. It would be appropriate to increase the number of data to be measured for these three low \mathbb{R}^2 parameters. Correlation analysis would be better if database is increased, covering the high and low range of each parameter. As such, these samples should be analysed frequently with FOSS NIRS DS2500 and sent to conventional chemical laboratories for reference method. Actually, FOSS NIRS DS2500 is a 'secondary method' as this tool need to be calibrated periodically with database gathered from a conventional/traditional laboratory. Diagrams showing the relationship between the FOSS NIRS DS2500 (predicted) and conventional chemical laboratory (measured/reference) are shown in **Figure 3, 4** and **5** for leaf, soil and fertiliser parameters respectively.

3.4 Statistics

As it is not the objective of this paper to give in-depth statistics for validation of prediction, only basic concepts on calibration of NIRS/FOSS NIRS DS2500 are discussed. Statistics is used to verify the accuracy and acceptance level of data, by using standard error of prediction (SEP), standard error lab (SEL) and standard error of cross validation (SECV). FOSS NIRS DS2500 already have a software for calibration purposes, called Foss Calibrator or WinISI.

For statistics purpose, data collected from NIRS/FOSS NIRS DS2500 are called as '*predicted values*'. Meanwhile data collected from a conventional laboratory are called as '*Lab/measured values'*. Any outlier from the analysis results of predicted values and measured values should be rejected. Analysis results from both sources namely FOSS NIRS DS2500 and Laboratory are keyed-in into excel format/software programme. Once the details of analysis results from both sources are keyed-in, the programme calculate (i) SEP, SEL (ii) and (iii) SECV (**Figure 2**). F-test is used to compare SEP and SEL ($F = square$ (SEP) / square (SEL)). The calculated F value is compared with Fcriteria (0.05, v1, v2). If the calculated F value is smaller than the Fcriteria, then the NIRS method is equally accurate as the reference method.

Finally, three validation status are determined by the Foss Calibrator namely, Calibrate, Adjust Bias and Valid. Based on these three-validation status, NIRS/FOSS NIRS DS2500 would be calibrated for better performance.

(a)

 (h)

(c)

Figure 2: An example of data input for analysis results from both (a) FOSS NIRS DS2500 (predicted) and (ii) Chemical laboratory (measured) done in STA Resources Tbk, (b) Based on acceptance criteria, SECV value input for determining validation status such as Calibrate, Adjust Bias, Valid, for each parameter and (c) SECV value is obtained from FOSS NIRS Programme for each parameter

Figure 3: Relationship of Ash, N, P, K, Mg, B, Cu and Zn elements between FOSS NIRS DS2500 (predicted) and conventional chemical laboratory (measured/reference) values for leaf sample

Figure 4: Relationship of CEC, N, Total P, K, Mg and Ca elements between FOSS NIRS DS2500 (predicted) and conventional chemical laboratory (measured/reference) values for soil sample

Figure 5: Relationship of moisture, N, P, K, Mg and B between FOSS NIRS DS2500 (predicted) and conventional chemical laboratory (measured/reference) values for fertilizer sample

3.5 Benefits of Using NIRS

The device /instrument for using NIRS technology is made more simple and easily available to endusers. The use of NIRS technology in oil palm industry is preferred mainly due to

- (i) time saving: There is a huge time-saving, as this instrument is able to detect the samples and scan the nutrient content, usually within 40 seconds. This results in more time saving as well as labour saving compared to those of Chemical laboratories that using standard methods. According to [15], under the NIRS technique in the laboratory most measurement takes a few seconds while several soil properties can be estimated from a single scan.
- (ii) Multi nutrient status by a single scanning: With NIRS technology, widely used elements in oil palm nutrition can be scanned/screened in a single scan in leaf samples, such as Nitrogen, Phosphorus, Potassium, Magnesium, Calcium, Borate, Copper and Zinc. It saves money as well as time [4]. Nevertheless, elements that not frequently tested also can be included for scanning, such as Ferrum (Fe) and Sulphur (S) in leaf samples.
- (iii) Use no chemicals: In oil palm industry, most of the chemical laboratories (traditional/standard methods), still need to use acids in the process of analysing nutrients/elements, for example Hydrochloric acid. However, sample preparation for NIRS scanning involves only drying and crushing. This means that all samples of leaf, soil and fertiliser are not subjected to the use of (hazardous) chemicals [15].
- (iv) Expenditure for chemicals that being used in a typical chemical laboratory would be approximately 20% of total cost of analysing a sample. As laboratory with the facility of NIRS technology should be calibrated periodically, there is still a need for a typical chemical laboratory to carry out their routine analysis in accordance with standard methods for respective sample type. As such for a standard chemical laboratory, there would be only a cost reduction of about 10-15% of total cost of analysing a sample. Chemical laboratory remain in full function as at least 10% of total sample of each type (namely leaf, soil and fertilizer) would be tested for the purpose of FOSS NIRS DS2500 calibration.

With the above four items, NIRS technology gave a more productive as well as an efficient way for identifying nutrient contents especially for leaf, soil and fertilizer samples in oil palm industry [4]. Although NIRS technology has a potential for highly sustainable with reduced usage of chemical items, we still need a standard chemical laboratory. The results of NIRS technology are usually referred and compared with the results of standard chemical laboratory for calibration of data or to validate the results of those from NIRS.

A total of 1721, 2645 and 2850 samples comprising leaf, soil and fertilizers samples were analyzed using NIRS technology in 2020,2021 and 2022 respectively in STAR. Total savings after the deduction of expenditure for analysing reference samples in conventional/traditional chemical laboratory are Indonesian Rupiah 148,734,000, 537,480,900 and 595,787,356 for the years of 2020, 2021 and 2022 respectively. With assumption of conversion rate of $1 \text{ USD} = \text{Rp } 15,000$, there would be a savings of USD 9,915 (2020), USD 35,832 (2021) and USD 39,719 (2022), totalling USD 85,466 savings since embarkment of this NIRS technology/ FOSS NIRS DS2500 for chemical analysis in STAR. [4] also reported that FOSS NIRS DS2500 is able to reduce cost per sample approximately 68% of the analysis cost of traditional/conventional chemical laboratory.

Figure 6: Details on annual savings due to usage of NIRS technology/FOSS NIRS DS2500 for determination of nutrient values in leaf, soil and fertilizer samples in STA Resources Tbk

4. Conclusions

The recent introduction of NIRS technology in oil palm industry with a reasonable prediction of the chemical contents especially in plant tissue studies has enabled the industry to achieve several benefits. The device /instrument for using NIRS technology is made more simple and easily available to end-users.

The use of NIRS technology in oil palm industry is preferred mainly due to (i) time saving as all measurements takes a few seconds only, (ii) Multi nutrient status by a single scanning, (iii) no usage of hazardous chemicals and thus more sustainable and (iv) highly potential to reduce expenditure on chemical laboratory (traditional/conventional) for determining nutrient status especially in plant tissues as well as in soil and fertilizers. Unification usage of applied science of statistics, biochemistry of plant tissues, appropriate sampling methods in the fields had resulted in a practice where huge use of chemicals $(>10\%)$ are eliminated while accuracy of data are retained.

Although NIRS technology has a potential for highly sustainable with reduced usage of chemical items, we still need a traditional/conventional chemical laboratory. The results of NIRS technology are usually referred and compared with the results of standard chemical laboratory for calibration of data or to validate the results of those from NIRS. In short, NIRS technology/ FOSS NIRS DS2500 gave a more productive as well as an efficient way for identifying nutrient contents especially for leaf, soil and fertilizer samples in oil palm industry.

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Appendix 1: Flow-chart for operating NIRS technology /FOSS NIRS DS2500 for leaf, soil and fertilizer samples in STAR

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