Benefits of Using Near Infrared Analysis in Palm Oil Mills for Quality Control – An IOI Experience

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The palm oil industry is an important pillar of the economy in some ASEAN countries, with huge acreage devoted to the planting of this high oil yielding crop. The total acreage under palm oil cultivation in 2017, has reached almost 5.77 million hectares, 11.90 million hectares and 0.75 million hectares in Malaysia, Indonesia and Thailand respectively. There is a huge number of crude palm oil (CPO) extraction mills and refineries constructed to extract and refine the palm oil. It has been a constant drive for the industry to enhance the oil extraction process efficiency in the mills and refineries.

Sample analysis provides insights into the quality of palm oil fruits, effectiveness of oil extraction process and loss in the waste. Common samples tested in the palm oil mill operation include press cake fibre (PCF), sludge and CPO. Chemical analysis using titration and Soxhlet extraction are often used, but they have shortcomings including being time consuming, requiring heavy use of solvents and solvent disposal issues. Some chemicals, such as hexane, can be health hazard to the operator. In recent years, a palm oil analyser based on an FT-Near IR has been introduced by PerkinElmer to perform these analyses and the technical merits of these new techniques are worth mentioning. The collaborative efforts between IOI Mills and PerkinElmer using the Frontier FT-Near IR Palm Oil analyser, in several Peninsula Malaysia and Sabah mills are discussed. The samples analysed so far are PCF, sludge and CPO.

TABLE 1. STANDARD CHEMICAL ANALYSIS FOR SLUDGE, CPO AND PCF AS ESTABLISHED BY MPOB

Sample Matrix	Sludge 	Crude Palm Oil (CPO) 	Press Cake Fiber (PCF) 
Objective of analysis	Oil loss in sludge indicates oil recovery effectiveness	FFA and moisture indicated quality of CPO. Determine price of CPO.	Oil loss in fiber indicates extraction efficiency
Conventional Wet Chem Method	nHexane Soxhlet extraction followed drying in oven until weight stabilises at 105°C.	KOH titration method for FFA and oven drying until weight stabilises at 105°C.	nHexane Soxhlet extration followed by drying in oven until weight stabilises at 105°C.
Turn Around Time	4 hr - 6 hr	4 hr - 6 hr	5 hr - 8 hr

The standard chemical analysis for sludge, CPO and PCF are well established by MPOB and briefly described in *Table 1*. Wet chemistry methods, while simple to perform, are very time consuming. This means that the chemical data, pertaining to oil loss in PCF and sludge, which indicates the milling process efficiency was not made available to the plant manager during batch processing. Chemical data is usually available on the following day. Yield losses cannot be recovered or corrected as a result. When adjusting milling parameters, plant manager will only get to see the result the following day. This limits the ability of plant manager to manage mill operation process control and yield losses. Plant manager can only evaluate the oil extraction rate (OER) yield and understand the extraction situation in hindsight.

The FT-Near infrared technique uses a beam of infrared radiation which passes though or is reflected from the sample. The spectrum obtained contains information about the sample analysed. For CPO, the sample was placed in a heated glass vial, while for PCF and sludge, the samples were placed in a petri dish and the infrared radiation is reflected from the bottom of the petri dish. Chemical analysis data was entered into chemometric models for PCF, sludge and CPO, which were built with these sampling techniques with over 200 spectra collected with chemical analysis data. Chemometric means performing calculations on measurements of chemical data. The chemometric models correlate the information in a set of known measurements to the desired property of oil content, moisture and FFA. These models take a period over several months to build to get a true representation of the data population. These calibration models require a well spread out data range of chemical values to create good calibration curves but on many occasions, we have clustering of chemical values. The models after completion and validation were used to predict the related properties. Unknown samples can then be predicted with these models in a relatively short time compared to chemical analysis methods. For oil loss analysis, a single NIR scan will only take <10 min to prepare and produce results. Using RAPID NIR, conventional lab wet chemistry sometimes will take around 12 hr - 20 hr to complete. RAPID NIR presents an easy sample preparation compared to lab wet chemistry which involves drying, Soxhlet extraction, and weighing, *i.e.* processes that increase the probability of human error in sample handling and calculation.

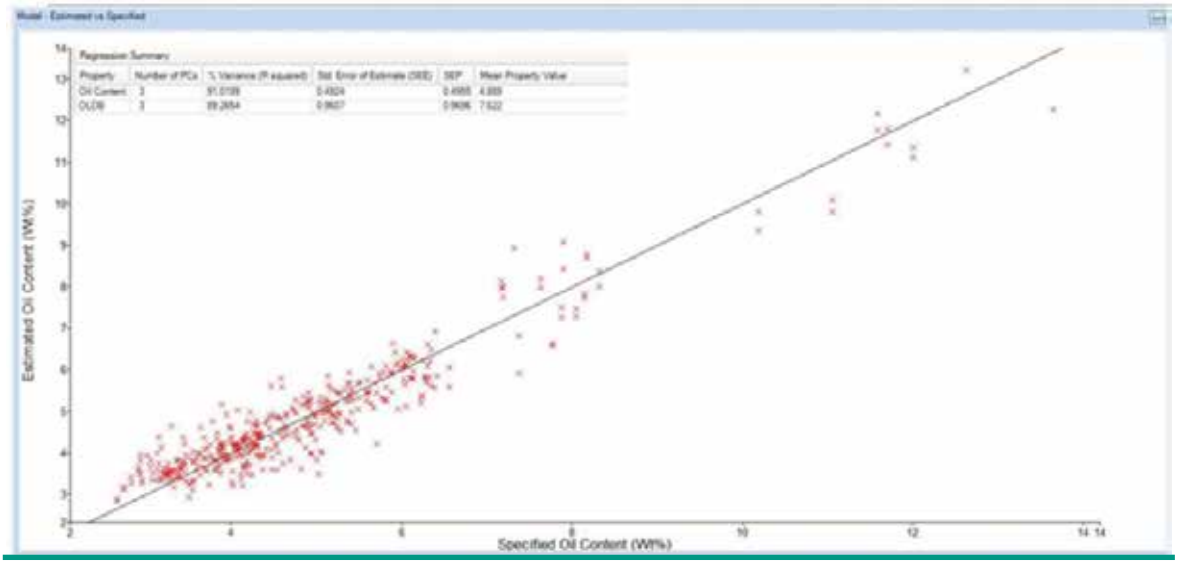

Figure 1. Oil content calibration model based on specific oil content (from wet chemistry analysis) and estimated oil content (modelled via chemometric algorithm).

TABLE 2. NIR TECHNIQUES FOR DIFFERENT SAMPLES, ACCESSORY USED AND TIME TAKEN FOR ANALYSIS

NIR Technique	Sludge 	Crude Palm Oil (CPO) 	Press Cake Fiber (PCF) 
Mode	Diffuse Reflectance	Heated Transmission	Diffuse Reflectance
Turn Around Time	4 min	2 parameters in 4 min	4 min
Consumables	**No chemical** used and no sample preparation	**No chemical** used and no sample preparation	**No chemical** used and no sample preparation

Besides fast turnaround time, other advantages are little sample preparation is required and no chemical was used. A larger number of samples per day can be analysed since each instrument analysis does not require much time. RAPID NIR allows tighter and more frequent process monitoring on process oil loss. Averagely in IOI, four times (based on 20 hr milling) more sampling and testing frequency were achievable compared to conventional lab method. Before implementation of NIR, the laboratory in IOI mill can only do a day composite analysis and give an after-action report for post-process review. In contrast, with NIR, the IOI Mill laboratory was able to conduct a four hourly sampling and monitoring. Immediate action can be taken with the process adjustment based on the fast NIR results. The ability to use RAPID NIR in troubleshooting situation where the mill needs to know the results rapidly for process

Quant Results

Sample Name	FFA (%)	IV	Moisture (%)
CPO001	2.7632	52.9957	0.2836

Figure 2. Variate parameters for CPO can be predicted simultaneously via the built calibration models.

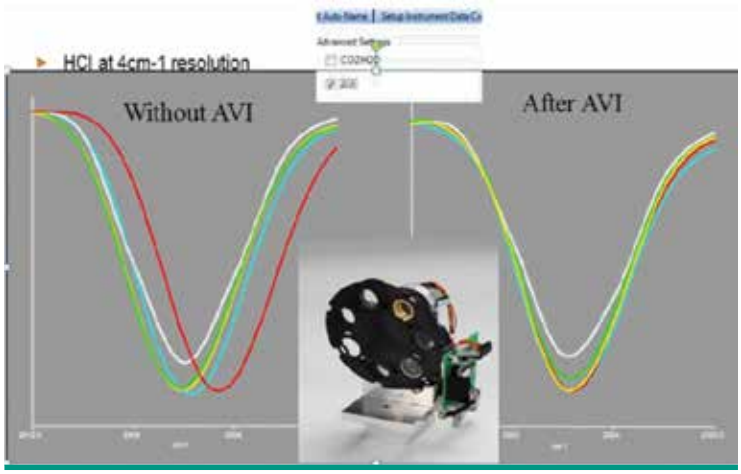

Figure 3. HCl peaks from different FTIR instruments with and without the AVI correction.

control setting change gives Plant manager better control over the mill operations. The potential saving from oil losses is another source for the return of RAPID NIR investment.

Table 2 shows the NIR techniques for different sample types, the accessory used, and the time taken for each analysis. *Figure 1* displays the oil content calibration model built based on specific oil content (from wet chemistry analysis) and estimated oil content (modelled via chemometric algorithm).

With the models completed, IOI operators were trained to operate the instrument and to perform predictions with unknown samples. On some occasions, cross-checks were performed with chemical analysis data. Standard operating procedures were made to ensure that the essential steps are understood and followed by the operators. New concepts such as Standard Error of Laboratory (SEL) which is the error of the reference chemical analysis method, Standard Error of Prediction (SEP) which is the accuracy of the NIR-method were introduced. The SEP is the sum of the reference error and the error of the NIR system which is always larger than SEL. One of the great advantage of the Near IR technique is that it is possible to obtain multiple parameters at once with a single prediction based on multiple built calibration models as shown in *Figure 2*. Each of these parameters needs to be performed individually with chemical analysis. This provides considerable time savings in sample analysis. This speed in sample analysis can translate to faster decision making relating to product quality for incoming raw materials, finished products and in-process control.

When two additional units of the Frontier FT-Near IR instruments were commissioned in Sabah mills, the calibration models from IOI mill were transferred to these two mills. Instead of creating new calibration curves, validation was performed on these two instruments to demonstrate that the models remain valid on the second instrument. The models were validated with some sample spectra collected with the instruments located at these two mills and some samples taken from the vicinity of these two mills. PerkinElmer FT-NIR has a unique tool, the Absolute Virtual Instrument (AVI), which can assist with the transfer of calibration models. This help to save tremendous amount of time spent developing calibration when new instruments are added or after a major instrument repair job. AVIs maintain the integrity of the calibration model when it is transferred to a new machine. *Figure 3* shows the results of HCl peaks from different FTIR instruments with and without AVI correction. This inconsistency exists for all commercial IR and NIR spectrometers. There is a significant improvement in consistency between the measurements. Calibration with AVI is simple, by just clicking on the Standardise button, the methane filter on the filter wheel rotates into the beam path and a methane spectrum will be collected. A correction function is generated from this measured methane spectrum with a 'theoretical' master. This correction can be applied to all subsequent spectra with the touch of a button.

The main benefits of AVI are improved calibration between instruments and easy recalibration when components in the instrument are changed. The AVI function helps to protect these valuable calibrations when these events occur especially when the user has many calibrations. The on-demand recalibration capability provides assurance throughout instrument life.

For validation of the models, spectra (>30 spectra) were scanned with some samples taken from these two mills, using the two instruments. The main purpose of validation for a calibration done on the same instrument is to check whether the calibration curve is representative of the sample set that the user is predicting. The validation curve determines what and how much adjustment to be made since different instruments and samples are used in two

different locations. In an ideal case, the calibration and validation curves fit exactly, and no correction is required, the calibration curve can be used as it is. However, in some cases, there are some differences in the slope and offset between the calibration and validation lines. An adjustment is then made to the calibration curve. If the validation proceeds well, the instrument is ready to be used. The ease of validation varies with sample type. PCF samples are relatively easier to validate and sludge samples less so.

Once the calibration has been finalised, prediction of unknown samples can be performed. Once the unknown sample spectrum is collected, the prediction procedure is entirely automatic. Unknown prediction spectra which is not representative of the calibration can be detected. Perkin Elmer's Advanced Algorithm provides two measures; the Mahalanobis Distance Ratio and the Residual Ratio. The total Mahalanobis distance ratio must be less than 1, and the Residual Ratio must be less than 3, if complete confidence is to be attached to the analysis. For each unknown sample analysis, the prediction is accompanied by Mahalanobis Distance Ratio and Residual Ratio to help confirming if the analysis is good.

Operators were taught to run these validated models for their unknown samples. The Standard Operating Procedure (SOP) sets out clearly the steps that should be performed with the validated models. Faster analytical results turnaround helps to improve lab efficiency and affects the way process control is managed. As the data obtained are almost real time, process such as pressure exerted on the press cake during pressing can be adjusted to maximise oil yield. Oil loss information from the press cake fibre is no longer something that you know much later after the batch run has been completed but is a controllable process with the availability of almost real time analysis data.

The return of investment (ROI) is always a consideration when purchasing a major piece of equipment like the palm oil analyser. The quick turnaround time, the larger number of samples the instrument can analyse in a given period of time, the multiple parameters that can be obtained simultaneously, solvent-free running *etc.* factors into the consideration of the purchase as a percentage of the cost of the investment. IOI has managed to reduce the purchase of Hexane in mills which NIR dropped by 20% - 40% after RAPID NIR was introduced. RAPID NIR enables chemical free analysis, reduction of laboratory scheduled waste, disposal cost and significant chemical exposure risk reduction for laboratory personnel.

How successful the instrument is performing in comparison with traditional chemical techniques becomes clear with the implementation of the palm oil analyser over time. IOI has over a period of three years, purchased seven units for Peninsular Malaysia and Sabah mills after considering the benefits with more experience in running the palm oil analyser. Implementing the palm oil analyser in the laboratory for these mills is a technology upgrade to improve yield to address a key challenge in the palm oil industry.