Use of near infrared spectroscopy in quality control of Korean brewed soy sauce

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Introduction

Korean Brewed Soy Sauce (Kanjang) is a liquid obtained by the process wherein molds (Aspergillus oryzae and/or Aspergillus sojae), bacteria (Bacillus subtilis) and yeast are cultured in soybean, salt water is added and the mixture is fermented and aged for no less than 90 days while being exposed to sunlight (Figure 1).^{1,2} The microorganisms, the concentration of the salt solution, fermentation time and temperature, etc, influence the quality of Kanjang. Total nitrogen and soluble solids contents are the quality factors of Kanjang (Table 1).^{3,4} Traditionally, Kanjang is fermented in a big jar and each jar produces different quality of Kanjang (Photograph 1). It is very difficult to measure the quality of Kanjang fermented in thousands of jars by analytical methods such as the Kjeldahl method for total nitrogen analysis. A rapid and accurate quantitative method is necessary for the quality control of Kanjang. A near infrared (NIR) spectroscopic method offers many advantages and may be an ideal method for quality control of large numbers of samples. In Japan, the NIR method has been accepted as the Japan Agriculture Standard method for screening the quality of soy sauce.⁵ Thus, the NIR technique was developed to evaluate the total nitrogen and brix of Korean brewed soy sauce.

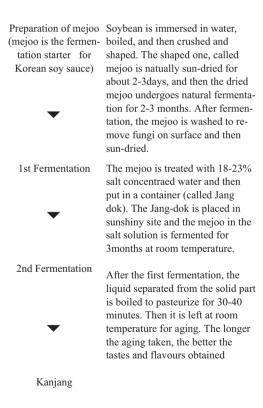


Figure 1. The manufacturing process of Korean brewed soy sauce.

Quality Factors	Contents		
1. Sensory properties	It shall have normal colour and flavor characteristic of the product, and shall not have off-taste and off-flavour.		
2. pH	4.0 - 6.8		
3. Total nitrogen content (%)	not less than 0.7		
4. Soluble solids content (%) (related with brix)	not less than 6.0		
5. Tar Food Colour	not permitted		
 6. Preservatives (gL⁻¹) Benzoic acid, Sodium benzoate Butyl p-hydroxybenzoate Ethyl p-hydroxybenzoate Propyl p-hydroxybenzoate Isopropyl p-hydroxybenzoate Isobutyl p-hydroxybenzoate 	Shall not be detected except following additives. not more than 0.6 (as benzoic acid) not more than 0.25 (as p-hydroxybenzoate) not more than 0.25 (as p-hydroxybenzoate)		

Table 1. Standards for Korean brewed soy sauce.

Materials and methods

Samples

A total of 511 homemade and commercial samples of soy sauce were collected. The 511 samples were divided into a calibration set (291 samples) and a validation set (220 samples).

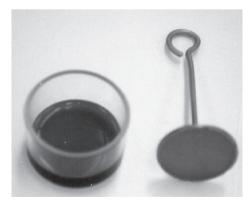
Chemical analysis, total nitrogen content and brix of soy sauce were determined by the Kjeldahl method and refractometer.

NIR reflectance measurements

Spectra of soy sauce were recorded as $\log 1/R$ at 2 nm intervals from 400 to 2500 nm using a Foss NIRSystems Model 6500 monochromator and quartz sample cup with gold surface immersible diffuser (Photograph 2).



Photograph 1. Big jars for ripening of Korean brewed soy sauce.



Photograph 2. Quartz sample cup and gold surface immersible diffuser.

Regression analysis

Infrasoft Internation (ISI) software was used to derive and validate NIR calibration for total nitrogen and brix of soy sauce using partial least squares (PLS) regression. The best equation was selected based on the lowest standard error of prediction (*SEP*).

Results and discussion

Korean brewed soy sauce is composed of about 66% of water, 25% of salt, 5% of proteins, 3% of sugars and 1% of minerals. The absorptions, at about 1450 nm and 1940 nm due to wa-

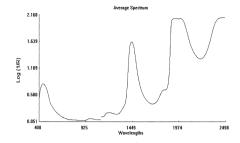


Figure 2. NIR spectrum of Korean brewed soy sauce.

ter, were strong in soy sauce (Figure 2). The results of PLS regression with the data between a chemical analysis and NIR analysis on total nitrogen and brix of calibration and validation set are shown in Table 2. The statistics for the chosen equation for total nitrogen were satisfactory ($R^2 = 0.992$ and SEC = 0.03%). This was utilising 1, 4, 4, 1 math treatment with scatter correction from 400 to 2500 nm. After prediction of the validation set, the *SEP* for total nitrogen was 0.04% with an r^2 of 0.989. Kobayashi *et al.*⁶ obtained 0.009% of *SEC* and 0.01% of *SEP* for total nitrogen determination. The brix equation resulted in an R^2 of 0.997 and *SEC* of 0.31. This was utilising a 1, 4, 4, 1 math treatment and scatter correction. Upon validation, an r^2 of 0.996 and a *SEP* of 0.36 was achieved. These results are considered to be sufficient to be of practical use, such as a screening test for maturity of soy sauce during the fermentation process; however, further experiment is necessary to improve the accuracy.

Table 2. Calibration and validation results for Korean brewed soy sauce.

Component	Calibration			Validation	
	Regression method	R^2	SEC	r^2	SEP
Total nitrogen	PLS terms : 9	0.992	0.03	0.989	0.04
Brix	PLS terms : 7	0.997	0.31	0.996	0.36

SEC: Standard error of calibration *SEP*: Standard error of prediction

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