Examination of compost maturity using reflectance

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SUMMARY

Composting is one of the most popular recycling processes for organic waste. Composting plays an important role in waste and by-product management and is becoming increasingly important in both sustainable energy management and circular economy. Composting transforms organic matter to produce a safe and stable by-product (compost) that can be applied to arable land in a similar way to fertilizer. Physical, chemical and biological methods can be used to monitor the process and to determine the maturity of the compost, as spectrometric/spectroscopic methods play an important role in the analysis of different environmental samples.

Our aim was to (1) non-destructively detect the effects of different additive ratios on the spectral properties of the composting process and the spectral data of different compost mixtures, (2) to find the wavelength ranges of the reflectance curve (inflection points) sensitive to compost maturity, (3) to determine the correlation between the inflection points and the chemical and physical parameters measured in compost by conventional methods.

The mixture of broiler and hen manure and zeolite was composted 62 days in windrow composting. In the composting experiment, the moisture content and temperature (°C) were measured every three days and compost samples were taken and in 10% destillated aquaeous suspension were measured the pH and electrical conductivity (mS cm⁻¹). Compost samples dried to mass stability were spectrally analyzed in the wavelength range 400–1000 nm with AvaSpec 2048 spectrometer.

Based on the results, the reflectance of mature compost were smaller in the last days of composting than the reflectance values of day 0 samples, thus compost maturity can be detected spectral in the VIS-NIR wavelength range. For the tested compost prisms, the reflectance of each sampling day shows a constant slope, with a significant overlap of the reflectance curves up to 400–700 nm wavelength range, and there was a breakpoint in the 700–750 nm wavelength range which was proved by binary encoding.

Keywords: composting, reflectance, spectroscopy, manure, maturity

INTRODUCTION

Composting is an environmentally friendly biological process of aerobic thermophilic microbial degradation of wastes and by-products by populations of the various microorganism which leads to a stabilised, mature, deodorised, hygenic product, rich in humic substances, free of pathogens and marketable as organic amendment or fertilizer (Haug, 1993; Galvez-Sola et al., 2010). Composting is an appealing solution for sustainable management of manure and it is importance to solve the problem of waste management. The compost quality for land application depends on its maturity and stability (Albrecht et al., 2008). A great number of physical, physico-chemical, chemical and biological methods used to study the properties of compost (Bernai et al., 1998; Itavaara et al., 2002; Wang et al., 2004; Kovács and Füleky, 2016), such as C/N ratio, humified organic carbon, cation exchange capacity, microbial respiration, enzyme activities (Chen, 2003; Castaldi et al., 2005; Tang et al., 2006; Tiquia, 2005). Visible (400-800 nm) and near infrared (800-2500 nm) reflectance spectroscopy (VIS-NIR) is an emerging tool for environmental analyses (Galvez-Solva et al., 2010).

VIS-NIR technique is non-destructive, with an easy sample preparation and rapid method. This technology has been used to determinate physical and chemical parameters in manure compost (Malley et al., 2005; Huang et al., 2008), C/N contents is sewage sludge and green waste compost (Albrecht et al., 2008), nitrogen content of poultry manure compost (Fujiwara and Murakami, 2007). The resulting spectra give a signature with important biochemical information about the character and number of functional groups (such as – CH, -OH, -NH), widely applied to investigate the decomposition process in soil, study the composting process (Gillon et al., 1993), and study the compost quality (Bonifazi et al., 2008).

MATERIALS AND METHODS

The laboratory experiments were carried out at the Institute of Water and Environmental Management, Research Centre for Organic Materials, University of Debrecen.

The broiler and hen manure used in the composting experiments was obtained from the Baromfi Coop Kft. The bulking agent used was zeolite, which was derived from Mád, a village in Borsod-Abaúj-Zemplén County in northeastern Hungary. Main characteristics of initial materials are shown in *Table 1*.

Experimental and Sampling Design

The compost windrows were of the same size and consisted of the same 40 kg of a mixture of broiler and hen manure in 1/3:2/3 ratio, to which 0.4-2.8 kg of zeolite was added in increasing amounts of 0-1-2-5-7%. The ratio of the two type of manures was the same as in the fermentation tanks of Baromfi Coop Ltd. The composting experiment was 62 days and the windrows were homogenised manually every three days.



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Table 1: Main characteristics of initial materials

Daramatar	Broiler Hen		Zaalita	
I al allietel	manure	manure	Zeonte	
pH _{H2O}	6.91	6.59	8.49	
OM (%) ¹	58.81	66.18	1.16	
EC (mS cm-1)2	11.10	12.78	0.65	
TN (%) ³	2.75	2.14	0.08	

¹OM: Organic matter; ²EC: Electrical conductivity; ³TN: Total nitrogen.

The sampling strategy was designed according to the standard for organic fertilizers (MSZ-08-0014-78) by sampling the front, middle and back prisms along the longitudinal axis of the prism in 15, 35 and 55 cm length sections, each at a depth of 12 cm. All samples was oven-dried 105 °C 24 hours (MSZ EN 21420-18:2005) and were sieved (< 500 μ m mesh).

Temperature was measured at the three points in the longitudinal section of the prisms used to design the sampling strategy: the first 15 cm, the middle 35 cm and the last 55 cm long sections, each at a depth of 12 cm. The 12 cm depth was determined by the length of the thermometer probe of the measuring instrument (PT100). The pH was measured by potentiometry method and the electrical conductivity by

ACTA AGRARIA DEBRECENIENSIS 2022-1

conductometry method in three replicates. The pH and electrical conductivity (mS cm⁻¹) were measured from a 10% distilled aqueous suspension after 24 hours of incubation.

VIS-NIR analysis

The AvaSpec 2048 spectrometer was used in the 400–1000 nm wavelength range, in the visible (VIS; 400-700 nm) and near infrared (NIR - Near Infra Red, 700–1000 nm) wavelength range, to analyse compost samples with a resolution of 0.566 nm and an accuracy of 1 nm. The spectrometer consists of three parts: a spectrometer (detector), 8 µm diameter fibre optics and a halogen light source (Figure 1). To ensure accurate measurements and to eliminate the interference of electromagnetic radiation from the fluorescent tubes, the measurements were performed in a closed laboratory box (Riczu, 2015; Bökfi et al., 2016; Nagy et al., 2012). For the calibration of the measurements, it is important that the reference panel WS-2 (made of white diffuse teflon and reflecting 98% of the light in the wavelength range 350-1800 nm) is placed at the same distance from the head of the fibre optics, because if the distance between the sample and the sensor is not the same as the distance between the reference panel and the sensor, the measured reflectance values may be distorted.





We recorded 5 reflectance spectra from each compost sample, calculated from the average of 30 measurements, which was important to remove measurement uncertainties due to heterogeneity. The reflectance measurement were exported to Excel file in Avasoft software, and then saved the Excel file to text file. This text file were imported to ENVI 5.3 software, were we determined the inflection points of the compost treatment reflectance curve.

Statistical analysis

The inflection points of the reflectance curves measured for different mixtures were determined by binary encoding, where the software used converts the reflectance curve into a convex and concave curve. Binary cenoding is in fact a hyperspectral analysis method (Mazer et al., 1988). The inflection points were determined in ENVI (The Environment for Visualizing Images) Classic software environment. To determine the maturity-sensitive wavelengths of the reflectance



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curve, we select the wavelengths where the curve changes sign from the binary encoding database. Studies by Buiten (1993) show that during the composting process, inflection points shift from the red wavelength range (690–730 nm) to the near infrared range (700–1000 nm). The position of the inflection points can be used to infer, for example, the health of the plants, the chlorophyll content and the maturity of the compost.

To prove the correlations between the position of the inflection points and the measured physicochemical parameters, a Pearson correlation matrix was constructed using R software in the RStudio user environment (R Core Team, 2017) (p<0.05).

RESULTS AND DISCUSSION

Spectral characteristics of raw materials

Before setting up the composting experiment, the spectral characteristics of the used raw materials (broiler and hen manure, zeolite) were analysed in the 400–1000 nm wavelength range (*Figure 2*).

Figure 2: Spectral profiles of broiler manure, hen manure and zeolite in the 400–1000 nm wavelength range



The reflectance curves of the two types of manure have the same slope, no significant difference can be detected, but in both wavelength ranges the reflectance of hen manure is higher than that the broiler manure. This can be explained by the difference in organic matter content between the two raw materials, which could be measured by conventional analytical methods, since the organic matter content of broiler manure was 58.81%, while that of hen manure was 66.18%. In the 400-1000 nm wavelength range, spectral separation of broiler and hen manure is possible, but the wavelength range above 2000 nm may be suitable for determining mixing ratios. For soil organic matter, the absorption in the 780-2500 nm wavelength range is due to the properties of -OH, SO₄ and CO₃ groups, as well as water and CO₂, and their combinations (Clark, 1999). Absorption points in the 400-780 nm wavelength range for soils are mainly associated with iron-containing minerals such as hematite (Mortimore et al., 1984), which is not observed for manures.

Due to its greyish-white colour, zeolite has a high reflectance (above 55%), which can be observed up to 900 nm. In the VIS-NIR range, no decrease in reflectance is observed for zeolite, but above 1000 nm local minimum points are observed, which can be

explained by the crystal water content of calcium chloride, a sensitivity to moisture content.

Spectral properties of composts in the 400–1000 nm wavelength range

In the case of the control prism, it is observed that the reflectance of each sampling day shows a constant increase, with a significant overlap in the 400–700 nm wavelength range and a breakpoint in the 700 nm wavelength range (*Figure 3*).

In the near infrared range, reflectance curves are more distinct. The day 0. reflectance was the highest in the wavelength range studied for both control and zeolite-treated prisms.

The effect of zeolite resulted in a higher reflectance in the whole test range compared to the control already on day 0, which can be explained by the effect of zeolite on reflectance. Also for this treatment, the reflectance of the sample on day 0 was the highest, which can be explained by the immaturity of the compost (*Figure 4*).

The maturity of the compost justifies the decrease in reflectance over the range tested, i.e. as the colour of the compost darkens, the reflectance of the sample decreases.



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Figure 3: Changes of reflectance during 62 days of composting in the control windrow

Figure 4: Changes of reflectance during 62 days of composting in the 5% zeolite windrow



Location of inflection points in the 400–1000 nm wavelength range

In the 400–1000 nm wavelength range, the inflection points are located between 710 nm and 760 nm for each experimental setup. The change in the position of the inflection points also occurs between day 1 and day 8, the days on which the mixture has already entered the thermophilic phase. The 7% zeolite has the largest wavelength range where the inflection points are located between 712.918–749.614 nm for this treatment. The largest shift from the red to the near infrared range (39.696 nm) is observed for this treatment. These show that the higher the proportion of zeolite mixed in, the

more the inflection points are shifted towards the near infrared.

However, it can also be observed that the inflection point displacement increases with increasing zeolite percentage, but for the control, 1% and 2% zeolite, the inflection point position shows a decreasing trend by day 62 of composting, even for 5% and 7% zeolite, a further displacement of inflection points is observed, so for these two treatments I will not present the correlations further.

To demonstrate the correlation between inflection points and chemical properties measured from composts in the 400–1000 nm wavelength range, Pearson correlation was performed to determine the strength and direction of the relationship between the



different parameters and whether the relationship was significant at the p<0.05 significance level (*Table 2*). I considered it important to determine the correlations because spectrometric studies could only determine the

intrinsic and characteristic spectral properties of the material, in my case a mixture of two types of manure and zeolite, but correlation studies can often prove their relationship with other factors under investigation.

Table 2. Correlations	between	inflection	points and	measured	parameters
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Maanna daaraa kaas	Name of the compost prisms			
Measured parameters	Control	1% zeolite	2% zeolite	
Inflection points - pH	0.05	0.08	0.25	
Inflection points – Electrical conductivity	0.22	0.14	0.06	
Inflection points - Temperature	0.25*	0.28*	0.29*	
Inflection points – Moisture content	0.004	0.096	0.44	

*significant correlation at p<0.05

A weak correlation was found between pH, electrical conductivity and moisture content and inflection points. For pH and temperature, although a weak correlation was proved (R^2 value 0.14–0.25), the strength of the correlation increased with increasing zeolite mixing ratio. The correlation tests showed a weak positive correlation between inflection points and temperature, which was significant for all treatments and the control (p<0.05).

CONCLUSIONS

Based on our results, the spectral properties of the raw materials largely determine the reflectance evolution. For all compost prisms investigated, the reflectance of all sampling days shows a constant slope, with a significant overlap of the reflectance curves up to the 400–700 nm wavelength range, and a breakpoint in the 700–750 nm wavelength range.

The effect of zeolite also resulted in a higher reflectance (compared to the control) over the whole test range on day 0, which can be explained by the effect of zeolite on reflectance. The reflectance of the compost samples with zeolite was also highest on day 0. The high reflectance can be explained by the immaturity of the compost. As the composting process progresses, the compost darkens (due to humic substances), during which the reflectance was also lower, and therefore a tendency for lower reflectance of the samples at later times was observed. Also for the zeolite treatments, the lowest reflectance was observed for the sample of day 53. The maturity of the compost justifies the decrease in reflectance over the range tested, i.e. as the colour of the compost darkens, the reflectance of the sample decreases, which has been proved.

In the wavelength range 400-1000 nm, the inflection points of the reflectance curves were determined, which is of great importance for mixing studies. The aim of my investigations was to find out whether the amount of added zeolite in the broiler manure and hen manure mixtures can be detected, not only at the time of mixing and during the initial composting stage, but also during the composting and compost maturation process. In the 400-1000 nm wavelength range, the inflection points are located between 710 nm and 760 nm for each experimental setup. Compared to the control, the inflection points for 7% zeolite shifted from the red to the near infrared range. These results indicate that the higher the proportion of zeolite used, the more the inflection points shifted towards the near infrared region.

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