近赤外分光分析法による日本国産大豆の粗蛋白,粗脂肪,水分含量の定量

誌名	食品総合研究所研究報告 = Report of National Food Research Institute
ISSN	03019780
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巻/号	51号
掲載ページ	p. 11-14
発行年月	1987年11月

食総研報(Rep. Natl. Food Res. Inst) No.51, 11～14(1987)

Near-Infrared Reflectance Spectroscopic Analysis of Protein, Oil and Moisture in Japanese Soybeans

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A near-infrared reflectance spectroscopic method (NIRS) was evaluated for the determination of protein, oil and moisture contents in Japanese domestic soybeans.

A multiple linear regression analysis with the data obtained by standard-laboratory methods (protein by Kjeldahl method, oil by ethyl ether extraction and moisture by drying method at 130°C for 2hr) and the NIRS method was carried out to make a calibration. The accuracy of the NIRS method was found to be adequate when the standard-laboratory values for a set of samples that were not included in the calibration, were compared. It is concluded that the NIRS method is suitable for the determination of the above components.
(Received Apr.10, 1987)

Introduction

Near infrared reflectance spectroscopic (NIRS) analysis of protein, oil and moisture contents in cereals and food products has now received keen attention because of its rapidity and convenience. More recently, practical applications of NIRS analysis on major components of chemically complex products such as food, forage etc. have become possible by remarkable advances in the computer technology.

The use of NIRS analysis for determining protein and oil in soybean seeds were reported by Hymowitz *et al.*[1] and Rinne *et al.*[2] with saitisfactory results.

This paper describes the feasibility of NIRS method for determining protein, oil, moisture, total sugar and ash contents in Japanese domestic soybeans which shows a wide range in chemical compositions as compared with U.S. ones.

Materials and Methods

Materials and instruments: Instruments for near infrared spectroscopic analysis were the Technicon InfraAlyzer Model 400 and the Neotec Research Composition Analyzer Model 6350.

Soybean samples were collected from major producing place in Japan. Forty five samples croped in 1980 were used for the experiment using the Technicon instrument. Fifty seven samples croped in 1981 and 84 samples croped in 1982 were used in the experiment using the Neotec instrument.

Standard-laboratory analysis: Moisture was determined by oven drying at 130°C for 2 hr. Protein was done by micro-Kjeldahl method with a Digital Kjeldahl Analyzer (Mitsubishi Chemical Co., Ltd. KN-01). Oil was by Soxhlet extraction with ethyl ether. Total sugar was by Somogyi colormetric method after hydrolysis in 0.7 N HCl.

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Ash was determined by incineration method.

NIRS analysis: The samples were divided into 2 sets. One set were for carrying out a calibration, and the other (prediction set) were for confirmation of accuracy of the calibration established.

Calibration for moisture, protein, oil, total sugar and ash were carried out by stepwise multiple linear regression analysis with the data between a standard-laboratory analysis and NIRS analysis as mentioned in a previous paper[3].

Results and Discussion

Results with InfraAlyzer 400 (Technicon Instrument Co., Ltd.): Results of multiple linear regression analysis with the data between a standard-laboratory analysis and NIRS analysis on moisture, protein, oil, total sugar and ash of calibration set (30 samples) and wavelengths used in the regression are shown in Table I. The multiple correlation coefficient (R) between both the data for moisture, protein and oil with the regression obtained were 0.995, 0.987 and 0.958, respectively.

The standard error of estimate (SEE) for those components were 0.104% 0.336% and 0.338%, respectively.

The values of SEE for those component were satisfactory small for routine application. However, ones for total sugar and ash were not enough good to be accepted.

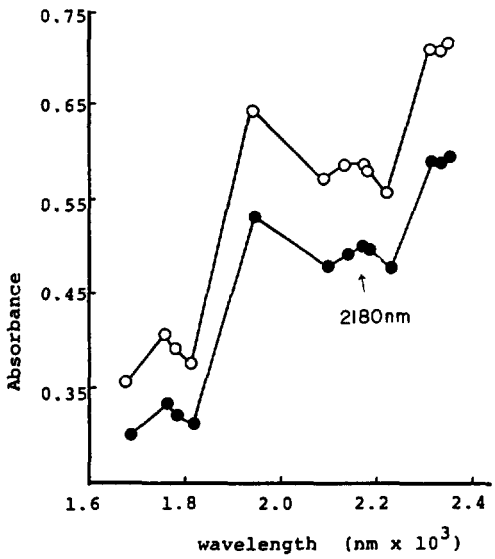

Fig. 1. Comparison of absorbances of near infrared refletance of two soybean samples which have different content of protein. Curve (○——○) has 29.8% of protein. Curve (●——●) has 37.4% of protein.

Figure 1 shows absorbance which is defined by log (1/R), where R is reflectance at various wavelengths for two samples which had different content of protein. The absorbance at 2180 nm where is assigned to -CONH- group[5] is clearly

Table 1. Results of calibration established by multiple linear regression analysis between the absorbances of near infrared reflectance by the InfraAlyzer 400 and the data of moisture, protein, oil, total sugar and ash contents by standard-laboratory analysis of 30 soybean samles.

Components	M	SD	R	SEE	Used wavelengths (nm)
moisture	11.32(%)	0.985	0.995	0.104(%)	1778, 1940, 2139, 2180, 2230, 2348
protein	35.13	1.955	0.987	0.336	1680, 1940, 2139, 2180, 2310, 2348
oil	16.56	1.101	0.958	0.338	1680, 1759, 1940, 2100, 2180, 2310, 2348
total sugar	18.66	1.313	0.874	0.634	2100, 2180, 2230, 2310, 2348
ash	4.83	0.268	0.864	0.139	1680, 1759, 1778, 2100, 2139, 2180, 2230, 2348

M ; mean of chemical values
SD ; standard deviation of mean
R ; multiple correlation coefficient
SEE ; standard error of estimate

different between two samples. It was confirmed that absorption at 2180 nm was employed in the calibration for protein, as shown in Table 1.

Using prediction set (15 samples), accuracy of the calibration established was shown in Table 2. The standard error of prediction (SEP) on moisture, protein and oil were 0.142% 0.497% and 0.354%, respectively.

This accuracy indicates that the InfraAlyzer 400 can be used satisfactorily for the determination of the above components in Japanese domestic soybeans.

Table 2. Accuracy of NIRS analysis using the InfraAlyzer 400 as compared with a standard-laboratory analysis of moisture, protein and oil contents of prediction set.

Components	R	SEP
moisture	0.983	0.142 (%)
protein	0.987	0.497
oil	0.968	0.354

R ; correlation coefficient between a standard-laboratory values and NIRS values
SEP ; standard error of prediction

Results with Research Composition Analyzer 6350 (Neotec Co.,Ltd.): Results of multiple linear regression analysis with the data between a standard-laboratory analysis and NIRS analysis on moisture and protein of calibration set (39 samples croped in 1981 and 53 samples croped in 1982) are shown in Table 3.

The multiple correlation coefficient (R) between both the data for moisture were 0.990 for 1981's crop and 0.981 for 1982's one, and the R for protein were 0.943 for 1981's crop and 0.932 for 1982's one.

The standard error of estimation (SEE) for moisture were 0.152% for 1981's crop and 0.175% for 1982's one, and the SEE for protein were 0.705% for 1981's crop and 0.763% for 1982's one.

The absorption at 2208 nm selected for determination of protein is associated with a combination of the fundamental valence and deformation vibration of N-H[4], and absorption at 1984 and 2162 nm are assigned to -CONH- and -$CONH_2$ group[5),6)].

Using prediction set (18 samples croped in 1981 and 31 samples croped in 1982), accuracy of the calibration established was shown in Table 4.

Table 4. Accuracy of NIRS analysis using the Research Composition Analyzer 6350 as compared with a standard-laboratory analysis of moisture and protein contents of prediction set.

	1981's crop		1982's crop	
Components	R	SEP	R	SEP
moisture	0.985	0.143 (%)	0.981	0.161 (%)
protein	0.950	0.674	0.953	0.741

R ; correlation coefficient between a standard-laboratory values and NIRS values
SEP ; standard error of prediction

Table 3. Results of calibration eatablished by multiple linear regression analysis between the absorbances of the second derivative spectra of near infrared reflectance by the Research Composition Analyzer 6350 and the data of moisture and protein contents by a standard-laboratory analysis of calibration set (39 samples croped in 1981 and 53 samples croped in 1982)

	1981's crop			1982's crop		
Components	R	SEE	Used wavelengths (nm)	R	SEE	Used wavelengths (nm)
moisture	0.990	0.157 (%)	1376, 1646, 1836, 1368	0.981	0.175 (%)	1348, 1370, 1408, 1774
protein	0.943	0.705	2208, 1222, 2162, 1984	0.932	0.763	2208, 1692, 1808, 2206

R ; multiple correlation coefficient
SEE ; standard error of estimate

The standard error of prediction (SEP) for moisture were 0.143% for 1981's crop and 0.161% for 1982's one. The SEP for protein were 0.674% for 1981's crop and 0.741% for 1982's one, which were accurate as compared with the result of Rinne *et al.*[2)]

The results of NIRS analysis on moisture and protein of soybeans croped in 1981 and 1982 indicate that NIRS method has possibility of a rapid determination for those components of Japanese domestic soybeans with an acceptable accuracy.

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近赤外分光分析法による日本国産大豆の粗蛋白，粗脂肪，水分含量の定量

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近赤外分光分析法(以下近赤外法)を用いて，日本全国で栽培，収穫された大豆の粗蛋白，粗脂肪，水分含量を測定した。既存の分析法(粗蛋白；ケルダール法，粗脂肪；エチルエーテル抽出法，水分；130°C，2時間乾燥法)と近赤外法による測定値間の重回帰分析を行ない各成分の検量線を求めた。また，検量線作成に用いなかった試料につき各成分値に対する近赤外法の測定精度を調べた結果，近赤外法は既存の分析法に代わり，国産大豆の上記成分の測定に十分使用しうることが確認された。