P-07-02

A validation study of raman spectrometric on-site method to measure solid fat content (#103)

Michiyo Motoyama[1], Shigenobu Hayashi[2], Keiko Jimura[2], Ikuyo Nakajima[1], Genya Watanabe[1], Keisuke Sasaki[1]

[1] Institute of Livestock and Grassland Science, National Agricultural and Food Research Organization (NARO), Tsukuba, Japan; [2] Research Institute for Material and Chemical Measurement, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan

Introduction

It is well known that crystalline states of fat affect decisively on physical properties such as mechanical strength and melting point of fat-based foods. In the meat industry, however, the importance of the crystalline states of fat within meat tissues has not been fully recognized. It is partly because of a lack of on-site technique which can tell us the crystalline states of fat.

In order to clarify the fat crystalline state in meat, a Raman spectrometric technique using a portable device had been developed.[1] At the ICoMST 2014, we reported that a Raman technique successfully predicted (R^2=0.99) the Solid Fat Content (SFC) of fat samples (n=18).[2] The objective of this study was to conduct further validation using a larger number of samples.

Methods

Samples

Fat samples (n=108) including pure lards, modified lards, beef tallows, butters, shortenings, margarines, fat spreads, interesterified fats, hydrogenated fats, fractionated fats, and triacylglycerol reagents were prepared.

Solid Fat Content (SFC) measurement

SFC was measured by a conventional NMR method in accordance with an official method.[3] Measuring temperature was 20°C or the ambient temperature (26°C).

Raman spectroscopic measurement

Raman spectra (range 1850–500 cm^{-1}) of the samples were obtained just before the SFC measurement. A portable Raman spectrometer with 785-nm laser excitation (ProRaman-L-785C, Enwave Optronics, USA) equipped with an optic-fiber probe was used. The optic-fiber probe was introduced to a metal block where samples were maintained at the same measuring temperatures as the SFC measurement. Obtained spectra were baseline corrected and normalized.

Statistical analysis

To evaluate the SFC predictability of Raman spectra, principal components regression (PCR) analysis was carried out using The Unscrambler software (version 9.6, CAMO software) assigning the spectral data as independent variables and the SFC data as dependent variables. Full cross validation was performed to create a reliable PCR model with a significance value of 0.05.

Results

As a result of PCR analysis, three main principal components were detected and explained 89% of the total variance of measured SFC (explained 51, 33 and 6% by each components). The determination coefficient (R^2) of the regression model was 0.88 (Fig. 1).

Similar to the previous report [2], however, a significant deviation between measured and predicted SFC values (>30%) was present particularly for the samples whose SFC were close to 100% (indicated by an ellipse in Fig. 1).

Conclusion

Using more than one hundred fat samples, it was shown that SFC could be accurately predicted by their Raman spectra.

The observed deviation between the measured and predicted SFC values may be derived from the difference in measuring principles between the NMR and Raman methods: The NMR method detects gross amount of hydrogen nuclei in solid phase, while Raman spectra detects molecular structure of fats in solid phase where a few types of crystal polymorph exist. Detected three principal components may correspond to polymorphic types, and the significant deviation in predicted values of high-SFC samples may be brought by their polymorphic differences.

In the previous study [2], SFC can be predicted more accurately (R^2 = 0.99) by the calculation formula (α_c) with only two Raman spectral variables. In order to develop a more accurate Raman technique, we will find reliable variables which are not sensitive to crystal polymorphic types.

Achknowledgements

M. M. thanks Prof. Satoru Ueno at Hiroshima University for a sample and Ms. Yumiko Endo at NARO for her technical assistance. This study was supported in part by Grant-in-Aid for Scientific Research (B) 24380148 and Grant-in-Aid for Young Scientists (B) 25850098 from the Ministry of Education, Culture, Sports, Science and Technology.

Literature

1. Motoyama, M., Chikuni, K., Narita, T., Aikawa, K. & Sasaki, K. (2013). In Situ Raman Spectrometric Analysis of Crystallinity and Crystal Polymorphism of Fat in Porcine Adipose Tissue. Journal of Agricultural and Food Chemistry 61(1): 69-75.
2. Motoyama, M., Hayashi, S., Jimura, K., Chikuni, K., Nakajima, I., Aikawa, K. & Sasaki, K. (2014). Development of Raman spectrometric on-site method to measure solid fat content. Abstracts of ICoMST2014 poster V-33.

Notes

3. American Oil Chemists' Society, AOCS Official Method Cd 16b-9 Revised 1999. Solid Fat Content (SFC) by Low-Resolution Nuclear Magnetic Resonance—The Direct Method.

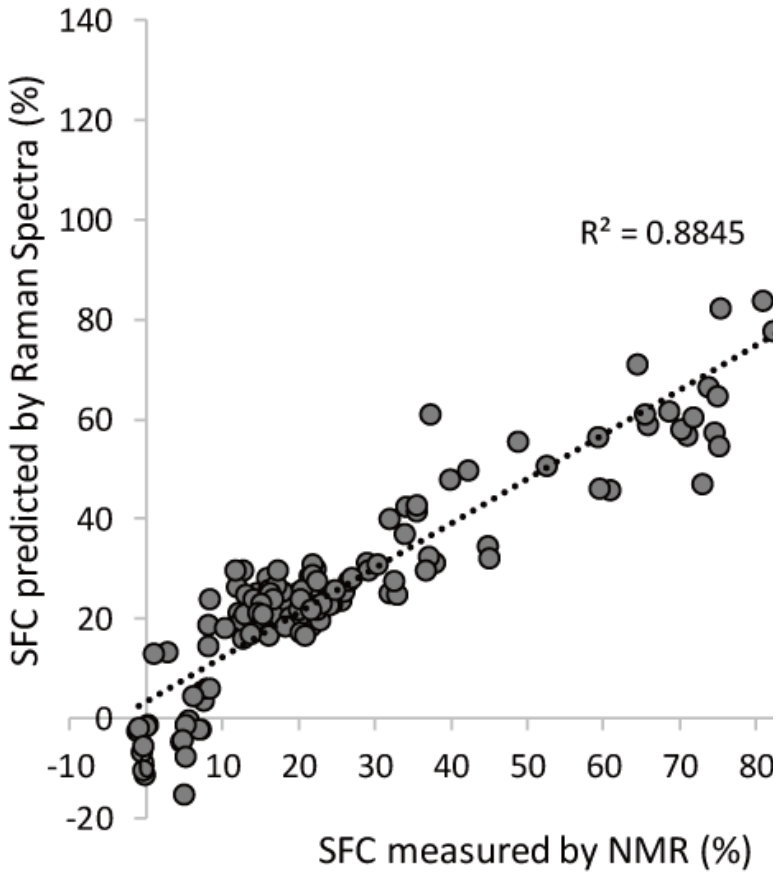

Fig. 1. Result of PCR analysis

Notes