

## **The NIRS method to support practical feed formulation**

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### **Abstract**

Feedstuffs used in poultry nutrition are derived from multiple sources and exhibit a large variation in nutritional value. Most of the techniques used to quantify are not practical, being time consuming, slow and in some cases expensive, hence often final diets are more variable than desired. Reducing variation will improve the production efficiency in the mill and also the bird performances. The object of this paper is to evaluate, through the development of proximate parameters NIRS calibrations and a simple example of formulation table value management, the potentials of NIRS as a tool to support feed formulation. NIR calibrations explained between 80 and 98% of the variation for the overall proximate criteria. A simulation has been performed using the Crude Protein content variation of 33 batches to evaluate the potential gain between 3 cases : Case 1 : where a margin of safety is used on the table value for all the period of time (Table value = mean-0.5xStandard Deviation of the set), Case 2 : where the table value is reviewed time to time depending on the wet chemistry analysis availability, Case 3 : where NIRS analysis are used each 3 batches following delivery time to review the table value. The NIRS method has been shown to be capable of being a practical, accurate and rapid method of nutrient prediction which would allow feed costs reduction through a more important quality control pressure and a more reactive adjustment of the feed formulation.

### **Introduction**

#### **Feed Formulation**

Overall, the animal feed industry is based on the search for the least cost formulation. Ingredient quality and prices may vary a lot as a function of their origin and the market. Several factors can affect the formulation but the 3 most important in balancing a diet are nutrient requirements of the bird, nutrient contents of the feedstuffs and nutrient availability. It is well known that there is a variation in nutrient content of ingredients and to be sure that the animal requirements are met, it is usual to provide a safety margin when formulating the diets. This is the case for vitamins but it is also used for protein.

It has been demonstrated through different formula simulations that one of the principal ways to produce an optimal feed formulation is a good knowledge of the ingredients nutrient composition and availability (BUSHMAN, 1998).

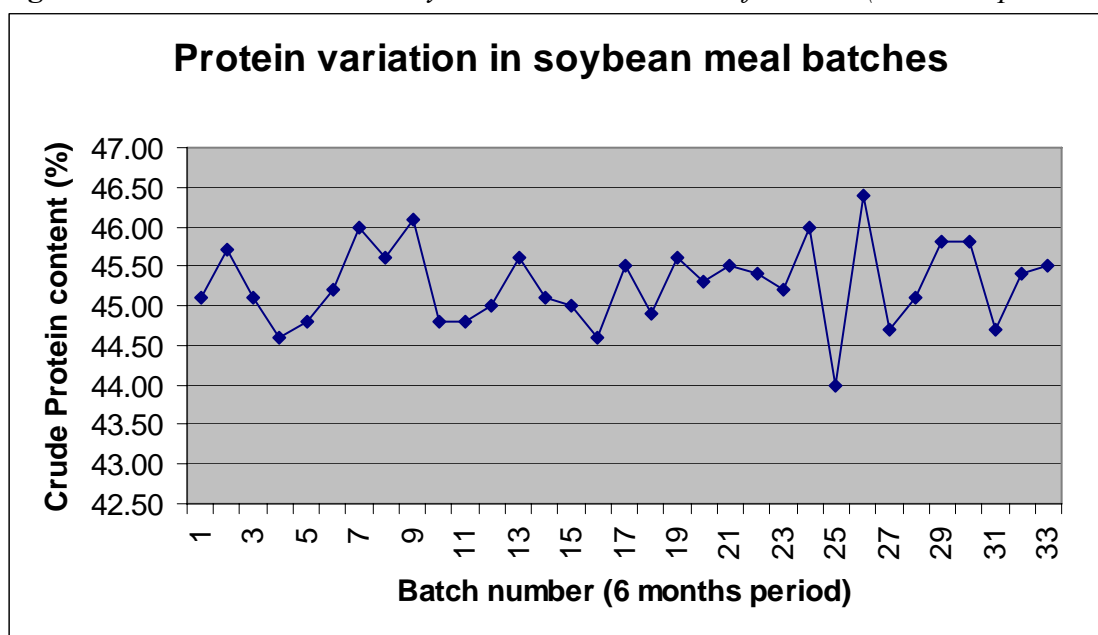
On the world – wide basis and particularly in Europe, animal feed are comprised of a more or less large range of ingredients. The different raw materials used are formulated at least costs with the goal to maximize the animal performances of the animals to which they are given. The objective of the feed formulation process is by the way to mix thee different feedstuffs in order to obtain the optimum balance of nutrients to fit with the nitrogen and energy requirements.

#### *Feedstuffs variability*

The figure 1 represents the protein evolution of soybean meals batches on a one year period of time by one feed-mill. These different samples are identified to be one identical product.

It is quiet obvious that in this case , the customer has to improve the analysis pressure to get a clearer view of the variations trends of the arriving samples. Some of the soybean batches may sometimes have more than the specified amounts of nutrients, which can lead to final diets nutrient deficiencies, and on an economical aspect, some batches could have been discounted.

**Figure 1 :** Protein variation in soybean meal batches in a feed mill (6 months period of time)



As it has been widely demonstrated through several scientific publications (VAN BARNEVELD, 2003), that deficiencies of the main nutrients like energy, proteins, amino acids, will get a direct impact on animal performances. On the other hand, relative excess of such nutrients will lead to possible accretion of fat and waste of amino acids which can increase nitrogen pollution impacts. Often, these nutritional deficiencies can cause measurable animal performances problems. For this reason, nutrients are often given a margin of safety for naturally occurring variance. But using a margin of safety does not reduce variability, which is the real problem.

The raw materials used by TECHNA customers are from multiple sources and origins, and also characterized by a large variation in nutritional value. Quiet often, on a routine basis, the quantification /evaluation techniques are not always available for our customers. In addition, quality control plans are not always well developed depending on the company size and means. The results of this situation is that the majority of feed companies apply a reduced quality control pressure on nutritional value of raw material they used. As a consequence, often diets formulated are quietly more variable than formely expected in term of nutritional composition.

Here is one of the key points on which TECHNA has worked for several years to help its customers on a day to day basis to optimise feed formulation and animal performances in order to achieve their objectives.

### **NIRS methodology**

The technique Near Infrared Reflectance Spectroscopy was fist developed as a method of measuring water content in grains (NORRIS, 1964). This technique uses a principle which has been recognized for over 200 years. Bonds between organic molecules absorb a specific wavelength range of light in the near infrared region, and the near – infrared colour of the sample provides information about its composition. The functioning of NIRS and the main principles have been explained by different authors (VAN KEMPEN and JACKSON, 1996).

NIRS has proven to be a valuable tool for quality control linked to its speed analysis and low operating costs and accuracy. It has been used for a wide range of applications in animal nutrition such as proximate chemical composition of poultry feeds (MARTINEZ et al., 2003), apparent metabolisable energy (VALDES and LESSON, 1994 a,b), or total and digestible amino acids (BODIN et al., 1999). In the field, calibrations for detection of potential feed contaminations with meat and bone meals have been also elaborated with success (PEREZ-MARIN et al., 2004). NIRS provides the ability to analyse feed ingredients at a cost much lower than and a speed much faster than conventional laboratory methods.

## **Materials and methods**

### *Calibrations development*

A NIRS 5000 model (Foss Tecator, Sweden) was used in TECHNA lab to scan the different samples (raw materials and complete feed). For this type of instrument, the optical values recorded as  $\log 1/R$  ( $R$ = Reflectance) were taken at 2 nm intervals over the wavelength range 1100 to 2498 nm. Spectral data were correlated with the different proximate analysis using WINISI III version 1.50e (Infrasoft International, Port Mathilda, PA, USA). The partial least squares (PLS) regression was used. The spectral data were primarily subjected to a derivative math treatment 1,4,4,1 or 2,5,5,1 or 2,6,4,1 depending on the parameter studied and a standard normal variates and detrending scatter corrections. These procedures allow us to obtain the optimal information coming from the spectrum and to reduce the particle size effect.

Two cycles of outlier eliminations were allowed, based on the spectral proximity or  $H$  value ( $H$  value larger than 4.00 = elimination) and the student  $T$  value ( $T$  value larger than 2.50 = elimination). The spectral proximity is a statistical value which indicate if an unknown spectra is identical or close to a reference set of spectra. The  $T$  value shows if a sample fits or not with the calibration model. Performance of the PLS calibration were tested by cross-validations experiments. This procedure avoids the need to set aside samples for a validation set. In its original form, the idea was the following : one sample is dropped from the calibration test and an entire calibration is made with the remaining samples which is used to predict the samples left out. The standard error of cross validation ( $SECV$ ) / *mean* and the standard deviation of the reference population (*stdev*)/ $SECV$  ratio were calculated to obtain an estimation of the predictive ability of our NIR calibrations. The difference between true and predicted were calculated to calculate an average  $SECV$ . The number of samples used, the performances of the calibrations for each type of feedstuffs and each parameter are presented in table 1.

### **Reference methods and samples preparation**

All the samples were analysed in TECHNA lab using wet chemistry techniques defined through AFNOR procedures : Moisture (NFV 18-109), Protein (NFV 18-120), Starch (3<sup>rd</sup> EU directive 72.199), Cellulose (NFV 18- 03-040), Ash (NFV18-101), Fat (NFV18-117).

Depending on their nature, samples were ground or not before scanning. In order to grind the samples through a 1mm sieve, we used a Retsch ZM 1000 grinder. For one type of feedstuff, if the accuracy of the calibrations was equivalent, we preferred for practical reasons to keep the calibrations developed on the ungrounded set of samples.

**Table 1** : Summary of NIRS calibrations statistics for proximate analysis in wheat, corn, soybean meal (SB Meal) and soybean SB Seed (% as received)

	samples	mean	stdev	SECV	R <sup>2</sup>	SECV/mean	Stdev/mean
<b>Wheat</b>	<b>393</b>						
Moisture		13.04	1.19	0.21	0.97	0.02	5.67
Protein		11.34	1.55	0.31	0.96	0.03	5.00
Starch		60.34	1.49	0.75	0.75	0.01	1.98
<b>Corn</b>	<b>318</b>						
Moisture		14	0.84	0.27	0.90	0.02	3.11
Protein		8.05	0.73	0.26	0.87	0.03	2.80
Starch		63.03	1.10	0.72	0.57	0.01	1.53
Fat		3.84	0.33	0.22	0.54	0.06	1.50
<b>SB Meal</b>	<b>404</b>						
Moisture		12.23	0.72	0.27	0.86	0.02	2.67
Protein		45.5	1.03	0.46	0.80	0.01	2.24
Fat		1.87	0.46	0.14	0.90	0.07	3.29
Cellulose		5.45	1.07	0.33	0.91	0.06	3.24
Ash		6.05	0.33	0.15	0.79	0.02	2.20
<b>SB Seed</b>	<b>120</b>						
Moisture		10.97	1.50	0.19	0.98	0.02	7.89
Protein		34.69	1.18	0.40	0.89	0.01	2.95
Fat		19.59	1.03	0.25	0.94	0.01	4.12

### Results and discussions

For the illustration of our purpose, calibrations performances for wheat, soybean meals (SBMeals), corn, wheat and soybean seeds are presented in table 1. TECHNA lab has developed NIR calibrations to predict proximate analysis on a larger range of feedstuffs like pea, alfalfa, rapeseed meals, sunflower meals, cereals bran...

The mean, standard deviation of the different chemical composition parameters, resulting from the analysis of the samples and the statistical results of NIR spectroscopy calibration are presented in Table 1.

Table 1 shows that NIR calibrations explained between 80 and 98% of the variation for the overall proximate criteria, excepted for starch and fat in our corn data base. For these two parameters, the R<sup>2</sup> obtained explained only 60% of the variation. Several hypothesis can therefore be brought to the fore. First, the actual structure of our calibration set is probably limited in term of variation for these two parameters, which means that new samples have to be selected to expand our NIRS and reference data base. On the other hand, if we focused on starch, this parameter is often considered to be quiet variable in term of accuracy (moving from 3 to 5%) depending on the cereals.

If we compare on an overall point of view, the SECV / mean ratio, the majority of the values obtained for each raw material are comprised between 1 and 3 % excepted for starch in corn, fat and cellulose in soybean meal. Accuracy of the reference methodology and structure of the

calibration set might be the main explanation. On the other hand if we use the ratio Stdev/SECV as an indicator, the main part of the values are comprise between 2.5 and 5 which are considered adequate for quality assurance (EDNEY et al., 1994).

By the way, even if a wide range of nutritional parameters have not been yet calibrated using NIRS, the TECHNA calibrations can be one additional tool to rapidly evaluate global nutrient value of feedstuffs of its customers. On an applied point of view, it is interesting to simulate different simple cases to demonstrate how NIRS can impact on some feed formulation habits.

If we go back to the soybean meal population represented on figure 1 which varies from batch to batch, there are 8 times when protein content fell below 45% of crude protein. Under practical conditions, very often the wet chemistry analysis have been obtain after use in the complete feed. If no safety margin is used, lower protein composition of soybean meal than expected can conduct to protein deficiency in the final feed with possible animal performance troubles which are not always easy to quantify. In this case, because NIRS allows analysis directly at reception in a very short period of time, the value of NIRS using could have been the possible discount on the different batches times the total amount of soybean meal purchased.

On the other hand, soybean meal batches may sometimes contain more than the specified amount of nutrients. Thus, the value of NIRS using could be the average reduction in feed formulation costs times the total amount of soybean meal purchased per year time the probability that the product will be of higher value.

Table 2 illustrated the final errors obtained for each of three cases in front of 33 soybean meals crude proteins analysis which represented roughly six months of control for one feed-mill. This population is characterized by a mean (45.3%) and a standard deviation (0.52).

Case 1 : where a margin of safety is used on the table value for all the period of time (Table value = mean-0.5xStandard Deviation of the set)

Case 2 : where the table value is reviewed time to time depending on the wet chemistry analysis availability

Case 3 : where NIRS analysis are used each 3 batches following delivery time to review the table value.

The total error of such type of estimation has two components : the Bias and the Standard Deviation of the error. The total error is equal to  $(\text{Bias}^2 + \text{Standard Deviation of the error}^2)^{0.5}$ .

In Table 2, it appears that using a margin of safety in feed formulation (Case 1) conducts to a continuous underestimation (negative bias value -0.2697). For Case 2 and 3, the bias are nearly equal to zero, the difference is observed on the standard deviation which is around 35% more when the table value is reviewed time to time depending on the wet chemistry analysis availability. Information given by NIRS does not reduce the variance of nutrient composition from batch to batch, but due to its speed ability analysis and costs, it reduces the error of estimating the nutrient value of a particular batch.

**Table 2** : Example of errors associated to 3 cases of table value management on soybean meal Crude Protein values

Case 1			Case 2			Case 3		
CP value anl	Table value	Delta	CP value anl	Table value	Delta	CP value anl	Table value	Delta
45.10	45.00	-0.10	45.10	45.50	-0.40	45.10	45.30	-0.20
45.70	45.00	-0.70	45.70	45.50	0.20	45.70	45.30	0.40
45.10	45.00	-0.10	45.10	45.50	-0.40	45.10	45.30	-0.20
44.60	45.00	0.40	44.60	45.50	-0.90	44.60	44.87	-0.27
44.80	45.00	0.20	44.80	45.50	-0.70	44.80	44.87	-0.07
45.20	45.00	-0.20	45.20	45.50	-0.30	45.20	44.87	0.33
46.00	45.00	-1.00	46.00	45.50	0.50	46.00	45.90	0.10
45.60	45.00	-0.60	45.60	45.50	0.10	45.60	45.90	-0.30
46.10	45.00	-1.10	46.10	45.50	0.60	46.10	45.90	0.20
44.80	45.00	0.20	44.80	45.50	-0.70	44.80	44.87	-0.07
44.80	45.00	0.20	44.80	45.50	-0.70	44.80	44.87	-0.07
45.00	45.00	0.00	45.00	45.50	-0.50	45.00	44.87	0.13
45.60	45.00	-0.60	45.60	<b>45.20</b>	0.40	45.60	45.23	0.37
45.10	45.00	-0.10	45.10	45.20	-0.10	45.10	45.23	-0.13
45.00	45.00	0.00	45.00	45.20	-0.20	45.00	45.23	-0.23
44.60	45.00	0.40	44.60	45.20	-0.60	44.60	45.00	-0.40
45.50	45.00	-0.50	45.50	45.20	0.30	45.50	45.00	0.50
44.90	45.00	0.10	44.90	<b>45.00</b>	-0.10	44.90	45.00	-0.10
45.60	45.00	-0.60	45.60	45.00	0.60	45.60	45.47	0.13
45.30	45.00	-0.30	45.30	45.00	0.30	45.30	45.47	-0.17
45.50	45.00	-0.50	45.50	45.00	0.50	45.50	45.47	0.03
45.40	45.00	-0.40	45.40	45.00	0.40	45.40	45.53	-0.13
45.20	45.00	-0.20	45.20	45.00	0.20	45.20	45.53	-0.33
46.00	45.00	-1.00	46.00	<b>45.20</b>	0.80	46.00	45.53	0.47
44.00	45.00	1.00	44.00	45.20	-1.20	44.00	45.03	-1.03
46.40	45.00	-1.40	46.40	45.20	1.20	46.40	45.03	1.37
44.70	45.00	0.30	44.70	45.20	-0.50	44.70	45.03	-0.33
45.10	45.00	-0.10	45.10	45.20	-0.10	45.10	45.57	-0.47
45.80	45.00	-0.80	45.80	45.20	0.60	45.80	45.57	0.23
45.80	45.00	-0.80	45.80	45.20	0.60	45.80	45.57	0.23
44.70	45.00	0.30	44.70	45.20	-0.50	44.70	45.20	-0.50
45.40	45.00	-0.40	45.40	45.20	0.20	45.40	45.20	0.20
45.50	45.00	-0.50	45.50	45.20	0.30	45.50	45.20	0.30
<b>Bias</b>		<b>-0.2697</b>	<b>Bias</b>		<b>-0.0030</b>	<b>Bias</b>		<b>-0.0001</b>
<b>Standard Deviation</b>		<b>0.52</b>	<b>Standard Deviation</b>		<b>0.56</b>	<b>Standard Deviation</b>		<b>0.41</b>
<b>Total Error</b>		<b>0.58</b>	<b>Total Error</b>		<b>0.56</b>	<b>Total Error</b>		<b>0.41</b>

The more or less important margin of safety used in feed formulation often depends on the confidence the formulator gets in the available data and analysis in order to adapt the table values. As an example, if the population average of 33 soybean meals is used to fix the table value for crude protein, the number of samples used to calculate the mean will impact the maximum relative error defined by the Student statistics.

For a 5% probability level, the maximum relative error of the mean is calculated as:

$$d_r (\% \text{ of the mean}) = (1.96 \times \text{coefficient of variation}) / (\text{number of samples})^{0.5}$$

As an example, table 3 illustrates the evolution of this maximum relative error in function of the samples number used to calculate the table value for crude protein inside the population of 33 soybean meals.

**Table 3** : Evolution of the maximum relative error of the mean in function of the number of samples used

<i>Number of samples used to calculate the mean</i>	<i>d<sub>r</sub> = the maximum relative error of the mean</i>
N = 5	1.00 %

N = 11	0.67 %
N = 33	0.39 %

Using a typical broiler formula (growing period 30 to 42 days of age, ME = 3050 kcal/kg, CP = 18.5% and Digestible Lysine = 1.00% as received) and a soybean meal price equal to 206 €/T, it is possible to do a rough simulation of the economical impact for the three different cases of table value management on soybean meal we previously described.

Between Case 1 and Case 2, the gain could be estimated around 0.32€/T of feed.

Between Case 1 and Case 3, the gain could be estimated around 0.37€/T of feed.

The difference between Case 2 and Case 3 should appear small (0.05€/T) but if this delta is applied to the overall parameters predictable by NIRS and to the overall ingredients in a broiler feed range, a 2€/T of feed could be assumed. For an organization dealing with 350 000 broilers per week with an estimated consumption of 3.5 kg per broiler, the possible gain would be 125 K€ for one year.

### Conclusions

It has been demonstrated that NIR calibrations can be used to predict proximate composition of a wide range of feedstuffs. This type of application illustrates the potentials of NIRS as a rapid control tool. This technique is non destructive, non-time consuming and can be used at the point of ingredient delivery. The example used to demonstrate the possible application in formulation process and table value management, only based on the protein content of soybean meal is simple but relevant. Today, a wide range of parameters predictable through NIRS are available on the market, such as ME (Metabolisable Energy) or digestible amino acids in feed ingredients. Thus, it is quiet easy to evaluate the ability of such technique which would allow the feed producers and integrators to reduce costs and to improve flock performance through a more relevant quality control and a more reactive adjustment of the feed formulation.

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