Measurement of Lipid Content of Garbage Compost Using Near Infrared Spectroscopy

Takayuki FUJIWARA1,2,†, Keiichi MURAKAMI1, Minoru TAKEMOTO3, and Syunrokuro FUJIWARA3

1Agricultural Research Division, Mie Prefectural Science and Technology Center, 530, Ureshinokawagita, Matsusaka, Mie 515–2316, Japan 2Present address: Industrial Research Division, Mie Prefectural Science and Technology Center, 5-5-45, Takajaya, Tsu, Mie 514–0819, Japan 3Kanagawa Agricultural Research Center, 1617, Kamikisawa, Hiratsuka, Kanagawa 259–1204, Japan

The determination of the lipid content of garbage compost by near infrared spectroscopy (NIRS) was investigated, because lipid content is known to be an inhibitor of plant growth and germination. The garbage compost samples, which were produced by electric garbage processors, were obtained from canteens in schools and factories, food service facilities, homes, restaurants, and a hospital. Lipid contents of the samples were spread over wide range (0.28–17.70%), and the main fatty acids in most samples were palmitic, stearic, oleic, and linoleic acids. The reflectance spectra of air-dried and milled compost samples were measured using a scanning monochromator. Second derivative spectra from 700 to 2500 nm and multiple regression analysis were used to develop calibration equations for lipid content. The main wavelength of the best calibration equations was 2310 nm, which was attributed to the absorption bands of the CH2 stretch-bend combination of fatty acids. The standard error of prediction (SEP) of the best calibration was 1.24%. Although the fatty acid composition of the hospital sample was very different from others, it did not cause large errors in the estimates. In conclusion, NIRS is thought to be a practical method for evaluating plant inhibition caused by garbage compost.

Key words: garbage compost, near infrared spectroscopy, lipid, fatty acid

1. Introduction

The annual amount of food waste, such as dead stock, food leftovers, and kitchen refuse, that is generated during the manufacture, distribution, and consumption of food is twenty billion tonnes in Japan, and only less than 10% of it is recycled [1]. In order to promote the reduction of food waste, the "Law for Promotion of Recycling and Related Activities for the Treatment of Cyclical Food Resources" was enacted in 2001 [1, 2, i]. Another law, the "Law for Promoting the Introduction of Sustainable Agricultural Production Practices" , which was enacted in 1999, encourages the application of compost or organic materials as one of the techniques to secure a good farming environment [ii]. As a result, the production of compost from food waste and the application of this compost became important activities for both the reduction of waste and the promotion of sustainable agriculture. In contrast to industrial food waste, which is discharged in large quantities, household refuse and food waste from restaurants or factory canteens are discharged dispersely [3]. To dispose of such garbage sanitarily and efficiently, electric garbage processors have been developed and introduced to various places [3, 4], such as canteens in schools and factories, welfare facilities, restaurants, and hospitals [5]. Since one use advertised for the treated garbage ('garbage compost' hereafter) produced by such devices is as compost or fertilizer [3], application of it to farmland is expected to support organic farming. However, the application of garbage compost to farmland raises concerns about some of its constituents, such as lipid, salt, and heavy metals, inhibiting crop growth and polluting the soil [1, 3, 6]. Especially, the lipids in garbage compost are known to inhibit the germination and growth of plants [3, 6]. Takemoto studied the inhibition of the growth of komatsuna (Brassica rapa L. Perviridis Group) by garbage compost lipids, and reported that the main cause of the inhibition was the immobilization of nitrogen and the suppression of nitrogen mineralization [3]. He
highlighted the importance of measuring the lipid content of garbage compost intended for agricultural utilization. The Soxhlet extraction method, which is the standard procedure for analyzing lipid content, has some drawbacks, such as long analysis time, requirement of technical skill, and the use of solvents. Therefore, he devised two simple methods of estimating the lipid content of garbage compost [3]. One involved evaluating the extent of the permeation of lipid into oil-absorbing paper. The other was an evaluation of the turbidity of a mixture of water and an ethanol extract of the garbage compost. However, these methods cannot quantify lipid content. For the production and application of good garbage compost, it is necessary to develop a more precise method of determining lipid content, to ensure that the compost is harmless to crops. Compost found to have a high lipid content should be fermented again, or be re-composted after being mixed with other materials such as cattle feces and pruning residues [3, 4].

Near infrared spectroscopy (NIRS) is an analytical method that can be used to determine the constituents of samples easily, rapidly, and nondestructively. Because many fatty acids have absorption bands in the near infrared region, NIRS has been used to determine the fat and oil content of various foods [7]. However, there are a few reports of NIRS being applied to food waste compost. Masui et al. reported that NIRS could accurately determine the lipid content of the compost of tofu (soybean-curd) refuse in a study of the management of the compost’s fermentation [8]. Since NIRS is associated with high costs and large equipment, the near infrared moisture sensor – with a lower cost and smaller size – has been developed, and it is used in household electric garbage processors [9]. If precise lipid analysis of garbage compost by NIRS is developed, it is anticipated that it will be used not only for the evaluation of the compost using an ordinary near infrared spectrophotometer, but also as the lipid sensor in electric garbage processors.

Most of the lipids in many foods are fatty acids, and each food has its own fatty acid profile [10]. Since the composition of food wastes largely depends on the waste source, the fatty acid composition of garbage compost is assumed to vary widely. This variation could affect the accuracy of the lipid determination by NIRS.

In this study, we collected various garbage compost samples and analyzed their fatty acid composition, then investigated the accuracy of the estimation of their lipid content by NIRS.

2. Materials and Methods

2.1 Compost samples

The garbage compost samples (number of samples \( n = 88 \)) were obtained from canteens in schools and factories, food service facilities, homes, restaurants, and a hospital, in Mie and Kanagawa prefectures, Japan. The compost was from food waste, such as cooking refuse and leftovers, and produced using electric garbage processors. The samples were air-dried and milled (cyclone sample mill fitted with 1.0 mm screen, UDY Corporation, Fort Collins, CO, USA) for the lipid analysis, calibration, and spectra capture. Twenty of the samples were also used for analyzing lipid composition.

2.2 Reference analysis

2.2.1 Lipid content

The lipid content of two grams of each sample was measured using an eight-hour diethyl ether extraction (Soxhlet extraction method) [11]. The extract was dried and weighed. The lipid content was calculated as a percentage of dry weight of the extract.

2.2.2 Fatty acid composition

The major fatty acids of the samples were measured as fatty acid methyl esters, which were prepared by a boron trifluoride–methanol method, using FID temperature–programmed gas chromatography (GC) [11].

The lipid extracted from one gram of each sample by Soxhlet extraction was saponified using 3 mL of 0.5 mol L\(^{-1}\) sodium hydroxide–methanol (80°C for 15 min) and then methylated with boron trifluoride–methanol. The methyl esters of the fatty acids were extracted with hexane and refined with saturated sodium chloride solution. The hexane fraction, which was dehydrated by sulfuric anhydride sodium, was used for GC analysis.

A gas chromatograph (G-5000A, HITACHI Co., Tokyo, Japan) equipped with a FID detector and fitted with a capillary column (CP-Sil 88 for FAME, GL Sciences, Tokyo, Japan) was used to determine the methyl esters of the fatty acids. The column temperature was raised from 180°C to 220°C at a rate of 5°C per min.

2.3 Near infrared spectroscopy

2.3.1 Near infrared spectra

The reflectance spectra of each sample were measured using an NIR spectrophotometer (Model 6500, FOSS-NIRSystems, Laurel, MD, USA) with a micro sample cup. Samples were scanned 32 times from 400 to 2500 nm, and
data were collected every 2 nm. Spectra were obtained as \( \log(1/Re) \) with a ceramic tile used as a reflectance standard.

All procedures of correcting spectra and handling data were conducted using the Vision software package (ver.3.2, FOSS-NIRSystems).

### 2.3.2 Regression analysis

Data of each sample were divided into a calibration set \((n=48)\) and prediction set \((n=40)\). These sample sets were selected so that the distribution of lipid content was similarly spread and care was taken to ensure that the calibration set did not concentrate around any point in the range.

To suppress constant background signals and to enhance the visual resolution, second derivative spectra of each sample were used for calibration and prediction [12]. When producing the derivatives, segment size and gap size were set to 10 and 0, respectively.

The calibration equations were developed using multiple linear regressions, using the second-derivative values of absorbance \(d^2\log(1/Re)\) and the lipid contents of the calibration set. The data of \(d^2\log(1/Re)\) from 700 nm to 2500 nm, which corresponds to the near infrared region, were used for calibration. The accuracy of the calibration equations was determined by evaluating the differences between the values predicted by the equations and the reference analysis values of the prediction set.

### 3. Results and Discussion

#### 3.1 Lipid content and fatty acid composition of the samples

Table 1 shows the sources and lipid content of the compost samples. The lipid contents of the samples were widely spread and there was no relationship between lipid content and sample source.

Figure 1 shows fatty acid composition of the garbage compost samples. The dominant fatty acids of the samples, except the sample from the hospital, were palmitic (C16:0), stearic (C18:0), oleic (C18:1), and linoleic (C18:2) acids. The ratio of these fatty acids to some extent depended on the samples. Interestingly, the fatty acid composition of the hospital sample was very different from the others, as it contained high proportions of linolenic (C18:3) and eicosapentaenoic acids (C20:5). This may have been caused by the fatty acid component of the hospital diet.

#### 3.2 Near infrared spectra and absorption bands of fatty acids

The wavelengths that showed a high negative correlation coefficient \((r > |0.8|)\) between \(d^2\log(1/Re)\) and lipid content were 1716, 1760, 2146, 2310, and 2344 nm. To make a robust calibration equation, it is important to select the main wavelengths which have not only high correlations between \(d^2\log(1/Re)\) and the constituents being calibrated for but also absorptions related to the constituents. Therefore, we examined the attributions of the wavelengths mentioned above.

Figure 2 shows second derivative spectra of a compost sample and the same sample with added fatty acids. Since the second derivative transfers peak maxima into minima [12], absorption peaks have been turned into troughs. The absorption peaks of the compost samples were observed at approximately these wavelengths (i.e., 1760, 2146, 2310, 2344 nm).
Fig. 2 Spectra of garbage compost with added fatty acids. Control: a garbage compost sample without addition of fatty acids (lipid content=4.65%). Each fatty acid was 5% of the compost sample.

and 2344 nm), except 1716 nm. Although an absorption peak at 1716 nm was not observed in the compost samples, this region is thought to be the absorption band of lipids because Masui et al. [8] and Nakamichi et al. [13] reported absorption peaks of a Soxhlet (chloroform/methanol) extract of compost of tofu refuse at 1712 nm, and soybean oil at 1716 nm, respectively. Osborne and Fearn [7] reported that the major near infrared absorption bands in fat or oil are due to the long chain fatty acid moiety. Of the wavelengths mentioned above, 1716 and 1760 nm have been associated with the CH2 stretch first overtone band, 2310 and 2344 nm with the CH2 stretch-bend combinations band [7]. Parts of the composts’ second derivative spectra reflect the addition of palmitic, stearic, oleic, and linoleic acids (Fig. 2). Increases in peak height were observed with additions of these fatty acids at 1760, 2310, and 2344 nm. On the other hand, the additions of oleic (C18:1) and linoleic (C18:2) acids increased absorption at 1716 and 2146 nm, but the additions of palmitic (C16:0) and stearic (C18:0) acids did not affect absorption very much (Fig. 2). This phenomenon at 1716 nm supports the observation by Sato [14], who reported that oleic (C18:1) and linoleic (C18:2) acids had absorption peaks at approximately 1710-1725 nm, whereas palmitic (C16:0) and stearic (C18:0) acids had them at 1725-1730 nm. As a result, the absorption band of fatty acids in this region was shifted to the shorter wavelength as the unsaturation in the moieties increased [14]. Also, the phenomenon at 2146 nm supports the association of an absorption peak at this wavelength region with the combination band of CH stretch-bend and CC stretch-bend due to cis-unsaturation of unsaturated fatty acids [7, 15].

Although the other absorption bands, such as that for the CH2 stretch second overtone around 1200 nm and for the third overtone around 928 nm, have been reported [15], a high correlation between d^2log(1/Re) and lipid content was not observed.

Therefore, we carried out multiple regression analysis with 1716, 1760, 2146, 2310, and 2344 nm as the first wavelengths.

3.3 Regression analysis

Table 2 shows the results of the multiple regression analyses and the predictions of the calibration equations, which showed good results for the determination of lipid content. Although the second or later wavelengths (970, 1800, and 1900 nm) were selected in many equations, these were not absorption peaks of fatty acids nor were any special absorption peaks observed in the second derivative spectra of either the fatty acids or the samples, and the spectra of the samples overlapped each other. These phenomena were similar to those observed by Kawano et al. [16], who reported on the wavelength of 878 nm for the calibration equation of the Brix value of peach. Although the calibration equations based on one wavelength, which is attributed to lipids, was relatively accurate, the addition of more wavelengths led to better results (Table 2). It was assumed that the wavelengths such as
970, 1800, and 1900 nm were selected as correction factors for the main wavelengths of calibration equations.

The calibration equation described below showed the smallest bias-corrected standard error of prediction (SEP), of 1.24%.

\[ C = 1.86 - 757A_{2146} - 317A_{2310} + 313A_{1902} - 3138A_{1062} \]  

(1)

where \( C \) is lipid content (%), and \( A_\lambda \) are \( d_2\log(1/Re) \) at \( \lambda \) (nm).

Figure 3 shows comparisons of predicted and actual values of lipid content of the calibration equation (1). Since the estimated value of the sample from the hospital had only a small error (+1.5%), variation in fatty acid composition did not seem to affect the accuracy of NIRS very much. Remarkably, the calibration equation with only two wavelengths (2310 and 1900 nm) also showed a relatively small SEP. In general, calibration equations including the term of 2310 nm showed high accuracies.

To standardize the predictive accuracy of NIRS, the ratio of standard deviation of prediction set to SEP (RPD) and the ratio of range in prediction set to SEP (RER) were defined [17]. In agricultural commodities, values of RPD > 3 and RER > 10 are considered to indicate successful calibrations [18]. The values of RPD and RER of calibration equations for lipid content with two or more wavelengths are higher than the above standard (Table 2). Although the SEP was higher than that of reported by Masui et al. [8] for tofu refuse compost (SEP = 0.815), the accuracy of our lipid content estimates are reasonable, because the sources of the compost and the composting systems differed between samples.

There have been independent moves to establish quality standards for garbage compost [3, 6]. For example, Zenkoku Syokuhin Risaikuru Kyokai (the National Food Recycling Association) set the standard for the lipid content of dry matter at 5% or less [6]. It is expected that NIRS will be used as a method for assessing whether lipid content is below an established standard like this. However, note that the NIRS method is prone to some error and it will be necessary to consider the error esti-

---

**Table 2** Results of multiple regression and errors between predicted and actual lipid content of garbage compost as determined by near infrared spectroscopy using second derivative spectra.

<table>
<thead>
<tr>
<th>Wavelength used (nm)</th>
<th>( R )</th>
<th>SEC (%)</th>
<th>SEP (%)</th>
<th>Bias (%)</th>
<th>RPD</th>
<th>RER</th>
</tr>
</thead>
<tbody>
<tr>
<td>2344</td>
<td>-0.934</td>
<td>1.54</td>
<td>1.80</td>
<td>0.37</td>
<td>2.8</td>
<td>8.5</td>
</tr>
<tr>
<td>2344, 1800, 1900, 970</td>
<td>0.965</td>
<td>1.17</td>
<td>1.47</td>
<td>0.47</td>
<td>3.4</td>
<td>10.5</td>
</tr>
<tr>
<td>2310</td>
<td>-0.928</td>
<td>1.61</td>
<td>1.70</td>
<td>0.09</td>
<td>2.9</td>
<td>9.1</td>
</tr>
<tr>
<td>2310, 1900</td>
<td>0.957</td>
<td>1.27</td>
<td>1.34</td>
<td>0.54</td>
<td>3.7</td>
<td>11.5</td>
</tr>
<tr>
<td>2310, 1900, 2374</td>
<td>0.965</td>
<td>1.15</td>
<td>1.32</td>
<td>0.47</td>
<td>3.8</td>
<td>11.7</td>
</tr>
<tr>
<td>2310, 1900, 2374, 970</td>
<td>0.972</td>
<td>1.05</td>
<td>1.27</td>
<td>0.39</td>
<td>3.9</td>
<td>12.1</td>
</tr>
<tr>
<td>2146</td>
<td>-0.887</td>
<td>2.00</td>
<td>2.89</td>
<td>0.17</td>
<td>1.7</td>
<td>5.3</td>
</tr>
<tr>
<td>2146, 2310</td>
<td>0.932</td>
<td>1.59</td>
<td>1.54</td>
<td>0.12</td>
<td>3.2</td>
<td>10.0</td>
</tr>
<tr>
<td>2146, 2310, 1902</td>
<td>0.962</td>
<td>1.22</td>
<td>1.27</td>
<td>0.47</td>
<td>3.9</td>
<td>12.1</td>
</tr>
<tr>
<td>2146, 2310, 1902, 1058</td>
<td>0.966</td>
<td>1.15</td>
<td>1.24</td>
<td>0.37</td>
<td>4.0</td>
<td>12.4</td>
</tr>
<tr>
<td>1760</td>
<td>-0.886</td>
<td>2.00</td>
<td>2.30</td>
<td>0.37</td>
<td>2.2</td>
<td>6.7</td>
</tr>
<tr>
<td>1760, 1902, 2310, 2370</td>
<td>0.965</td>
<td>1.17</td>
<td>1.28</td>
<td>0.35</td>
<td>3.9</td>
<td>12.0</td>
</tr>
<tr>
<td>1716</td>
<td>-0.899</td>
<td>1.90</td>
<td>2.52</td>
<td>0.34</td>
<td>2.0</td>
<td>6.1</td>
</tr>
<tr>
<td>1716, 2344, 1800</td>
<td>0.954</td>
<td>1.32</td>
<td>1.43</td>
<td>0.27</td>
<td>3.5</td>
<td>10.7</td>
</tr>
</tbody>
</table>

\( R \), Multiple correlation coefficient; SEC, standard error of calibration; SEP, bias-corrected standard error of prediction; Bias, mean difference between analyzed value using the standard method and predicted value using near infrared spectroscopy; RPD, Ratio of standard deviation of prediction set to SEP; RER, Ratio of range in the prediction set to SEP; ○, The calibration equation with lowest SEP.
mates in the evaluation of compost quality. Usually, as the errors about a calibration equation have a Gaussian distribution, 95% of the errors on average will be within ±2 (standard error of calibration) [19]. Although NIRS will require such attention to errors, it is considered to be very practical method of evaluating plant inhibition by garbage compost, because it is sufficiently accurate for determining lipid content. Furthermore, it has many advantages; for example, it requires less technical skill, it has short analysis time, it requires no reagents, and it enables the measurement of other constituents, such as fertilizer nutrients [20] and raw materials [21], at the same time.

4. Conclusion

The main fatty acids of most garbage compost samples were palmitic, stearic, oleic, and linoleic acids. Some clear absorption peaks were observed in the second derivative spectra of compost samples and fatty acids. In those absorption peaks, five wavelengths of 1716, 1760, 2146, 2310, and 2344 nm had a high correlation between $d^2\log(1/Re)$ and lipid content as determined by the standard analytical method. The lipid content of garbage compost could be determined with sufficient accuracy by NIRS using calibration equations including these wavelengths, especially 2310 nm. The best SEP of 1.24% was obtained from the calibration equation based on the terms of 2146, 2310, 1902, and 2370 nm. Although one sample from a hospital had a very different fatty acid composition from others, a large error was not observed in the estimates. It is considered that NIRS is a practical method of evaluating plant inhibition caused by garbage compost.

Acknowledgment

The authors would like to thank Dr. K. Kagotani and Mr. A. Ohmi in Tsuji Oil Mill Co., Ltd. for their help in the GC analysis of fatty acids.

REFERENCES

[18] D. F. Malley, L. Yesmin, R. G. Eilers; Rapid analysis of hog


URL cited

近赤外分光法による生ごみ堆肥の脂質含量の測定

藤原孝之1,2,*, 村上圭一1, 竹本 稔3, 藤原俊六郎3

1 三重県科学技術振興センター農業研究部
2 三重県科学技術振興センター工業研究部
3 神奈川県農業技術センター

「持続性の高い農業生産方式の導入の促進に関する法律」（1999年施行）により、減化学肥料栽培および有機質資材の有効利用による土作りの推進、地力の維持が促進されている。一方、「食品循環資源の再生利用等の促進に関する法律」（食品リサイクル法、2001年施行）では、食品廃棄物の有効利用の促進法を推進するため、事業所や家庭から排出される生ごみの堆肥化が注目されており、電動式生ごみ処理装置の普及が進んでいる。しかし、生ごみ堆肥の中には農作物の生育に悪影響を及ぼすものもあり、その原因の一つとして堆肥中の脂質が知られているため、近年では自営体が生ごみ堆肥の脂質含量の最低基準値を設定する動きがある。このような背景から、生ごみ堆肥の脂質含量を簡便に測定する方法が望まれているが、迅速性と定量性を兼ね備えた方法はまだない。近赤外光分光法は、食品そのの簡単・迅速な非破壊分析法として知られており、生ごみ処理機の水分センサーにも実用化されている。近赤外光分光法により生ごみ堆肥の脂質含量が測定できれば、市販の近赤外光分光計を用いた生ごみ堆肥の植物生育阻害程度の簡易評価が可能になるほか、電動式生ごみ処理装置の組み込みに関する応用も期待される。そこで、近赤外光分光法による生ごみ堆肥の脂質含量の測定精度を検討した。三重県および神奈川県の工場食堂、学校、給食センター、一般家庭、飲食店および病院において製作された生ごみ堆肥88点を採取し、実験に用いた。堆肥試料の脂質含量は、最低0.28%、最高17.70%と大きくばらつきがあった。代表的な試料の脂肪酸組成を分析したところ、ほとんどどの試料ではパラミチン酸、ステアリン酸、オレイン酸およびリノール酸が多かった。堆肥試料の700～2500 nmにおける近赤外スペクトルの二値微分値と、ジェチルエーテル抽出法による脂質含量の相関が高く、かつ前述の脂肪酸のピークが認められる波長は2344, 2310, 2146, 1760および1716 nmであったので、これらを主波長として重回帰分析により検量線を作成した。最も良好な測定精度を示した検量線、前記のうち2310 nmおよび2146 nmを含む4波長の組み合わせ多列式であり、SEP（検量線評価時の標準誤差）は1.24%であった。なお、病院で採取した生ごみ堆肥1点については、他の堆肥と脂肪酸組成が大きく異なったが、近赤外光分光法による測定誤差は小さかった。近赤外光分光法による脂質含量の測定値を用いて生ごみ堆肥の農作物への安全性評価を行う場合は、その測定誤差を考慮して使用することが必要であるが、今回の測定精度であれば十分に実用性があると考えられた。