



# Non-invasive indicators in the poultry manure composting process

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ORIGINAL RESEARCH

## Abstract:

**Purpose:** The study presents how to develop and optimize a spectral measurement methodology for monitoring the composting process of broiler and hen manure using zeolite. Models were set up to determine the pH, electrical conductivity (EC,  $\text{dS m}^{-1}$ ), and moisture content (MC, w/w%) from reflectance data using spectral indices to establish a rapid, non-invasive, non-destructive method for the monitoring of the composting process.

**Method:** An open composting experiment was set up, in which a mixture of broiler and hen manure was composted with a zeolite additive (1 w/w%) for 62 days. During the experiment, samples were taken to determine MC, pH, and EC measured from a 10% distilled water extract. Principal component analysis (PCA) was performed for the indices to determine the wavelength ranges that could be used to test the material quality of the compost.

**Results:** Spectral index-based monitoring methods were developed. The  $\lambda_{2115}/\lambda_{1993}$  index-based estimation model with good performance (RMSE 2.42 m/m%) is suggested to measure the moisture content in composts. The electrical conductivity (RMSE 1.38  $\text{dS m}^{-1}$ ) and pH (RMSE 0.28 pH) are recommended to monitor with the  $\lambda_{812}/\lambda_{941}$  index-based models.

**Conclusion:** Based on the results, identified indices are suitable for determining the physico-chemical parameters of compost, which is of great importance for intensive, semi-intensive, and extensive composting plants, as spectral analyses can replace time-consuming analytical analyses.

**Keywords:** MC, EC and pH prediction indices; Poultry manure management; VIS-spectroscopy; NIR-spectroscopy

## 1. Introduction

Since the 1990s, the number of chickens bred worldwide has doubled. Poultry farming has worldwide importance in the food industry, currently providing the largest protein production in the world (Nalunga et al. 2021). According to FAO (2018), more than 7.2 billion broiler chickens are slaughtered for meat in the European Union each year, which amounts to approximately 12 million tonnes of chicken meat (AVEC 2018). The Hungarian poultry sector is also significant and characterized by a high level of self-sufficiency within the EU (145%), with a strong export orientation (Hungarian Central Statistical (Office 2022)). However, the intensification of the poultry industry generates a large amount of biodegradable waste and by-products

(Garg 2012), of which the amount of manure generated is significant. Energy recovery (e.g. biogas production) and material recovery (e.g. composting) are the most widely used methods for the treatment and recovery of organic wastes and by-products (Wang et al. 2019). Composting plays an important role in the management of waste and by-products, as it is becoming increasingly important for both sustainable energy management and a circular economy (Ayilara et al. 2020).

Composts are valuable products as a potential organic nutrient supply material (Hannet et al. 2021; Fernando and Arunakumara 2021). Furthermore, in accordance with the European Green Deal, there is an overarching aim in the European Union to reduce the utilization of chemicals in agriculture (COM/2019/640 2019). Composts and other

organic matter can be a viable alternative to chemical fertilizers, especially poultry manure-based composts, which have a lower environmental impact compared to artificial fertilizers (Kiss et al. 2021). Animal manure is essential not only from the plant nutrient supply aspect, but also because it raises soil pH, improves soil CEC and water holding capacity, and provides stable, sustainable crop production (Materchera 2010). However, the use of immature compost can cause rapid decomposition. It can have several adverse effects on nutrient-rich soils, such as reduced oxygen (Martin et al. 2020) and redox potential (Chikae et al. 2007), ammonia release (Tang et al. 2006), and significant phytotoxicity (Albuquerque et al. 2006). The most dangerous effect of using immature compost is the immobilization of nitrogen, polysaccharides, and nutrients in the soil by microorganisms (Bernal et al. 1998a; Bernal et al. 1998b). Therefore, compost maturity should be considered a crucial factor that affects the successful application of compost to agricultural fields, therefore monitoring composting is an essential task.

Some of the most important parameters in compost maturity monitoring are moisture content (MC), pH, and electrical conductivity (EC). Physical, chemical, and biological methods can be used to monitor and analyze the composting process and the finished compost (Francou et al. 2005). According to Bernal et al. (2009), the factors affecting composting can be divided into two groups: one group of factors depends on the feedstock mixture used, such as nutrient content (Tiquia 2003; Baddi et al. 2004; Grigatti et al. 2004), C/N ratio (Bertoldi et al. 1983), pH, particle size, porosity, EC and MC (Tiquia and Tam 1998); the other group of factors depends on process management, such as oxygen concentration (Tiquia et al. 1996), temperature (Flynn and Wood 1996; Rynk et al. 1992) and MC (Ekinici et al. 2006; Xi et al. 2007).

However, composting processes must be properly controlled and the gradual change in the physico-chemical properties of composts over time must be carefully monitored to ensure that the final product has optimal properties. Some compost maturity methods provide little information, some are inaccurate or inefficient, and some are expensive or time-consuming. Consequently, a more convenient and reliable method is needed to monitor and trace the composting process and to determine the maturity of the compost. In addition to classical (analytical) methods, spectroscopic and colorimetric methods play an important role in the analysis of different environmental samples (Wang et al. 2004). Non-destructive methods (UV, VIS, NIR, MIR, FTIR) are widely used in soil sciences, especially for the analysis of organic matter changes due to different agricultural activities, farming systems, and soil amendments (Haberhauer et al. 2000). However, several authors have demonstrated the suitability of non-destructive method for composting e.g. the determination of physical and chemical parameters in manure composting (Malley et al. 2005; Huang et al. 2008), C/N ratio in sewage sludge and green waste composting (Albrecht et al. 2008), and monitoring nitrogen content in poultry manure composting (Fujiwara and Murakami 2007). The prediction of various parameters measured from com-

post such as total organic carbon, total nitrogen, ammonium, nitrate, total phosphorus, pH, EC, and biological characteristics using VIS-NIR spectroscopy has been reported by several authors (Huang et al. 2007; Malley et al. 2005; Albrecht et al. 2008; Ludwig et al. 2006; Michel et al. 2006).

Non-destructive methods for composting are not well-studied, although rapid analytical methods are essential in composting plants and can provide a solution for monitoring the composting process as opposed to time-consuming and sample-preparation-intensive measurements. Huang et al. analyzed fresh and dried composted poultry manure samples in the wavelength range of 1000 – 2500 nm and determined the MC, pH, EC, total carbon content, C/N ratio, total phosphorus, and total nitrogen, Ca and Mg content of the samples by classical and NIR methods (Huang et al. 2007). Based on their results, the NIR method can accurately determine the MC, EC, total carbon, total nitrogen, and iron content of the samples tested. Albrecht et al. and Malley et al. have shown that total C, organic C, C/N, sulphur (S), potassium (P), and pH of compost correlated with the near-infrared (NIR, 700 – 1100 nm) spectra of dry matter (Albrecht et al. 2008; Malley et al. 2005). McMorrow et al. (2006) found a correlation between cellulose, lignin, and water content reduction during composting and shortwave infrared (SWIR, 1100 – 2500 nm) transmission. In addition, spectroscopic techniques have several advantages, such as rapidity, they are environmentally friendly methods, non-destructive, and allow a multitude of quality parameters to be detected simultaneously (Biyada et al. 2020; Rueda et al. 2023).

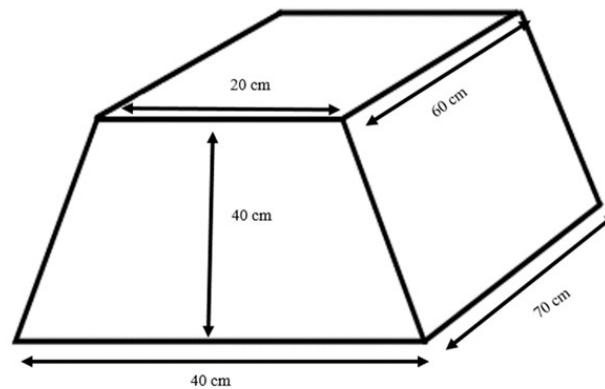
However, the spectral properties of the composts are also highly dependent on the origin. Therefore, the applicability of non-destructive methods and monitoring models for composting usually depends on the input materials and always requires further validation and adaptation of methods to a certain compost product.

Based on the above, the goal of this study was to develop and optimize a spectral methodology for monitoring the composting process of broiler and hen manure using zeolite. The aim was to set up models to determine monitoring parameters of composting (pH, EC, and MC) from reflectance data using spectral indices in the visible (VIS, 400 – 700 nm), near-infrared (NIR, 700 – 1100 nm), and shortwave infrared (SWIR, 1100 – 2500 nm) spectral regions to establish a rapid, non-invasive, non-destructive method for the monitoring of the composting process.

## 2. Material and methods

### 2.1 Structure of the model prism

A composting model experiment was set up to develop the monitoring method of the above parameters. The composting time was determined based on Karanja et al. and the model experiment resulted in 62 days of successful open prism composting (Karanja et al. 2019). The experimental prisms were the same size and consisted of the same 40 kg of a 1/3 : 2/3 mixture of broiler and hen manure (Fig. 1). The broiler and hen manure used in the composting experiments came from the largest poultry company in the north-eastern region of Hungary. The manure rates were



**Figure 1.** Dimensions of the compost prism.

the same that was used in the Company's composting plant. The composting materials were regularly rotated for optimal aeration (Jusoh et al. 2013). To broiler and hen manure mixture, 1 w/w% zeolite was added. Zeolite is a widely used additive for optimizing the composting process (Li and Burns 2006; Csiba 2012). Composting prisms were placed in a completely randomized arrangement in the composting area. Kiba et al. proved that there were no significant differences between the chemical properties (except for C, N, and Ca content) of deep-litter broiler and deep-litter hen manure (Kiba et al. 2020). However, our previous studies have shown that differences in microbiological properties between the two types of manure can be detected (Gorliczay et al. 2021). The characteristics of the manures and the additives used are presented in Table 1.

It can be concluded that deep manure management results in a high dry matter content (w/w%) of broiler manure and hen manure, as the dry matter content of manure decreases for broiler manure by the end of the 6-week rotation. The pH of the manures was slightly alkaline to neutral, while the EC and total nitrogen content (w/w%) were almost the same. The organic matter content-based on the loss on ignition (w/w%) was higher in hen manure (+7.37 w/w%) compared to broiler manure. The difference in housing technology also showed higher biological activity in hen manure. The zeolite used as an additive has a high dry matter content of over 90 w/w%, an alkaline pH (pH 8.49), and a low EC (0.65 dS m<sup>-1</sup>). It has no significant total nitrogen and or-

ganic matter content. The microbiological differences were the reason for using a mixture of the two types of manure for composting, as the deep litter hen manure is suitable as an inoculum to initiate the composting process.

Mineral composition of zeolite: clonoptilolite 43%, crys-tobalite 10%, quartz 10%, potassium feldspar 9%, mont-morillonite 7%, amorphous SiO<sub>2</sub> 5%, illite 3%. Its main constituents are silica tetrahedra, but aluminum atoms can sometimes replace silicon in the lattice points, so it is composed mainly of silicon (51.8%) and aluminum (4.8%), with sodium (0.1%), iron (0.7%), calcium (0.7%) and magnesium (0.3%).

## 2.2 Basic physicochemical analyses of compost

### Moisture content

The MC of the compost samples was determined by the gravimetric method before rotation. The MC was adjusted on day 0 of composting, then checked again on days 1 and 2 of composting, and from then on the MC was checked every three days, each time wetting the prisms to 60 w/w% moisture.

### Temperature

Temperatures were 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).

**Table 1.** Characteristics of the two types of manure and zeolite used as feedstock.

Parameters	Broiler manure	Hen manure	Zeolite
Dry matter content (w/w%)	65 – 70	63 – 67	92 – 94
pH (10 w/w% aqueous suspensions)	6.91	6.59	8.49
EC (dS m <sup>-1</sup> )	11.10	12.78	0.65
Total nitrogen content (w/w%)	2.75	2.14	0.08
Loss of ignition (w/w%)	58.81	66.18	1.16
Aerobic bacteria number (g <sup>-1</sup> )	10 <sup>5</sup>	10 <sup>8</sup>	-
Most Probable Number of spore bacteria	10 <sup>3</sup>	10 <sup>3</sup>	-

**Table 2.** Descriptive statistics of the measured compost parameters.

Samples	Parameters	Min.	1st Qu.	Median	Mean	3rd Qu.	Max.	St. Dev.	CV <sup>4</sup>
<b>Compost (Week 1)</b>	MC <sup>1</sup>	40.90	42.35	46.70	45.19	47.45	48.40	2.79	6.17
	T <sup>2</sup>	50.80	51.95	52.50	55.59	60.95	64.30	5.15	9.26
	pH	6.80	6.95	7.00	7.00	7.00	7.30	0.11	1.57
	EC <sup>3</sup>	5.84	5.90	5.92	5.93	5.95	6.11	0.06	1.01
<b>Compost (Week 9)</b>	MC <sup>1</sup>	35.60	38.70	40.40	43.87	50.80	51.90	7.02	16.00
	T <sup>2</sup>	11.60	12.10	12.70	14.67	17.70	18.40	3.057	20.79
	pH	7.30	7.56	7.80	7.76	7.98	8.02	0.20	2.57
	EC <sup>3</sup>	5.63	5.75	6.02	5.92	6.04	6.21	0.19	3.21

<sup>1</sup>Moisture content (w/w%)<sup>2</sup>Temperature (°C; in the windrow)<sup>3</sup>Electrical conductivity (dS m<sup>-1</sup>)<sup>4</sup>Coefficient of variation (%)

### pH and electrical conductivity (EC)

The pH and EC were measured by potentiometric and conductometric methods, respectively, from a 10 w/w% distilled aqueous suspension after 24 h of aging a shaker base, based on the studies of (Janczak et al. 2017; Irshad et al. 2013).

Three replicates were made for each parameter and the mean value was calculated.

### 2.3 Spectral measurements of compost

The compost samples were dried for mass stability according to the recommendations of Albrecht et al. (2008) because the MC of the samples in the reflectance curves can mask the real physicochemical characteristics and functional groups. AvaSpec 2048 spectrometer was used in the wavelength range 400 – 1000 nm, visible (VIS; 400 – 700 nm) and near-infrared (NIR-Near Infra Red, 700 – 1000 nm), with a resolution of 0.566 nm (Nagy et al. 2014). The AvaSpec-NIR256-2.5-HSC spectrometer was used for the 1000 – 2500 nm wavelength range measurements, with 6 nm spectral resolution. The spectrometers consist of three parts: a spectrometer (detector), an 8 μm diameter fiber optic, and a halogen light source. The tests were performed in a closed laboratory cabinet to ensure accurate measurements and eliminate electromagnetic radiation interference from

the fluorescent tubes and environment (Nagy et al. 2012). For both spectrometers, the reference panel WS-2 was used as a white reference.

Five reflectance spectra were recorded for each compost sample, calculated from the average of thirty measurements each, which was important to filter out measurement uncertainties due to heterogeneity (Riczu 2015; Bökfi et al. 2016).

### 2.4 Spectral model calibration and validation

A database was created from the spectral data, and PCA was performed by right-angle rotation with varimax rotation using SPSS 24 software. This procedure extracts the information from the original data matrix by reducing the dimensionality to a small number of principal components (PCs). PCA reveals those underlying features responsible for similarities and differences between the materials under study. The loading vectors or factor weight, corresponding to the PCs, provide information on the contribution of the spectral regions to the differentiation between samples. Here the loadings are combinations of the two regions (visible and near infrared) and can potentially result in a more comprehensive characterization of compost parameters. Indices were established based on spectral regions with high factor weights.

To evaluate the predictive models, the following indicators were selected: (1) the coefficient of determination (R<sup>2</sup>); (2)

**Table 3.** Correlation matrix of the measured parameters (p < 0.05).

	MC	T	pH	EC
MC <sup>1</sup>	1			
T <sup>2</sup>	0.27	1		
pH	-0.22	<b>-0.93*</b>	1	
EC <sup>3</sup>	<b>0.55*</b>	0.07	0.04	1

<sup>1</sup>Moisture content (w/w%)<sup>2</sup>Temperature (°C; in the windrow)<sup>3</sup>Electrical conductivity (dS m<sup>-1</sup>)

**Table 4.** Factor weights of five principal components related to physicochemical parameters in the VIS-NIR range.

	PC1 <sup>1</sup>	PC2 <sup>1</sup>	PC3 <sup>1</sup>	PC4 <sup>1</sup>	PC5 <sup>1</sup>
MC (w/w%) <sup>2</sup>	-0.1823	-0.2400	0.0423	<b>-0.5872</b>	0.0715
pH	-0.1792	-0.0862	-0.0567	<b>-0.8509</b>	0.1138
EC (dS m <sup>-1</sup> ) <sup>3</sup>	-0.1767	0.0605	-0.0102	<b>0.9280</b>	-0.0942

<sup>1</sup>PC1-5: Principal component 1-5<sup>2</sup>MC (w/w%): moisture content<sup>3</sup>EC (dS m<sup>-1</sup>): electrical conductivity

the root mean square error (RMSE); (3) the normalized root mean square error (NRMSE); (4) the mean relative error (MRE); (5) the mean error (ME) (Zuo and He 2021).

#### 1) Coefficient of determination (R<sup>2</sup>)

$$R^2 = 1 - \frac{\sum_{i=1}^n (y_i - \hat{y}_i)^2}{\sum_{i=1}^n (y_i - \bar{y})^2} \quad (1)$$

The root-mean-square error (RMSE) was calculated to test the reliability of the models or indices (Nagy et al. 2018).

#### 2) Root mean square error (RMSE)

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (y_i - \hat{y}_i)^2}{n}} \quad (2)$$

The normalized root-mean-square error (NRMSE) was also calculated. The normalization was necessary to make the different data and variables comparable.

#### 3) Normalized root mean square error (NRMSE)

$$NRMSE = \frac{RMSE}{(\bar{y})} \quad (3)$$

where  $y_{ii}$  is the predicted values,  $\hat{y}_i$  is the measured values,  $(\bar{y})$  is the average, and  $n$  is the number of elements.

RMSE and NRMSE provide validation options to determine the deviation of the estimated values from the measured values, and the additional strength of the method is that it analyzes the data in pairs (estimated-measured data pairs).

RMSE and NRMSE values were applied to the indices formed in the spectral studies to validate the applicability of the prediction model.

#### 4) Mean relative error (MRE)

$$MRE = \frac{1}{n} \sum_{i=1}^n \frac{|(y_i - \hat{y}_i)|}{y_i} * 100\% \quad (4)$$

#### 5) Mean error (ME)

$$ME = \frac{1}{n} \sum_{i=1}^n (y_i - \hat{y}_i) \quad (5)$$

The coefficient of variation (CV), which is the percentage of variance relative to the mean was used in performance analyses.

Statistical analyses were performed using R software in the RStudio user environment. All statistical tests were evaluated at the 95% confidence level.

## 3. Results and discussion

### 3.1 Composting process and compost characteristics

A summary of descriptive statistics for the observed compost parameters (MC, T, pH, EC) investigated in this study is presented in Table 2. Based on Wang et al.; Kalembasa et al., Becher and Pakuła, the optimum MC for most fungal substrates, sewage sludge, manures, and fermentation residues was between 40–60 w/w%, which was ensured during the nine weeks of composting (Wang et al. 2019;

**Table 5.** Prediction models in the NIR range and results of their validation.

Prediction model	R <sup>2</sup>	Regression Equation	RMSE <sup>1</sup>	NRMSE <sup>2</sup> %	MRE <sup>3</sup> %	ME <sup>4</sup>
Index 1 $\lambda_{812}/\lambda_{941}$ MC	0.3998	MC = $-65.79 * \lambda_{812}/\lambda_{941} + 88.93$	6.99	16.33	5.43	-0.078
Index 2 $\lambda_{812}/\lambda_{941}$ EC	0.1340	EC = $7.13 * \lambda_{812}/\lambda_{941} - 2.61$	1.38	18.07	17.88	0.0002
Index 3 $\lambda_{812}/\lambda_{941}$ pH	0.3467	pH = $-5.53 * \lambda_{812}/\lambda_{941} + 11.16$	0.28	3.93	3.07	-0.0004

<sup>1</sup>RMSE: root-mean-square error<sup>2</sup>NRMSE: normalized root-mean-square error<sup>3</sup>MRE: mean relative error<sup>4</sup>ME: mean error



**Table 6.** Factor weights of four principal components related to physicochemical parameters in the NIR range.

	PC1 <sup>1</sup>	PC2 <sup>1</sup>	PC3 <sup>1</sup>	PC4 <sup>1</sup>
MC (w/w%) <sup>2</sup>	-0.0680	<b>-0.4629</b>	-0.1479	-0.4213
pH	0.0495	-0.2874	0.0942	<b>-0.7965</b>
EC (dS m <sup>-1</sup> ) <sup>3</sup>	0.1570	-0.3035	-0.0094	<b>0.5187</b>

<sup>1</sup>PC1-4: Principal component 1-4<sup>2</sup>MC (w/w%): moisture content<sup>3</sup>EC (dS m<sup>-1</sup>): electrical conductivity

Kalembasa et al. 2012; Becher and Pakuła 2014). The MC determines the amount of oxygen available for microbial decomposition of organic matter and allows microorganisms to move from one place to another place (Matsushita et al. 2004). The MC ranged from 40.90 – 48.40 w/w%, with an average MC of 45.19 w/w%. By the ninth week (the last week of composting), the average MC had decreased minimally (43.87 w/w%). The compost prism was moistened at mixing, the MC was determined, and additional water was added if necessary to ensure an MC of around 60 w/w%. The zeolite (especially the dehydrated zeolite) can absorb large amounts of water, as the small grains have a high specific surface area (Soudejani et al. 2019), and for this reason, it was observed that even if the prism was always wetted to 60 w/w% MC, the MC was always below 60 w/w%.

Measurement of compost T is feasible by non-invasive methods, but to monitor the composting process and to identify the composting stages, the core temperature of the windrow was important. The T greatly influences the metabolism and population dynamics (composition and density) of microorganisms and microbes (Arslan et al. 2011). High T limits the activity of microbes, slowing down the decomposition of the organic matter, and T above 70°C increases the risk of spontaneous combustion and ammonia emissions (Ponsá et al. 2010). The core temperature of the prism under study was found to be independent of the ambient T (Avnimelech et al. 2004). The T evolution was closely related to the available humidity because when the humidity in the prism

was low, the T started to decrease slowly. After a rapid warming of the compost prism, the T remained high, as evidenced by the first week's minimum (50.80°C) and maximum (64.30°C) temperatures. By the ninth week of composting, the T had dropped significantly (average 14.97°C), had recovered to ambient temperatures, and demonstrated the composting stages well (Fig. S1).

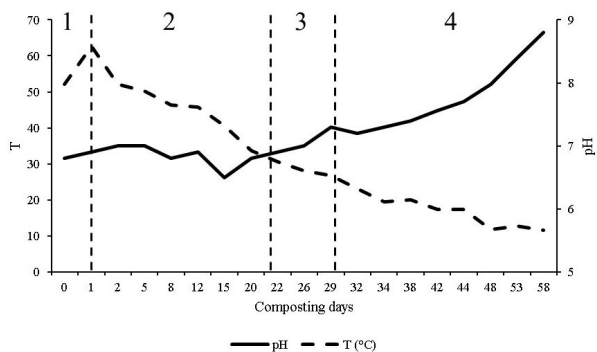
In the present study, composting was carried out with zeolite, but the pH of the tested prism ranged between pH 5 and 8.1. Chen et al. (2013) and Albuquerque et al. (2006) have shown that the optimal pH for the composting process was between pH 6 and 8, as both highly alkaline and acidic pH have a negative effect on biodegradation. In the case of pH, similar results to ours were reported by Irvan et al. (2018). They found that acidic pH conditions during the thermophilic stage of composting inhibit pathogen growth and that the frequency of mixing affects pH in such a way that the free space of carbon dioxide between compost particles was removed from the prism by mixing, preventing the accumulation of carbon dioxide from reducing pH. The pH values of the composts did not change significantly in the first and ninth weeks. When composting manure, the rising pH (pH > 8) releases large amounts of ammonia, which can lead to high nitrogen loss and odor, so it is important to use additives that affect the pH of the medium, e.g. wood shavings are acidifying, while zeolite is alkalizing. Zeolite keeps the medium in the neutral range during the nine weeks of composting.

During composting, the EC should decrease, but if aero-

**Table 7.** Prediction models in the NIR range and results of their validation.

Prediction model	R <sup>2</sup>	Regression Equation	RMSE <sup>1</sup>	NRMSE <sup>2</sup> %	MRE <sup>3</sup> %	ME <sup>4</sup>
Index 1 $\lambda_{2115}/\lambda_{1993}$ MC	0.4980	MC = 49.282 * $\lambda_{2115}/\lambda_{1993}$ - 6.9059	2.42	5.71	4.49	-0.0001
Index 2 $\lambda_{1922}/\lambda_{2127}$ pH	0.4009	pH = -4.1057 * $\lambda_{1922}/\lambda_{2127}$ + 11.156	0.28	3.89	20.66	0.0029
Index 3 $\lambda_{1922}/\lambda_{2127}$ EC	0.1360	EC = -9.4741 * $\lambda_{1922}/\lambda_{2127}$ + 16.481	1.38	18.11	2.88	0.0002

<sup>1</sup>RMSE: root-mean-square error<sup>2</sup>NRMSE: normalized root-mean-square error<sup>3</sup>MRE: mean relative error<sup>4</sup>ME: mean error

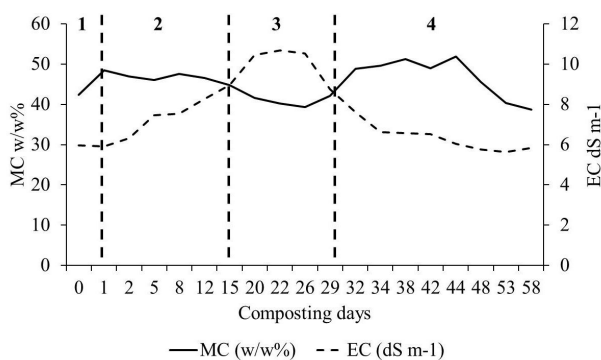


**Figure 2.** Evaluation of pH and temperature (T) in different stages of composting. (The numbers in the figure indicate the stages of composting. 1: Initial stage, 2: Thermophilic stage, 3: Mesophilic stage, 4: Maturing stage.)

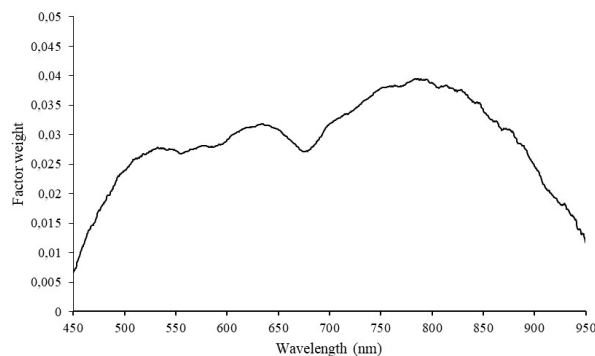
bic dominance was not archived in the prism, mineral salts and ammonium ions released from the decomposition of organic matter cause an increase in EC (Irvan et al. 2018). This is probably the reason for the slight increase in EC in the present study. The EC of the compost increased on days 0–12, whereas it increased between days 13–29 and started to decrease from day 40 (Fig. 1). With the addition of zeolite, the EC values decreased compared to the EC of the base material. By the ninth week of composting, the EC values of the compost ranged from 5.06 to 5.75 dS m<sup>-1</sup>.

Pearson correlation matrix ( $p < 0.05$ ) was used to explore the relationship between the chemical components measured in the composts. The relationship between pH, EC, T, and MC in the composts was evaluated (Table 3).

Based on the correlation matrix, in most cases, there were weak correlations and non-significant relationships between inorganic chemical parameters measured in composts. A negative significant correlation between pH and T was found ( $r = -0.93$ ) (Table 3). This correlation can be explained by the fact that the more electrolytic a medium or solution is, the better it is as a conductor. Thus, the higher the MC content in the compost prism, the higher the dissolved salinity will be.



**Figure 3.** Evaluation of moisture content (MC) and electrical conductivity (EC) in different stages of composting. (The numbers in the figure indicate the stages of composting. 1: Initial stage, 2: Thermophilic stage, 3: Mesophilic stage, 4: Maturing stage.)



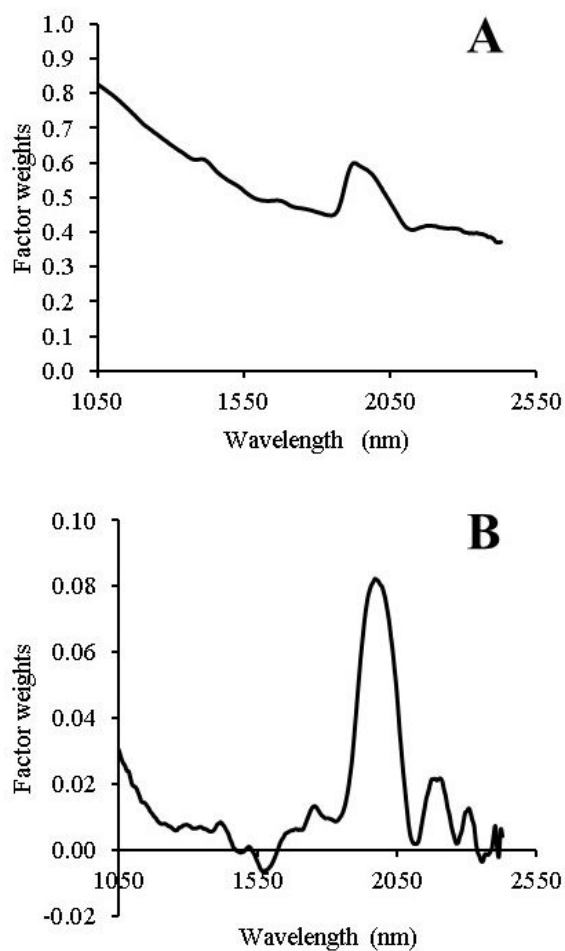
**Figure 4.** Factor weights for different wavelengths based on 4th principal component associated with the physicochemical parameters under study.

Based on T, the first 24–36 hours were the initial stage. Between days 2 and 15, the thermophilic phase is visible, when the T of the prism exceeded the ambient temperature reaching 64.5°C, as the maximum (Fig. 2). From days 16 to 37, the mesophilic phase is observed, during which the temperature gradually decreases, with the T in the prism ranging from 37.8 to 25.6°C. From days 38 to 40, the maturation phase is observed, during which the prism takes over the ambient T, and heat production gradually decreases.

The composting stages can also be applied to pH (Fig. 2). The pH was steady over the 58 days of composting, ranging from neutral to slightly alkaline throughout. There was a minimal decrease between days 0–21, probably due to the formation of organic acids, but it increased later in the composting process (stage 3) and was in the optimum range of 7–8 by day 58 (stage 4).

A positive significant correlation between EC and MC was found ( $r = 0.55$ ). The EC increased rapidly in stage 2, then reached a maximum in stage 3, and in the final stage decreased again reaching a constant value (Fig. 3). The reason for this can be the fact that with increasing T, biological activity and thus degradation increase. Organic acids are produced when organic matter decomposes, whether under aerobic or anaerobic conditions. On the other hand, the relationship can also be traced back to the ammonia/ammonium ratio. As the temperature increases, the pH increases, and so does the concentration of ammonium in the medium, as ammonium has a conductive property, acting as an acid (Emerson et al. 1975).

The physicochemical properties of the final compost produced from broiler and hen manure were investigated after the experiment. The compost produced at the end of the experiment had a neutral pH ( $7.76 \pm 0.2$ ), electrical conductivity of  $5.92 \pm 0.19$  dS m<sup>-1</sup> and dry matter content of  $56.13 \pm 7.02$  w/w%. In addition to these parameters, the total nitrogen content was determined by Kjeldahl method according to MSZ-08-1744-1:1998 Hungarian Standard and Bazrafshan et al. (2016), which was  $2.33 \pm 0.13$  w/w% in the compost produced. The organic nitrogen content was determined from the total nitrogen content by calculation. This is important because most of the nitrogen forms in stable, finished compost must be in organic form. While



**Figure 5.** Factor weights at different wavelengths based on the second principal component (Fig. 5 A) and the fourth principal component (Fig. 5B) associated with the physicochemical parameters under study.

$\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$  are immediately available to plants, organic nitrogen is only available slowly, with about 10% per year becoming available for uptake. The organic nitrogen content in the compost we produced was  $2.13 \pm 0.11$  w/w%. The organic matter content was determined from the loss on ignition according to the Hungarian Standard MSZ-08-0012-:1981, which was  $67.30 \pm 1.15$  w/w%. The evolution of the particle size composition was determined by sieving, which showed that 100% of the compost particles belonged to the particle fraction below 25 mm, while the particle fraction below 5 mm was 56 w/w%. Based on all these results, the physical and chemical properties of the compost produced by us comply with both the European Union (2019/1009 of the European Parliament and of the Council laying down rules on the making available on the market of EU fertilizing products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009 and repealing Regulation (EC) No 2003/2003)(2019/1009, n.d.) and Hungarian regulations (Decree of the Ministry of Agriculture and Rural Development No. 36/2006 (18. V.) (V.), n.d.) on the authorization, storage, marketing, and use of plant growth regulators).

### 3.2 Prediction indices in the 400 – 1000 nm wavelength range

Based on the results of the principal component analysis, five principal components were identified in the VIS-NIR range, with a 99.96% variance explained by the first two components. That component was selected, where the physicochemical parameters (MC, pH, EC) had the factor weights. In the 400 – 1000 nm range for the physicochemical parameters (MC, pH, EC), the factor weights in PC 4 were found to be the highest (Table 4).

The principal components were used to determine the component to which the physicochemical parameters under study (MC, pH, EC) belonged, and further worked with the factor weights belonging to the chosen component (Fig. 4). For the presentation of the factor weights, only the wavelength range between 450 – 950 nm was considered, as the factor weights in the 400 – 450 nm and 950 – 1000 nm ranges showed a large variance (similar to reflectance). The factor weights were used to determine the wavelengths where the factor weight was the largest and the smallest so that the variance between the two wavelengths was the largest. These wavelengths were 812 nm and 941 nm, from which the ratios  $\lambda_{812}/\lambda_{941}$  and  $\lambda_{941}/\lambda_{812}$  were calculated. Based on these data, prediction indices were developed to predict the physicochemical parameters studied, which are shown in the table below (Table 5).

Prediction models with NRMSE > 20% cannot be used with sufficient efficiency to predict the parameter. Based on these results, of the indices created in the 400 – 1000 nm wavelength range, Index 3 is the most suitable for predicting pH, as the  $R^2$  calculated from the ratio of pH to  $\lambda_{812}/\lambda_{941}$  wavelengths was 0.3467, RMSE was 0.28, and NRMSE was 3.93%. The accuracy of the model pH is 0.28, i.e. the difference between the measured and estimated value is 3.93%. Index 1 also gave good results in estimating the MC (NRMSE < 20%), as the coefficient of determination between  $\lambda_{812}/\lambda_{941}$  and MC was 0.3998, the measurement error (RMSE) was 6.99 m/m%, and the normalized RMSE was 16.33%.

The MRE values were calculated to test the reliability of the prediction models. According to Bertsch et al. (2008), predictive models with MRE values below 30% can be used to predict a parameter. Based on these results, the MRE values gave reliable results for each of the parameters investigated (MC: 5.43%, EC: 17.48%, pH: 3.07%). However, the lowest values were obtained for MC and pH, and thus the MRE values were used to demonstrate the accuracy of the prediction of these parameters.

The ME usually refers to the average of all errors in a set, defined as over or underestimation. For this validation procedure, the highest value was obtained for MC (-0.078), but for pH and EC very minimal differences between the estimated and measured values were detected (EC: 0.0002, pH: -0.0004).

As for the prediction indices of the VIS-NIR range, the models developed are suitable for the prediction of pH, MC, and EC, and thus the physicochemical parameters of compost can be determined by spectral (fast analytical) analyses. Similar research to ours has been carried out



by Sharma et al. (2005): they have developed prediction models to estimate pH and EC evolution in the 400 – 2500 nm wavelength range. Their results show that in the 400 – 1100 nm wavelength range, unlike ours, they have not been able to produce prediction indices.

### 3.3 Prediction indices in the 1000 – 2500 nm wavelength range

As with the analysis in the VIS-NIR range, a principal component analysis was performed in the NIR range, with a 96.89% variance explained by the first two components.

That component was selected, where the physicochemical parameters (MC, pH, EC) had the factor weights. In the 1000 – 2500 nm range, the factor weight in PC 2 was found to be the highest for MC, and the factor weights were found to be the highest for pH, and EC in PC 4 (Table 6). Therefore the data of PC 2 and 4 were used to identify MC, pH, and EC sensitive spectral regions (Fig. 5).

Based on the factor weights, the  $\lambda_{2115}/\lambda_{1993}$  index was established to predict MC, since the spectral feature at 1993 nm showed the highest factor weight suggesting high sensitivity for MC, whilst reflectance at 2115 nm possessed less variability in spectral feature relating to the MC. For pH and EC, the spectral feature at 1922 nm showed the strongest sensitivity with the minimum at 2127 nm, therefore, the  $\lambda_{1922}/\lambda_{2127}$  index was found to be promising for developing the prediction of pH and EC (Table 7).

In the NIR range (1000 – 2500 nm), much more reliable results were obtained compared to the VIS-NIR (400 – 1000 nm) range. The smallest correlation coefficient value was calculated for the EC ( $R^2 = 0.1360$ ) and for this model, the NRMSE between the predicted value and the measured value was 18.11% (medium strength), which is the difference between the model and the measured value, even though the RMSE was  $1.38 \text{ dS m}^{-1}$ . However, the indices calculated for MC and pH may be suitable for estimation. The  $R^2$  value between MC and the  $\lambda_{2115}/\lambda_{1993}$  ratio was 0.4980, indicating a positive, medium-strong correlation between the two parameters. The RMSE (2.42 w/w%) and NRMSE (5.71%) also suggest that the model can be used reliably to estimate MC. However, the best values were calculated between pH and the  $\lambda_{1922}/\lambda_{2127}$  ratio, as the root mean square error of measurement (RMSE) was 0.28, yet the normalized RMSE was 3.89%.

As in the VIS-NIR range, the MRE values were below 30% for all three parameters tested in the NIR range. The highest MRE value was obtained for pH (20.66%). These results indicate that the reliability of the prediction indices calculated for pH was low. In contrast, for MC and EC low values were calculated similarly to the VIS-NIR range (MC: 4.49%, EC: 2.88%). This also demonstrates that MC and EC can be reliably estimated from the prediction indices. For ME, low values were obtained, which supports the accuracy of the prediction of the four parameters under study. ME was -0.0001 for MC, 0.0029 for pH, and 0.0002 for EC.

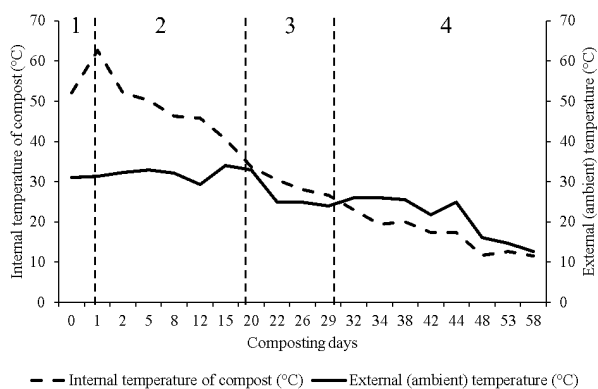
Based on these results, among the prediction indices calculated in the NIR range (1000 – 2500 nm), the MC, pH, and EC models can be used to predict the parameters. However,

the non-destructive analysis of composts has its difficulties: on the one hand, the input materials determine the applicability of spectroscopic methods and have a significant influence on the spectral curve (Chen et al. 2013). On the other hand, in most cases, only the physicochemical properties of compost of a certain composition or from a certain composting system can be determined. In the live-stock farm, rapid monitoring of the composting process of a mixture of broiler and hen manure was not sufficiently available, but with the indices developed in this study, rapid non-destructive tests for this type of compost are possible (Feng et al. 2022). However, several other studies are declaring that the VIS-NIR spectral range was suitable for pH, EC, and MC monitoring using other indices or estimation methods. Hong et al. measured soil samples and identified two wavelengths sensitive to moisture content (1360 and 1940 nm) and derived a soil moisture index (SMI) from these wavelengths ( $R^2$  0.9) (Hong et al. 2017). In contrast, the values were much lower, as the best  $R^2$  results (0.39 and 0.49) were obtained when estimating the MC. These differences come from the compost was made of complex organic materials, while soil also includes elements that are not organic compounds, such as rock particles or minerals. Based on these results, a lower  $R^2$  value gives reliable results for composts. On the other hand, Malley et al. (2005) took measurements at the cattle manure composting process and investigated prediction indices in the 360 – 1690 nm wavelength range and found a prediction certainty of 0.9  $R^2$  for pH. Reeves and Kessel (2002) worked with cattle manure and took measurements in the 400 – 2500 nm wavelength range and the calculated  $R^2$  for MC was 0.95, and for pH, the  $R^2$  value was 0.66. For pH and EC, measurements were made in the wavelength range of 1000 – 2500 nm by Huang et al. (2007) with the mix of cattle, pig, and chicken manure compost and the accuracy of their estimate was 0.57  $R^2$  for pH and 0.87  $R^2$  for EC. Based on their results, MC and EC can be accurately determined by the NIR method. Based on Reeves's results with poultry manure measurements, the MC can be estimated with high confidence in the VIS and NIR wavelength range, as the precision of the estimate (RMSE) was 4.44 w/w%, yet the  $R^2$  value was 0.84 (Reeves 2001), and this is confirmed by the results of Hong et al. (2017), as the  $R^2$  value for estimating MC was 0.9.

Infrared spectroscopy is a suitable analytical tool for the analysis and estimation of composting parameters in waste and by-product management, in particular for monitoring the composting process and determining the quality of compost. It is important to find methods to achieve circular farming and to use the products from livestock manure produced on the farm as organic nutrient supplements, thus keeping the by-products of agricultural activities on the farm.

## 4. Conclusion

The starting point of this research was to develop a non-invasive estimation method to determine the key compost quality parameters (pH, EC, MC) of (broiler and hen manure-based) composts by rapid analytical



**Figure S1.** Non-invasive indicators in poultry manure composting process.

methods and estimating equations, replacing the more time-consuming tests in an intensive composting plant. In summary, the use of spectroscopic data analysis as a tool to identify the physicochemical properties of compost was found to be appropriate. Using the method of principal component analyses to identify wavelengths with high prediction potential pH, EC and MC spectral indices were formed to monitor these parameters of the composting process. In this study, (i) pH was found to be monitored by using the  $\lambda_{19922}/\lambda_{2127}$  index (ii) the best performing MC estimation model using  $\lambda_{2115}/\lambda_{1993}$  index (iii) EC can be measured by  $\lambda_{812}/\lambda_{941}$  spectra index. The approach proposed in this study provides a simple and efficient (in terms of time and human resources) method to determine the maturity and stability of compost in a composting site.

#### Supplementary material:

Fig. S1. Evaluation of the internal temperature of the compost prism and the external (ambient) temperature in different stages of composting. (The numbers in the figure indicate the stages of composting: 1: Initial stage, 2: Thermophilic stage, 3: Mesophilic stage, 4: Maturing stage.)

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#### Authors' contribution:

The authors confirm the study conception and design: E.G., A.N., J.T.; data collection: E.G., A.N.; analysis and interpretation of results: E.G., A.N.; draft manuscript preparation: E.G., A.N., J.T. The results were evaluated by all authors, and the final version of the manuscript was approved.

#### Compliance with ethical standards

**Table 8.** Abbreviations.

Abbreviation	Meaning
CV	coefficient of variation
EC ( $\text{dS m}^{-1}$ )	electrical conductivity
MC (w/w%)	moisture content
ME	mean error
MRE	mean relative error
NIR	near infrared spectrum (800 – 2500 nm)
NRMSE	normalized root mean square error
PCA	principle component analysis
$R^2$	determination coefficient
RMSE	root mean square error
T ( $^{\circ}\text{C}$ )	temperature
VIS	visible wavelength spectrum (400 – 800 nm)

#### Conflict of interest:

The authors declare that there are no conflicts of interest associated with this study.

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