

# Can Near-Infrared Spectroscopy (NIRS) Quantify The Quality of Fishmeal Circulating

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# Can Near Infrared Spectroscopy (NIRS) Quantify The Quality of Fishmeal Circulating in Jember, Indonesia?

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**Abstract.** The objectives of this study were to determine the range of nutrient content of fishmeal circulating in Jember Regency, East Java, and to make a calibration standard for quick calculation using NIRS by  $R^2$  and RMSECV. This study used 11 fishmeal samples from around Puger, Jember and around Muncar, Banyuwangi. The physical examination included organoleptic tests (color, odor, and texture), density, and chemical analysis of fishmeal samples. The data obtained were analyzed descriptively. The results indicated that the nutrient content of fishmeal had greatly variation. The CP content presented a quite high ranged between 26.61% to 64.30%. The result of organoleptic test showed the color was yellowish brown, brown, slightly brown to dark brown. The odor was generally standard, which was fishy. Some fishmeal samples were moist in texture, such as fishmeal samples from Jember A (22.03%) and Muncar Banyuwangi B (19.65%). The average of fishmeal density was 0.528 g/ml that ranged between 0.341-0.726 g/ml. The measurement of  $R^2$  and RMSECV calibration of fishmeal using NIRS showed good results to predict CF, DM, and EE contents. The usage of NIRS technology was recommended for proximate analysis unless ash content due to the absence of spectrum absorption for minerals.

## 1. Introduction

Fishmeal necessity is always increase along with livestock industry development. This is due to fishmeal was a one of protein source in the feed manufacturer, especially for poultry and fish diets. Fishmeal in Indonesia is distinguished between import and local. Imported fishmeal is considered better because contained more than 60% protein and low fat. Contrary, local fishmeal had only 55-58% protein and included in grade C [1]. The fact is fishmeal circulating in East Java, specifically in Jember Regency had lower protein content and very diverse. It is caused by the composition of fishmeal consisted of fishery waste. Variations in the nutrient content of fishmeal is quite high so that it required an analysis of nutrient content before the rations formulated.

Alternative methods are required to deal on these problems. The solution is to find the nutritional value of feed ingredients, especially fishmeal, quickly, cheaply, easily and accurate. One such technique is the measurement using near infrared (NIR) light reflectance emitted into the material. Near Infrared Reflectance Spectroscopy (NIRS) is a non-destructive technology that analyze at high speed, simple, without pollution and chemicals, and more efficient at cost. NIRS technology requires a



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calibration standard of fishmeal circulating in Jember before NIRS operated. The objectives of this study were to determine the range of nutrient content of fishmeal circulating in Jember Regency, East Java, and making a calibration standard that can be used for quickly calculation using NIRS by  $R^2$  (coefficient terminated) and RMSECV (*root mean square error of cross validation*).

## 2. Methodology

This study was carried out at the Feed Technology Laboratory, Department of Animal Husbandry, Politeknik Negeri Jember (State Polytechnic of Jember). The fishmeal samples were taken from the fishmeal producer centres circulated in Jember, originating from around Puger, Jember and around Muncar, Banyuwangi, as well as from poultry shops and feed mills around Jember and Banyuwangi.

The instrument usage in this study was beaker glass 500 ml, measuring cylinder 100 ml, and digital scales for density measurement; proximate analysis instruments; Fourir Transform Infrared (FT NIR) Tango from the Bruker and Opus Software version 7.8 for calibration standard determining. The materials were 11 fishmeal samples circulating in Jember Regency and chemicals for proximate analysis including dry matter (DM), crude protein (CP), ether extract (EE), crude fiber (CF) and ash content based on Official Methods of Analyses [2].

Physical analysis included organoleptic tests (color, odor, and texture) and density determination without compacting. The density was calculated by the ratio of sample weight and sample volume on g/ml. Chemical analysis of fishmeal samples was intended to compare the data from chemical analysis [2] with the FT-NIRS method. The data of chemical analysis were used for calibration stage due to the chemical analysis had been carried out at the beginning before the NIRS analysis.

The procedures of FT-NIRS analysis consisted of FT-NIRS spectra collection that followed by calibration measurement. Spectral collection of fishmeal samples was carried out using approximately 25 g of samples. The sample was placed on a petri dish and arranged as evenly as possible then placed on the rotating sample. Samples were irradiated using infrared short wavelengths ( $12500-4000\text{ cm}^{-1}$ ) at temperatures of 16-20 °C. Irradiation was done in triplicate to reduce bias. The spectra value of samples was calculated in  $R^2$  where  $R^2$  was the reflectance value. Fishmeal samples were used to estimate modelling using OPUS 7.8 software integrated with NIRS. Modelling was determined using partial least square (PLS) regression analysis to correlate spectra results and laboratory data from chemical analysis.

The NIRS analysis was used to measure calibration. Calibration was done by NIRS modelling determination. The statistical values were performed by  $R^2$  (coefficient determination), RMSECV (root mean square error of cross validation), and RPD (residual prediction deviation or standard deviation ratio) [3].

$$R^2 = \frac{n \sum XY - \sum X \sum Y}{\sqrt{[n \sum X^2 - (\sum X)^2][n \sum Y^2 - (\sum Y)^2]}}$$

Note:

X = nutrient composition using proximate analysis

Y = estimated value using FT NIRS

n = number of samples

The coefficient determination ( $R^2$ ) showed the model ability to explain the diversity of dependent variable values. The greater value of  $R^2$  presented more able to explain the characteristics of dependent variables. The analysis result could be stated as good when  $R^2 > 80-95\%$  [4]. Root mean square error of prediction (RMSEP) was realized in the curve. Prediction / True curves covered the entire surface of the line and did not widen. The data obtained in this study were analyzed descriptively.

### 3. Results and discussion

The nutrient content of fishmeal circulating in Jember Regency was presented in Table 1. The results of observations indicated that the nutrient content of fishmeal had greatly variation. This result was not in accordance with the nutrient content standard of fishmeal that consisted of DM 86-100%, ash content 20.7-24.1%; CP 52.6-61.2%; EE 6.8-7.9%; CF 2.2-2.6% [5]. There was a more than 14% water content of fishmeal samples. It showed that the fishmeal was not durable to be stored, and become more easily damaged. The CP content presented a quite high ranged between 26.61% to 64.30% (Table 1). It was less suitable to be used as a protein source for feed which should be around 52.6-61.2%. The higher values of ash and CF content than the standard showed that there was a mixture of fishmeal with other ingredients, such as physical appearance of scales, spines, and some fish bones (Table 2). The quite variation of fishmeal quality, specifically the nutrient content, it was necessary to analyze first the nutrient content before formulating the rations. One of the analyses that could be carried out quickly was by using NIRS that it required a calibration standard before operating (Table 3).

Table 1. Nutrient content of fishmeal circulating in Jember Regency\*

| No. | Samples                    | Water | DM    | Ash   | CP    | EE   | CF    |
|-----|----------------------------|-------|-------|-------|-------|------|-------|
|     |                            | %     |       |       |       |      |       |
| 1   | Puger Jember (PJ A)        | 7.80  | 92.20 | 26.61 | 61.16 | 9.16 | 1.14  |
| 2   | Puger Jember (PJ B)        | 4.92  | 95.08 | 39.02 | 26.57 | 7.06 | 2.57  |
| 3   | Jember A (J A)             | 22.03 | 77.97 | 30.42 | 47.37 | 1.22 | 5.41  |
| 4   | Jember B (J B)             | 11.80 | 88.20 | 31.55 | 49.88 | 2.66 | 1.21  |
| 5   | Jember C (J C)             | 6.46  | 93.54 | 23.97 | 66.77 | 3.58 | 0.94  |
| 6   | Banyuwangi (B)             | 4.79  | 95.21 | 29.60 | 60.58 | 7.09 | 0.28  |
| 7   | Muncar Banyuwangi A (MB A) | 15.01 | 84.99 | 22.00 | 51.47 | 5.43 | 3.36  |
| 8   | Muncar Banyuwangi B (MB B) | 19.65 | 80.35 | 23.10 | 50.23 | 5.11 | 11.68 |
| 9   | Muncar Banyuwangi C (MB C) | 14.43 | 85.57 | 20.70 | 60.84 | 4.64 | 1.61  |
| 10  | Muncar Banyuwangi D (MB D) | 15.06 | 84.94 | 20.05 | 64.30 | 5.33 | 3.69  |
| 11  | Muncar Banyuwangi E (MB E) | 17.25 | 82.75 | 21.59 | 46.68 | 5.49 | 7.14  |
|     | Average                    | 12.66 | 87.34 | 26.24 | 53.26 | 5.16 | 3.55  |

DM=dry matter; CP=crude protein; EE= ether extract; CF=crude fiber

\*The result of proximate analysis from Feed Technology Laboratory, Politeknik Negeri Jember

The results of the organoleptic test and the density of fishmeal circulating in Jember Regency were presented in Table 2. It showed the color was yellowish brown, brown, slightly brown to dark brown. The color differences were possible from the fish types used and the mixture materials. Generally, the brown color of fishmeal originated from Maillard reaction due to the heating process that occurs during fishmeal manufacturer. This reaction result was known that may reduce the bioavailability of essential amino acids of fishmeal, such as lysine [6].

The odor of fishmeal was generally standard, which was fishy. Some fishmeal samples were moist in texture, such as fishmeal sample from Jember A and Muncar Banyuwangi B which appeared after proximate analysis had high water content (> 14%) around 22.03% and 19.65%, respectively. Whereas, there was a fishmeal which had a rather moist texture. It showed a water content slightly above 14%, such as fishmeal coming from Muncar Banyuwangi A (water content = 15.01%) and Muncar Banyuwangi E (water content = 17.25%).



Tabel 2. Organoleptic results and the density of fishmeal circulating in Jember Regency.

|    | Samples                    | Organoleptic    |            |   | Density<br>g/ml |
|----|----------------------------|-----------------|------------|---|-----------------|
|    |                            | Color           | Odor       | Texture   |                 |
| 1  | Puger Jember (PJ A)        | brown           | Fishy      | rough, fish meat fibrous, mixture with fish scales      | 0.500           |
| 2  | Puger Jember (PJ B)        | yellowish brown | Fishy      | rough like sand   | 0.726           |
| 3  | Jember A (J A)             | dark brown      | not fishy  | moist and powdery                                       | 0.506           |
| 4  | Jember B (J B)             | dark brown      | less fishy | powdery and mixture with fish bone and scales           | 0.576           |
| 5  | Jember C (J C)             | brown           | Fishy      | powdery and rough                                       | 0.543           |
| 6  | Banyuwangi (B)             | slightly brown  | Fishy      | inhomogeneous, mixture with fish head, bone, and scales | 0.341           |
| 7  | Muncar Banyuwangi A (MB A) | brown           | Fishy      | rather moist, rough, mixture with fish scales           | 0.454           |
| 8  | Muncar Banyuwangi B (MB B) | dark brown      | Fishy      | moist, powdery, and rough                               | 0.478           |
| 9  | Muncar Banyuwangi C (MB C) | brown           | fishy      | powdery and rough                                       | 0.512           |
| 10 | Muncar Banyuwangi D (MB D) | brown           | fishy      | powdery and rough                                       | 0.632           |
| 11 | Muncar Banyuwangi E (MB E) | brown           | fishy      | powdery   | 0.545           |
|    | Average                    |                 |            |   | 0.528           |

The average of fishmeal density circulating in Jember was 0.528 g/ml that it was in a range between 0.341-0.726 g/ml. The difference of fishmeal density may possible because the differences in the constituent materials. Fishmeal from Puger, Jember (PJ B) showed the highest density (0.726 g/ml). It was possible for the mixtures had a higher density which appears from its non-abrasive texture as generally the texture of fish meat, but such as sand. This condition was supported by the lowest CP content, which was 26.57%. Fishmeal from Banyuwangi (B) has the lowest density because this mixture of fishmeal did not homogeneous, so it requires a large volume (voluminous) and will produce a small density. Variations in specific gravity values are influenced by nutrient content of the material, particle size distribution, and particle surface characteristics. Specific gravity affects the homogeneity of particle dispersion and stability of a feed mixture [7].

NIRS technology was developed as a non-destructive method, can analyze at high speed, did not cause pollution, the use of sample preparations that are simple and do not require chemicals. NIR spectroscopy uses electromagnetic waves with wavelengths of 12,800 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> [8]. In this study, the infrared wavelengths used were 9400 cm<sup>-1</sup> to 4400 cm<sup>-1</sup>. The analysis process using NIRS is easy and quickly, cheaper and simple [9]. Quickly analysis of fishmeal using NIR technology was necessary a calibration standard from fishmeal samples that had known the nutrient content by proximate analysis. The equation of calibration was to estimate the nutrient content (DM, CP, EE, CF, and Ash) based on the reflectant data made by establishing a relationship between the nutrient content of the results of the proximate analysis with the reflectant value in the PLS regression process during calibration. The relationship between the alleged value of NIR with the results of laboratory analysis is presented in Figures 1, 2 and 3.

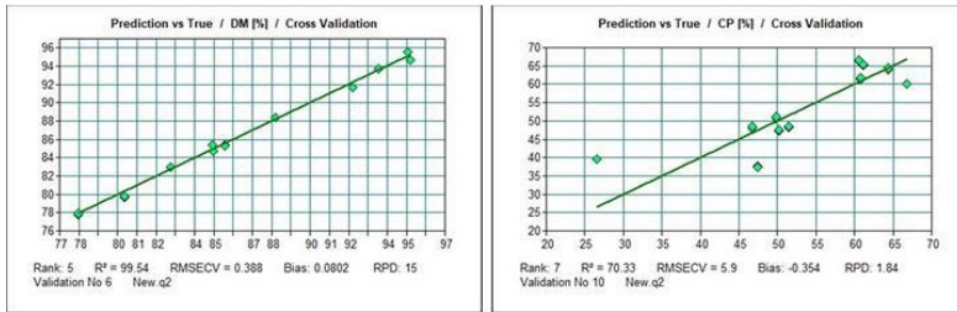


Figure 1. The ratio of DM (a) dan CP (b) estimated by NIR and proximate analysis

The results obtained show the laboratory value and the closest prediction value was the CF parameter with a value of  $R^2 = 99.73$  (Figure 2b), followed by DM parameters (Figure 1a), EE (Figure 2a), and the lowest CP was 70.33. The greater the value of  $R^2$ , the more the model was able to explain the characteristic of dependent variables. If  $R^2 > 80-95\%$ , the analysis can be stated as good [4].

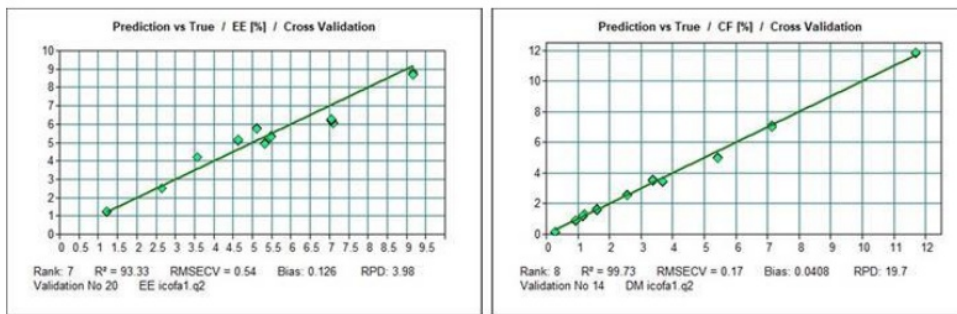


Figure 2. The ratio of EE (a) dan CF (b) estimated by NIR and proximate analysis

In this study, the calibration of  $R^2$  showed a good result to be used in predictions, especially for the CF, DM, and EE content of fishmeal circulating in Jember. The calibration of  $R^2$  value of CP was quite low (<80-95%). The low value can be caused by the non-uniform sample particle size and the presence of a mixture of other ingredients in the fishmeal which was quite varied.

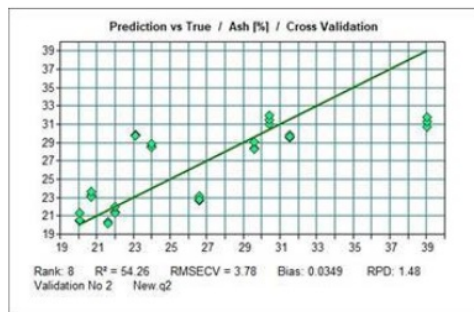


Figure 3. The ratio of ash content estimated by NIR and proximate analysis

Ash was an inorganic component in plants. The lowest  $R^2$  value was 54.26. The low prediction value for ash was due to the absence of absorption spectrum in NIRS for minerals. The infrared in NIRS vibrates the functional group which was an organic component in the sample, while the mineral was an inorganic component, so that NIRS was less able to predict the value of ash content accurately [10]. This condition was also evident from the results of RMSECV for ash content which was quite high at 3.78 (Figure 3) and CP at 5.9 (Figure 1b). RMSECV was good when the value is getting smaller. It indicated the measurement accuracy from the samples data to the calibration.

#### 4. Conclusions and recommendations

##### Conclusions

The physical and chemical quality of fishmeal circulating in Jember was quite varied so that NIRS technology was required to analyze the nutrient content of fishmeal as feed with highly speed. The measurement of  $R^2$  and RMSECV calibration of fishmeal circulating in Jember using NIRS showed good results to predict CF, DM, and EE contents. However, the lower CP value was caused by the non-uniform particle size and the presence of other ingredients in the fishmeal with a sufficiently varied amount.

##### Recommendations

The usage of NIRS technology was recommended for proximate analysis unless ash content due to the absence of spectrum absorption for minerals. The infrared on NIRS vibrates the functional group that was an organic component in the sample.

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