

Application of near infrared spectroscopy to measurement and control of moisture content during compost fermentation

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Introduction

Compost fermentation is one of the key technologies for municipal solid waste treatment and recycling of the residue from food processing. The compost fermentation is a microbial decomposition process and it is important to maintain the fermentation conditions at the optimal level. In particular, moisture content is one of the most important factors in hastening the compost fermentation.

Soybean-curd, called “tofu” in Japanese, is a traditional food which is eaten habitually in east Asia. The residue from the tofu processing, tofu refuse, putrefies very quickly with an unpleasant odour because of the high content of moisture and nutrients. Tofu refuse has been utilised as feed stuff. However, treatment of tofu refuse has recently become a serious problem because the amount used as feed stuff is on the decrease. Because tofu refuse does not contain toxic or harmful substances, it is very suitable for compost material.¹ Compost fermentation may be the most appropriate treatment for tofu refuse.

Near infrared (NIR) spectroscopy is widely used for rapid and non-destructive analysis in industries such as agriculture, food, pharmaceuticals, textiles, cosmetics and polymer production.² In fermentation processes, NIR has been applied to the measurement of ethanol concentration in the fermentation of molasses,³ wine,⁴ beer^{5,6} and rice vinegar.⁷ In animal cell culture, the concentrations of glucose, glutamine, ammonia and lactic acid have also been analysed simultaneously by NIR.⁸ NIR has been also applied to the measurement cell mass for mushroom cultivation in solid media.⁹

In the present study, NIR was applied to determine the moisture content in a compost made from tofu refuse. The application of NIR to measurement and control of moisture content during the compost fermentation process was discussed.

Materials and methods

Compost material and composter

The fresh tofu refuse used as the raw material for the compost in this study was supplied from a tofu factory (Eishoku Foods Co., Hatsukaichi, Hiroshima). Compost fermentation was performed in a home-use composter (Model EH4381B-H, Matsushita Electronic Co., Tokyo). The temperature of the compost in the composter was kept between 65–70°C by on-off control of the heater and by mixing with a triple impeller equipped in the composter. To accelerate the compost fermentation, compost made from tofu refuse in our laboratory containing microorganisms was used as the seed.

Compost fermentation

Tofu refuse (about 70% moisture content, 12 kg of wet weight) and the seed containing microorganisms (about 39% moisture content, 200 g of wet weight) were mixed in a plastic container. The fermentation was started by transferring the mixture into the composter. During the compost fermentation, water was supplied intermittently to the compost in order to control the moisture content. The compost was taken out to measure the moisture content of the compost by the drying method and the NIR method. The volume of the water added was calculated based on the moisture content measured. After addition of water to the compost, the compost was mixed well in the plastic container and the cultivation was restarted by returning the compost to the composter.

The reaction rate of the compost fermentation was defined as the total reduction in weight of the dry compost in the composter per unit time. The total weight of the dry compost in the composter was calculated by using the values of the moisture content measured and the total wet weight of the compost.

Experimental runs

Three experiments (Runs A–C) of compost fermentation were performed to confirm the effect of the moisture content on the compost fermentation. In Runs A and B, the moisture content of the compost was controlled at around 50% and at around 70%, respectively. In Run C, the moisture content was not controlled during the compost fermentation. In addition, an experiment (Run D) was performed to control the moisture content using the calibration equation from the NIR method during the compost fermentation process.

Measurement and control of moisture content

In Runs A and B, the moisture content of the compost was controlled by supplying water, the volume of which was calculated from an approximate moisture content of the sample and the total wet weight of the compost in the composter. The approximative moisture content was measured by the oven drying method (dried for 2 h at 130°C). Measurement of accurate values of the moisture content was also determined by the oven drying method (dried for 24 h at 130°C). In Run D, additional water volume was determined based on the value of total wet weight of the compost and the prediction of the moisture content using the NIR method.

Near infrared spectroscopy

The measurement of the near infrared (NIR) spectrum of the compost was carried out by the same procedure described in the previous study.¹⁰ A polyethylene bag (57 × 210 mm) filled with the sample was placed in a sample holder after being held at 25°C for 30 min. The reflectance values at wavelengths ranging from 400 to 2500 nm were measured at 2 nm intervals with a near infrared spectrometer (NIRS6500SPL, Nireco Co., Hachioji, Tokyo). All samples were measured in duplicate. To correct the shift of the baselines of the spectra, second derivative spectra ($d^2 \log R^{-1}$) were obtained numerically from the raw spectra.^{7,10}

Multiple linear regression (MLR) using the least squares method¹¹ was carried out between the NIR spectral data and the moisture content (Cact) obtained by the drying method for a calibration sample set ($n = 50$) to predict the moisture content (Cpre). The second derivatisation and the regression analysis were carried out using software called NSAS supplied by Nireco Co.

To evaluate the performance of the calibration equation obtained, validation was carried out using a validation sample set ($n = 35$), which was not used for calibration.

Results and discussion

Effect of moisture content on compost fermentation

Three experiments (Runs A–C) were performed to confirm the effect of the moisture content on compost fermentation, the results of which are shown in Figure 1. The development of the compost fermentation of Run B was better than that of Run A [Figure 1(b)]. The maximum values for the reaction rate of the compost fermentation of Runs A and B were 23.8 gh^{-1} and 27.9 gh^{-1} , respectively [Figure 1(c)]. Run C, on the other hand, showed a lower maximum value for the reaction rate than those of Runs A and B. These results suggest that the moisture content in the compost had a large effect on the reaction rate of compost fermentation. To promote compost fermentation, therefore, it is very important to measure the moisture content in the compost and maintain it at a suitable level.

NIR spectra of tofu refuse and compost

Figure 2(a) shows raw NIR spectra of fresh tofu refuse and the compost of Run B cultured for 0.96, 2.1 and 6.5 days. The moisture content in the fresh tofu refuse and the compost cultured for 0.96, 2.1 and 6.5 day was 72.0, 69.9, 70.2 and 70.5%, respectively. The three peaks observed at the wavelengths of 970, 1450 and 1940 nm in all spectra may be due mainly to the absorption of water.¹² The two peaks observed at around the wavelength of 2330 nm mainly depend on the absorption of the lipids originating from soybean¹³ and the polyethylene bag.¹⁰ The baseline of the spectrum shifted upwards as the fer-

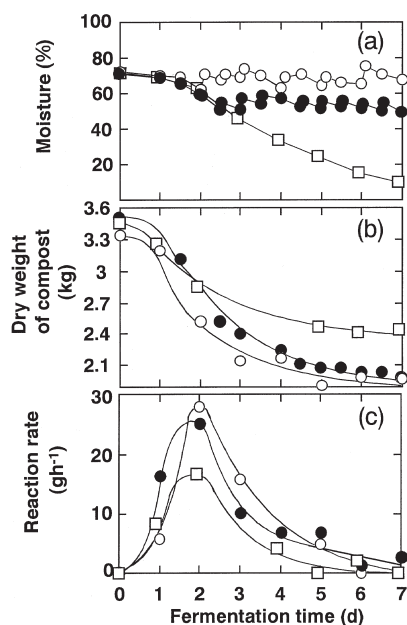


Figure 1. Time courses of moisture content (a), dry weight of compost material (b) and reaction rate (c). Symbols: ●, moisture content was controlled at 50% (Run A); ○, controlled at 70% (Run B); □, without control (Run C).

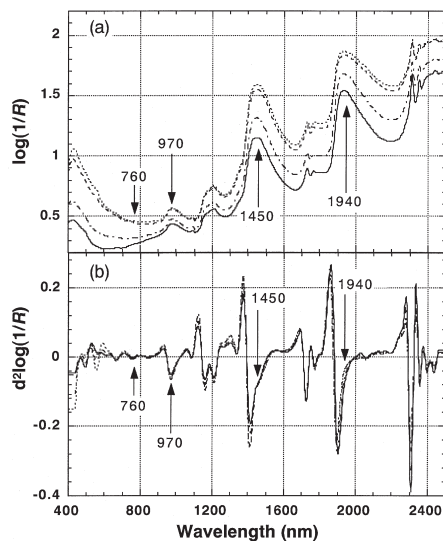


Figure 2. (a) Raw and (b) second-derivative NIR spectra of fresh Tofu refuse and the compost cultured for 0.96, 2.1 and 6.5 d of Run B. Moisture content in the fresh tofu refuse and the compost cultured for 0.96, 2.1 and 6.5 d was 72.0, 69.9, 70.2 and 70.5%, respectively. Lines; Tofu refuse (—) and compost cultured for 0.96 d (· · · · ·), 2.1 d (— — — —) and 6.5 d (- - - -).

mentation developed. The shift might be caused by a change in the properties of the compost. The effect of the properties of the compost could be made negligible using the second derivative spectrum [Figure 2(b)].

Figure 3(a) shows raw NIR spectra of the compost of Run A cultured for 3.0 days, of Run B for 2.1 days and of Run C for 5.0 days. The dry weight of the whole quantity of these compost in the composter was almost the same, i.e. the properties of the compost seemed to be similar to each other. The moisture content of the compost of Runs A, B and C was 51.3%, 70.7% and 24.0%, respectively. The baseline of the spectrum shifted upwards as the moisture content increased. The moisture content affected not only the absorption at the wavelength assigned to water but also the absorption at all wavelengths measured. The shift of the baseline of the spectrum of the compost observed in Figure 3(a) was corrected in the second derivative spectrum as shown in Figure 3(b). It is desirable to use the second derivative spectrum for measurement of the moisture content of the compost.

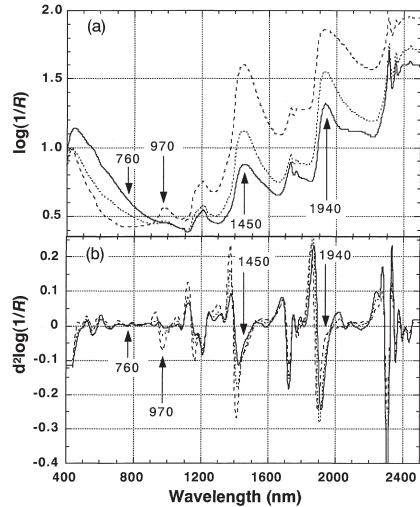


Figure 3. Raw (a) and second-derivative (b) NIR spectra of the compost of Run A cultured for 3.0 d, of Run B for 2.1 d and of Run C for 5.0 d. Moisture content of the compost of Runs A, B and C was 51.3%, 70.7% and 24.0%, respectively. Lines; Compost of Run A cultured for 3.0 d (- - - -), of Run B for 2.1 d (· · · ·) and of Run C for 5.0 d (—)

Calibration equation

In order to formulate a calibration equation, multiple regression analysis was conducted on the moisture content obtained by the drying method and that predicted by the NIR method for 50 samples ($n = 50$) of the compost of Runs A, B, C and three others. The results are shown in Table 1. Using the second derivative values at the wavelength of 960 nm, the best calibration equation was obtained. The

Table 1. Calibration and validation results for prediction of moisture content of compost.

Wavelength (nm)			Calibration ($n = 50$)		Validation ($n = 35$)	
λ_1	λ_2	λ_3	r	SEC (%)	r	SEP (%)
754	—	—	0.676	6.14	0.635	7.04
960	—	—	0.987	1.33	0.979	1.85
1406	—	—	0.865	4.18	0.834	5.03
1888	—	—	0.874	4.05	0.835	5.01
960	1406	—	0.988	1.31	0.978	1.91
960	1888	—	0.988	1.30	0.979	1.88
960	1888	1406	0.988	1.30	0.979	1.88

performance of the calibration equation could not be markedly improved using several values of $d^2\log R^{-1}$.

The following calibration equation was obtained using $d^2\log(1/R)$ at 960 nm.

$$C_{\text{pre}} = 41.85 - 693.8 \cdot d^2\log R_{960}^{-1} \quad (1)$$

The moisture content in the validation sample set ($n = 35$), which was not used for the calibration, was predicted using the calibration equation, Equation 1 and compared with the values of C_{act} . The standard error of prediction (SEP) was 1.85%. The values obtained by the conventional method and those obtained by NIR were very similar with $r = 0.979$.

Practical application

Real-time monitoring and control of the moisture content during compost fermentation (Run D) was performed using the calibration equation, Equation 1. Time courses of moisture content (a), dry weight of compost (b) and compost reaction rate (c) during the compost fermentation are shown in Figure 4. The fermentation was carried out using 50% as the target value of the moisture content of the compost. In Figure 4(a), the predicted values by the NIR method are represented by open symbols, while closed symbols denote those obtained by the drying method. The moisture content in the compost could be measured and controlled by the NIR method as shown in Figure 4(a). This result suggested that the progress of the compost fermentation was smooth using the NIR method to measure and control the moisture content during the fermentation process.

The results of this study suggest that NIR is a useful method for real-time monitoring and control of moisture content in compost fermentation. The operational procedure involved in NIR is very simple and non-destructive and the time required for the measurement is a few minutes. In addition, on-line monitoring using an optical fibre probe, might be possible instead of packing the compost sample in a polyethylene bag before the measurement as we did in this study. To develop an efficient composter, simultaneous measurement of the moisture, carbon and nitrogen content of the compost will be studied in the future.

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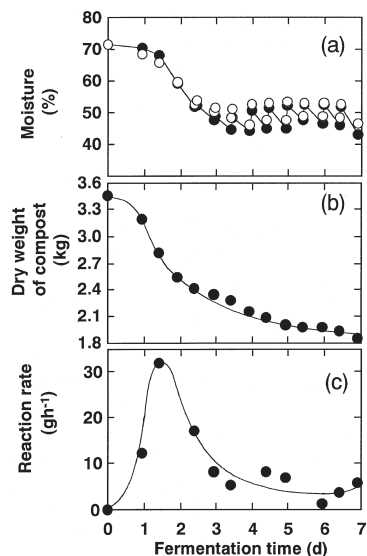


Figure 4. (a) Time courses of moisture content, (b) dry weight of compost material and (c) reaction rate during the experiment in which the moisture content was controlled using the prediction results of the NIR method (Run D). Symbols: ●, measurement of the moisture content by the drying method; ○, predicted values of the moisture content by the NIR method.

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