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Application News

No. A598

Spectrophotometric Analysis

Fluorescence Discrimination Analysis of Powdered Beverages Using Reflection-Type Fiber

Because the Shimadzu RF-6000 spectrofluorophotometer features a large sample compartment with high expandability, many samples that produce fluorescence, including glass, plastics, and fruits, can be measured by placing the sample in the sample compartment. It is also possible to measure samples that are larger than the sample compartment or cannot be moved to the sample compartment by using a reflection-type fiber measurement unit.*1 Although powder samples generally require pretreatment such as fixing the sample prior to measurement, pretreatment is not necessary when a reflection-type fiber unit is used.

This article introduces measurement of powdered beverages using a reflection-type fiber measurement unit.

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Reflection-Type Fiber Measurement Unit

Fig. 1 shows the appearance of the RF-6000 with the reflection-type fiber measurement unit attached. Fig. 2 shows the condition when the base unit for fixing the fiber is mounted in the sample compartment of the RF-6000. Excitation light is taken from the sample compartment through a fiber (green line in Fig. 2) and irradiated on the sample, which is set in the external sample compartment shown in Fig. 3. The fluorescence which is produced from the sample is captured by the end face of the fiber, and is then introduced into the spectroscope and detector of the RF-6000 once again through another fiber (yellow line in Fig. 2). In order to capture the fluorescence from the sample efficiently, it is necessary to adjust the distance from the end surface of the fiber to the surface of the sample being measured to no more than 10 mm (for the adjustment mechanism, see Fig. 3). Samples with a maximum size of 100(W) × 100(D) × 50(t) mm can be set in the external sample compartment.*2 The fiber is 1.5 m in length and supports measurements from the ultraviolet to the visible range (250 nm to 900 nm).

*1 Semicustom product.
*2 The size of the external sample compartment and length of the fiber can be changed.

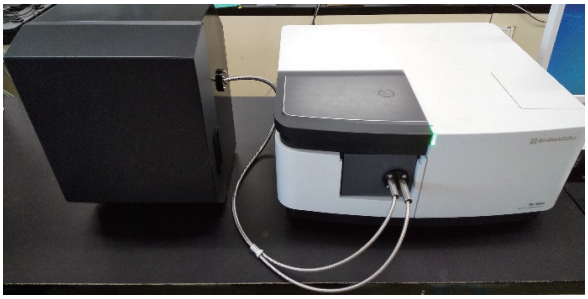

Fig. 1 Appearance of RF-6000 (Right) with Reflection-Type Fiber Measurement Unit Attached

Table 1 Measurement Conditions

Instruments	:	RF-6000, reflection-type fiber measurement unit
Spectrum Type	:	3D spectrum
Measured Wavelength Range	:	Ex 250 nm-800 nm, Em 300 nm -800 nm
Scanning Speed	:	12,000 nm/min
Wavelength Interval	:	Ex 5.0 nm, Em 1.0 nm
Bandwidth	:	Ex 5.0 nm, Em 5.0 nm
Sensitivity	:	Low

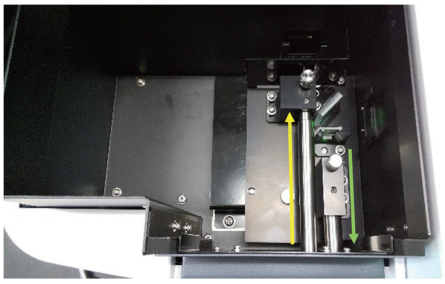

Fig. 2 Condition in Sample Compartment of RF-6000

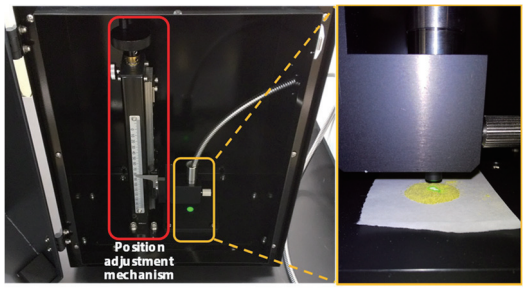

Fig. 3 External Sample Compartment of Reflection-Type Fiber Measurement Unit

Measurement of *Aojiru*, *Matcha*, and Green Tea Powders

Fig. 4 shows photographs of the eight types of commercially-available powdered beverages (*aojiru*, a vegetable drink made from green leafy vegetables; *matcha*, a type of powdered green tea; and green tea) used in the measurement. Two samples of two types of *aojiru* were prepared, one using green barley as the raw material and the other using kale. Powder samples can also be measured by using a solid sample holder (see Application News No. A508 for details). However, if a reflection-type fiber measurement unit is used, it is not necessary to place the sample into the holder, as mentioned above, and the measurement can be carried out simply by setting powder samples like those in Fig. 4 in the external sample compartment as-is.

Fig. 5 shows the 3D fluorescence spectra of the respective samples measured under the conditions in Table 1. Measurements were conducted three times with each type of sample by replacing the samples. The vertical axis (*y*-axis) of the 3D spectra shows the excitation wavelength (Ex), and the transverse axis (*x*-axis) shows the fluorescence wavelength (Em). The fluorescence intensity is shown by color.

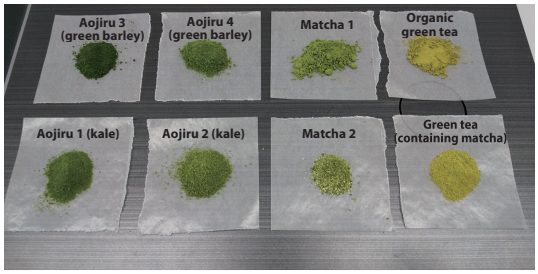

Fig. 4 Powder Samples

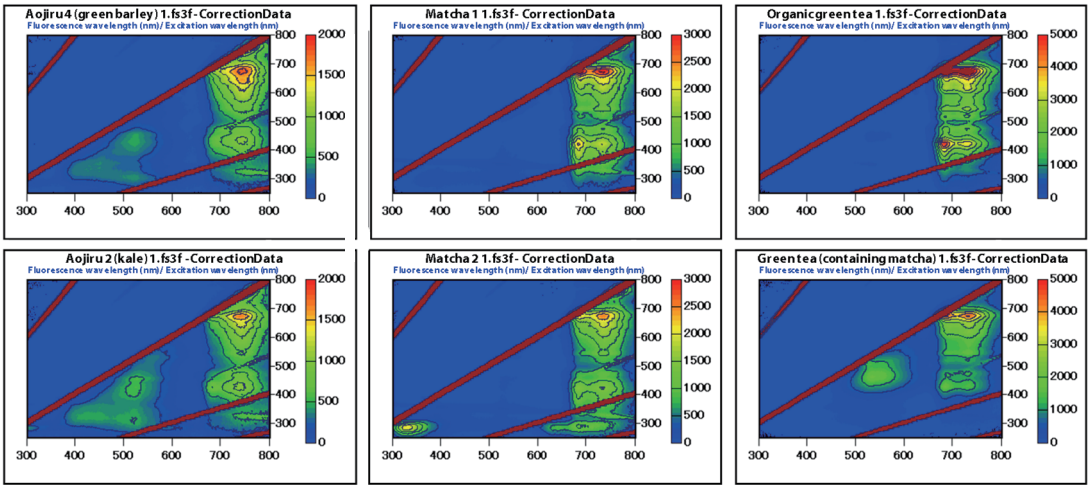

Fig. 5 3D Fluorescence Spectra of Powder Samples

With these samples, fluorescence was measured at around Em 740 nm with multiple excitation wavelengths. These are estimated to be fluorescence originating from chlorophyll. Depending on the sample, fluorescence was also confirmed at around Em 400 to 600 nm with Ex 300 to 500 nm. Using the representative peaks shown in Fig. 6, discrimination analysis was carried out with The Unscrambler®X multivariate analysis software.*3

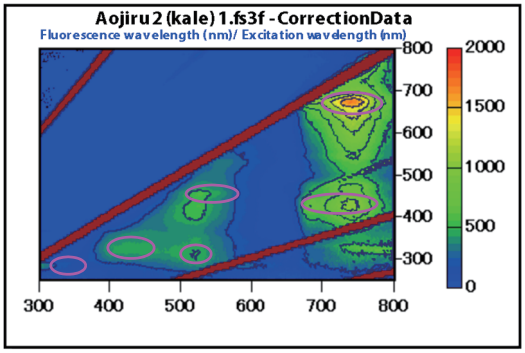

Fig. 6 Analysis Points in 3D Fluorescence Spectrum

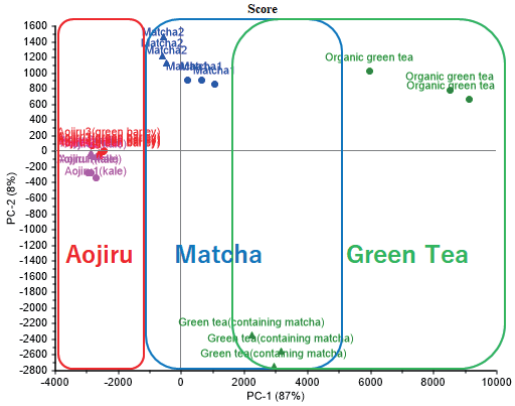

Fig. 7 Score Plot of Principal Component Analysis

Fig. 7 shows the score plot of the Principal Component Analysis (PCA). For details concerning reading of multivariate analysis results, please refer to Application News No. A508 and No. A524. Because the PC-1 axis (*x*-axis) is dominant in Fig. 7, it is inferred that *aojiru*, *matcha*, and green tea can be distinguished. The sample green tea (containing *matcha*) is plotted between *matcha* and green tea because it contains both *matcha* and green tea. Fig. 8 shows the score plot of the PCA for only *aojiru*. The PC-1 axis (*x*-axis) is dominant, and it can be understood that the raw materials of the *aojiru* samples (green barley and kale) can also be distinguished.

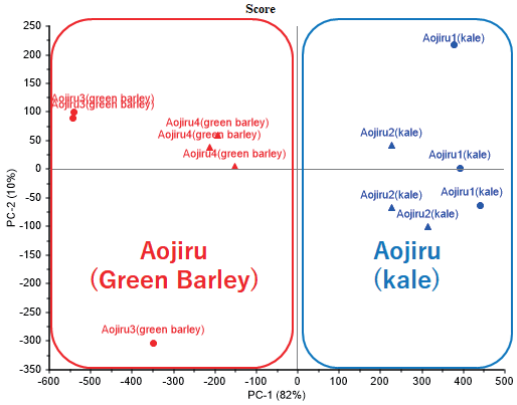

Fig. 8 Score Plot of PCA of *Aojiru* Samples

Conclusion

3D spectrometry and multivariate analysis of powder samples were carried out using a Shimadzu RF-6000 spectrofluorophotometer and a reflection-type fiber measurement unit. From the results of the multivariate analysis, it was possible to distinguish *aojiru*, *matcha*, and green tea, and classification of *aojiru* by raw materials was also possible.

Use of the reflection-type fiber measurement unit not only enables measurement of samples that cannot be measured in the sample compartment, but also eliminates the need for the pretreatment work required when measuring powders.

*3 The Unscrambler is a trademark or registered trademark of CAMO Software AS.

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