Review

Application of Near-Infrared Spectroscopy for Monitoring and/or Control of Composting Processes

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Abstract: The implementation of a suitable and ecologically friendly solid waste management plan is accepted as an essential need. Given that organic matter constitutes the majority of solid waste, composting has gained popularity as an alternative way of organic refuse recycling. Compost quality is defined by its stability and maturity, both of which must be assessed by measuring a large number of physical–chemical parameters, microbiological variables, and enzymatic activities. These procedures are complex and time-consuming, making it difficult to assess compost quality correctly. Spectroscopy methods could be used as an efficient alternative. In this work, general information about composting processes and near-infrared spectroscopy (NIRS) is given. A discussion and comparison of the different approaches of coupling NIRS and chemometric tools for the monitoring and/or control of composting processes are presented in this work.

Keywords: composting process; near-infrared spectroscopy; chemometric methods

1. Introduction

One of the twenty-first century's issues is increasing agricultural output while improving food safety and quality. The food sector must feed a rising population while simultaneously caring for the environment and maximizing natural resources in each location. Subsequently, there is also growing interest in solid waste management with minimum environmental impact. According to the literature data, composting is considered a low cost, efficient, and long-term treatment for solid waste. The importance of composting as a waste management method can be seen from the growing numbers of publications published in last 20 years (Figure 1). Due to their potential importance, composts can be used for agricultural reasons. Composts' organic content may actually enhance soil structure and lower the danger of erosion. Composts can also be useful fertilizers. They are an essential source of organic phosphorus and nitrogen, and they improve soil's cation exchange capacity. Based on the abovementioned, there is also a growing demand for the development of analytical methods to monitor the composting process and form assessments of compost quality and maturity. Traditional approaches for analyzing such materials are being phased out in favor of spectroscopic techniques, one of which is near-infrared spectroscopy (NIRS) (Figure 1). This technique has several advantages over traditional methods. The main advantages are as follows: (i) it is a nondestructive method of analysis; (ii) it does not pollute the environment (it does not use chemical reagents); (iii) it is a cheap and fast method; (iv) it measures several parameters comparatively; and (v) it enables on-site analysis (and online connection) for a large number of samples per minute.
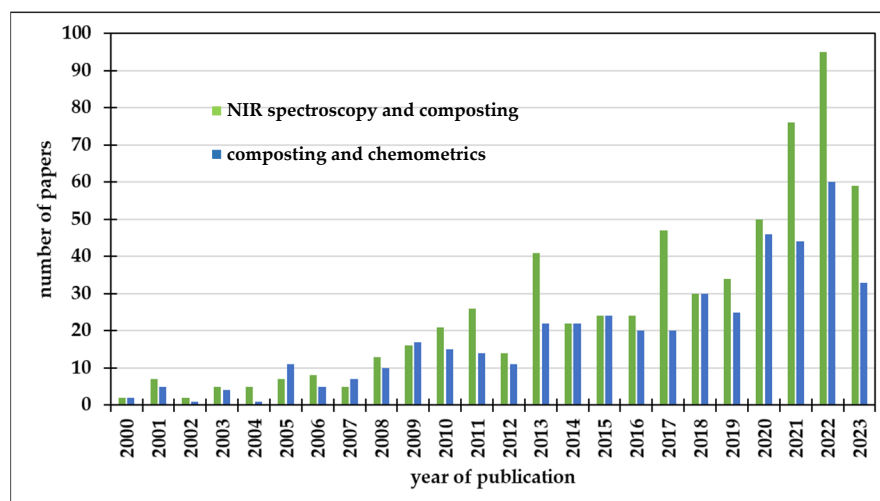
Figure 1. Number of publications according to Web of Science (WoS) in fields of (i) NIR spectroscopy and composting and (ii) composting and chemometrics.

Considering the importance of spectroscopy methods in composting process analysis, in this work, the application of NIR spectroscopy for the monitoring of composting processes has been revised. The motivation for this research was a lack of review papers presenting information on composting control and monitoring using NIR spectroscopy. This review includes some general information on composting technologies and some general information on NIR spectroscopy, followed by a comparison and discussion of specific examples of using NIR spectroscopy for composting process monitoring and for compost quality assessment. Furthermore, current challenges and future perspectives regarding composting and online measurements are also discussed.
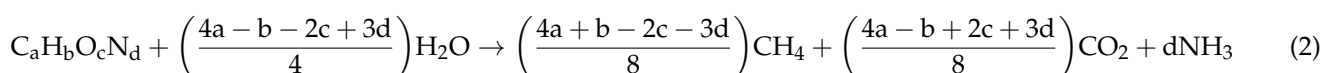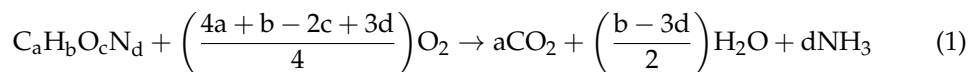
2. Literature Search Overview

A comprehensive systematic review of the important scientific articles was conducted using the core collection in the Web of Science database for the period of the last 23 years (period from 2000 to 2023). The keywords "composting" and "NIR spectroscopy" and "composting" and "chemometrics" were used to search the titles and abstracts of the articles. Only indexed papers (research and review papers) were selected for further analysis. For the field of NIR spectroscopy and composting, there were 315 articles; for the field of composting and chemometrics, there were 222 articles. Literature search was not focused on specific composting substrates but on potential application of spectroscopic techniques for monitoring and control of composting process.

3. Composting: General Information

The growth of the world's population, especially in developed countries in the last fifty years, has had an exponential effect on the amount of produced solid waste [1–3]. Sustainable waste management of any kind, including food waste, is seen as an approach in which the primary goal is to reduce waste in any part of the production cycle and, if possible, recycle and/or separate those components that are still usable in a sustainable way as possible and thus, reduce the related impacts, starting from resources, production, sale, consumption to use, and final disposal—thus covering the entire life cycle [4,5], which encourages the professional and scientific community to develop and implement new, greener strategies in solid waste management as soon as possible [6]. This increase in waste generation has put a pressure on the various components of the environmental system. As a result, the introduction of a suitable and environmentally favorable solid waste disposal plan has been identified as a vital requirement [6]. Considering that a major section of compact waste is organic matter [7], composting has attracted attention as an alternative method of organic waste recycling. Composting can divert waste from

landfill, mitigate groundwater contamination, reduce air pollution and greenhouse gas emissions, and generate usable goods [8]. As described by Meena et al. [9], composting can be categorized into two types according to the characteristics of the microorganisms responsible for the decomposition of organic wastes, namely aerobic (Equation (1)) and anaerobic (Equation (2)) [10]:

$$C_aH_bO_cN_d + \left(\frac{4a + b - 2c + 3d}{4}\right)O_2 \rightarrow aCO_2 + \left(\frac{b - 3d}{2}\right)H_2O + dNH_3 \tag{1}$$

$$C_aH_bO_cN_d + \left(\frac{4a - b - 2c + 3d}{4}\right)H_2O \rightarrow \left(\frac{4a + b - 2c - 3d}{8}\right)CH_4 + \left(\frac{4a - b + 2c + 3d}{8}\right)CO_2 + dNH_3 \tag{2}$$

As a number of studies indicate, the creation of the main byproducts (thermal energy, H_2O, and CO_2) is the result of the aerobic process of the biodegradation of organic matter [1,11] (Figure 2). In addition to the aerobic effect, during the decomposition of organic waste, there is also an anaerobic segment, and as stated in the term itself, it takes place without the presence of oxygen, so microorganisms that work in such conditions (anaerobic microorganisms, i.e., anaerobes) dominate the process of biogas production (e.g., methane) but also a strong-smelling byproduct called digestate (slurry mixture) [12,13].
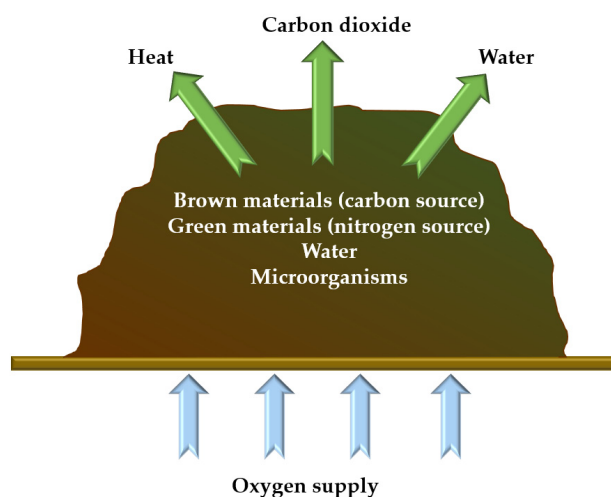

Figure 2. General scheme of aerobic composting process (adapted from Meena et al. [9]).

Although considered a robust technology with numerous advantages, there are still some challenges when performing composting. According to Cerda et al. [14], challenges in composting can be assigned to (i) composting material composition, (ii) odors produced during composting, (iii) process variable monitoring (temperature, oxygen supply, moisture content, pH, C/N ratio, and particle size), and (iv) composting mixture condition. During the controlled process of composting under aerobic conditions, in addition to aeration, it is necessary to control the following extremely important parameters: (i) temperature, (ii) humidity, (iii) degree of acidity, and (iv) the ratio of carbon and nitrogen. A prerequisite for good quality products during composting is the control of the aforementioned conditions [6]. An indication of microbial activities during the composting process is the change in temperature, which is justifiably considered a parameter that indicates the status of the process [15–17]. When the favorable conditions for the implementation of the composting process have been achieved, it will take place in several main stages, which we can follow according to the microorganisms that prevail in a certain stage [6]. The first phase is the so-called mesophilic, and it is dominated by fungi and bacteria that start with the decomposition of easily degradable macronutrients (proteins and simpler carbohydrates, such as sugars), and due to the microbial activity, the temperature rises evenly (25 to 45 °C), and the heat increases, which enables the transition to the next, thermophilic phase [6]. The thermophilic temperatures (above 55 °C) are desirable because they kill pathogens [18].

During the thermophilic phase, high-energy compounds (sugars and proteins) are broken down, and the supplies are depleted, which results in a decrease in temperature and the redominance of mesophilic microorganisms. The last phase is characterized by the humification of the material and presents the maturation phase [6,18].

Moisture is a variable closely related to microorganisms and should be in appropriate amounts through the composting cycle [19]. The most favorable moisture content at the beginning of the composting process depends on the physical state and size of the particles of the material. A moisture content around 50–60% should be satisfactory [13]. A low moisture content (less than 30%) can lead to dehydration of the compost, while excessive moisture content will limit the movement of oxygen; thus, anaerobic conditions will reduce the rate of composting [18,20].

The most favorable pH range for the composting process is between 5.5 and 8.0. Bacteria favor practically neutral pH, and fungi develop in acidic environments [21,22]. In the first stage of composting, the pH may decrease due to the organic acids which are produced during the degradation of organic compounds. Further mineralization leads to an increase in pH, and at the end of the process, it is around 8.0–8.5 [19,22]. Food waste can present a challenge for applying the composting method due to low pH, which is a consequence of the presence of short-chain organic acids. Therefore, several studies have investigated the use of alkaline materials (sodium acetate, sodium hydroxide) as pH control amendments in order to provide an improvement in the composting process [23].

The ratio of carbon to nitrogen (C/N ratio) indicates the degree of decomposition of organic matter and its identification; an optimal ratio is of great significance to obtain favorable composting results and highly depends on the properties of the composting material [8]. The ultimate for the active composting of most types of wastes is 25–30:1, and during the composting process, it decreases constantly due to mineralization of the carbon compounds [24,25]. Aeration is important for microbial growth and gas emissions [8]. Composting is an aerobic process where oxygen is consumed, and carbon dioxide is released [18,26]. Studies show that an optimal level of aeration will provide enough oxygen that is needed to evaporate excess moisture from the substrate due to the oxidation of organic material [27], while inappropriate aeration can lead to anaerobic conditions (in the case of insufficient aeration) or can lead to cooling and prevent the thermophilic conditions necessary for optimal decomposition rates (with excessive aeration) [28].

A well-executed composting process results in compost that is suitable for improving soil characteristics and will enable increased infiltration and water retention, and its extremely sustainable feature is that it will also provide a number of nutrients necessary for plant growth [29]. Ensuring microbial diversity is considered a key factor for healthy soil as it enables the implementation of biological processes and the circulation of nutrients, where enzymatic activities also have an important role in biochemical reactions. Enzymatic activities can be enhanced using organic fertilizers [6]. The final use of compost defines which requirements will be set for the quality of the compost. Compost used in the agricultural sector must not have a dominant smell, the amount of nutrients must be known, and it must contain only trace amounts of impurities (homogeneous and disinfected). When compost is used as a growing medium, some physical properties that will affect water availability to plants (e.g., particle size, density, and water-holding capacity) become extremely important [30].

The composting process can be performed in different types of facilities: open systems, such as windrows or piles (either aerated, static, or turned) and closed systems, such as reactors or composters [31] (Figure 3). Closed (in-vessel) systems have advantages over open systems: they require less space and provide better control of the process, and they involve high process efficiency [32–34]. For example, Pinto et al. [20] investigated different grape pomace composting treatments during 140 days: two of them composted piles by turning the pomace after some period, and one was a static pile. In their work, all treatments achieved the thermophilic phase (the temperature was above 45 °C) at the 2nd composting day, and those conditions continued for almost 50 days through the composting. Aeration

was achieved by turning the piles, increasing oxygen levels, and improving microorganism activity; consequently, the temperature increased and composting progressed faster. According to the rates of organic matter mineralization, the highest was in the static pile compared to the turned ones because the thermophile phase lasted longer without any temperature decrease [20]. Moreover, the composting process of poultry manure and sawdust in closed cylindrical and rectangular reactors was performed by Qasim et al. [35]. They investigated the effect of different aeration rates on the performance of the composting process. Their results showed that low aeration rates correspond to higher temperatures and a longer thermophilic phase. Furthermore, the literature reports many studies on vermicomposting of different types of organic waste [36–38]. Vermicomposting is defined as utilizing several earthworm species for composting organic matter [1]. Fertilizer rich in nutrients can be created from organic solid waste, and this requires aerobic conditions and earthworms. One species of earthworm (*Eisenia fetida*) has the potential to convert organic waste into products of a high nutritional value, but the environmental conditions (such as pH, temperature, and humidity) must be suitable [39].
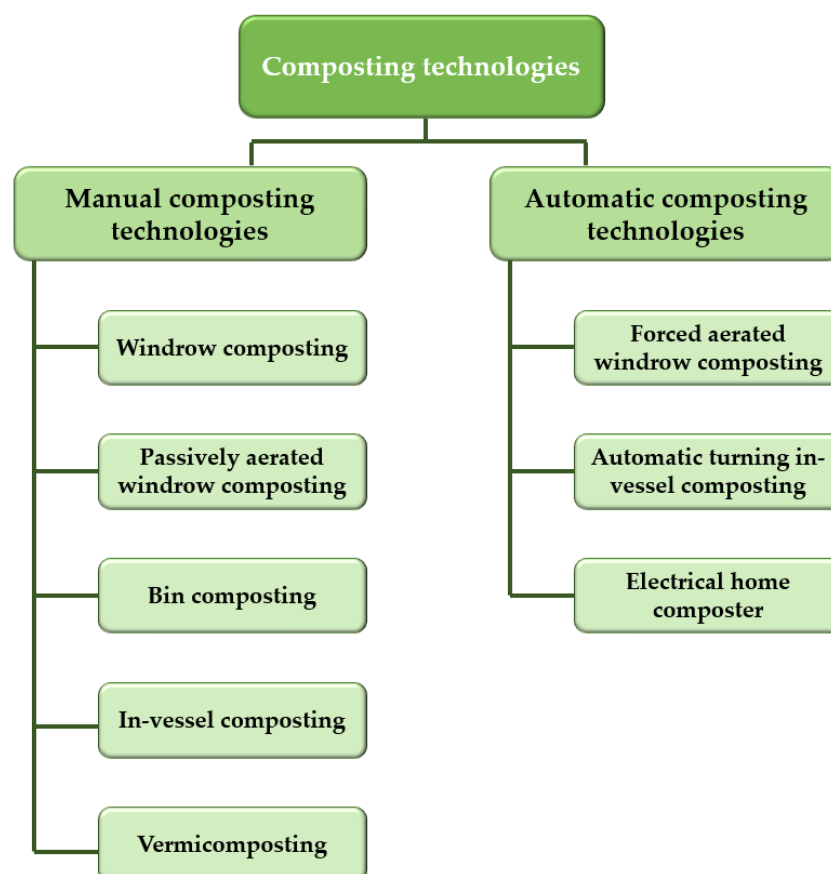
Figure 3. Composting technologies (adapted from Miguel et al. [31]).

4. Near-infrared Spectroscopy

Near-infrared (NIR) spectroscopy is a commonly used spectroscopic technique dealing with the absorption, emission, and reflection of electromagnetic radiation in a wavelength range from 800 to 2500 nm [40]. It comprises both electronic and vibrational NIR spectroscopy. Furthermore, the NIR wavelength range has been divided into three regions: the first one encompasses the wavelength range from 800 to 1200 nm, forming a visible near-infrared region. Characteristics of this region include high permeability, enabling its application in medicine and agricultural industries. It is also specific due to the appearance of bands, which are consequences of electronic transitions, higher-order overtones, and combinations of fundamental vibrations of the XH bonds (X = C, N, O, S) [41–43]. The

second region includes the wavelength range from 1200 to 2000 nm, containing a number of bands arising from the first and second overtones and combination modes. This wavelength range can be used for qualitative and quantitative analyses, but the permeability of the second region is low. The third region (2000–2500 nm) deals with the combination modes and can be used for various purposes, such as investigations about the structure of proteins; however, the third region is characterized by relatively low permeability [40,44].

Due to fact that NIR spectra are often complex and possess broad overlapping absorption bands, the extraction of useful information from the NIR data set requires the use of chemometrics. Chemometric tools are used for defining the relationship between spectral information and sample properties [41,45,46]. Data preprocessing represents the first chemometric step for reliable NIR spectra analysis. Algorithms, such as baseline correction (BL), multiplicative scatter corrections (MSCs), standard normal variate (SNV), Savitzky–Golay (SG), and smoothing (SMTH), help to remove useless information, minimizing deviations caused by different measurement conditions (e.g., light, instrumental drift, light scattering, interferences, etc.), all in order to achieve the quality of spectral data [47,48]. The high-dimensionality of NIR spectral data needs to be reduced in order to prepare them for classification analyses [49]. Principal component analysis (PCA) is the most widespread applied multivariate method for data exploration and reduction, with the aim to find potential likenesses and/or disparities within observations and identify clusters or outlines [50]. Furthermore, PCA converts NIR spectra into variables that are not linearly correlated and enables meaningful patterns to be described or predicted from complex spectral prints. PCA is also very useful for the determination of important wavelengths that could contribute to further sample discrimination [48]. In addition to PCA as an exploratory technique, NIR spectra can also be modeled using partial least squares regression (PLS), partial least squares discriminant analysis (PLS-DA), linear discriminant analysis (LDA), and soft independent modeling using class analogy (SIMCA) (Figure 4). These classification algorithms are commonly utilized for classification or sample grouping into clusters according to their similarities or common spectral characteristics [48–52].
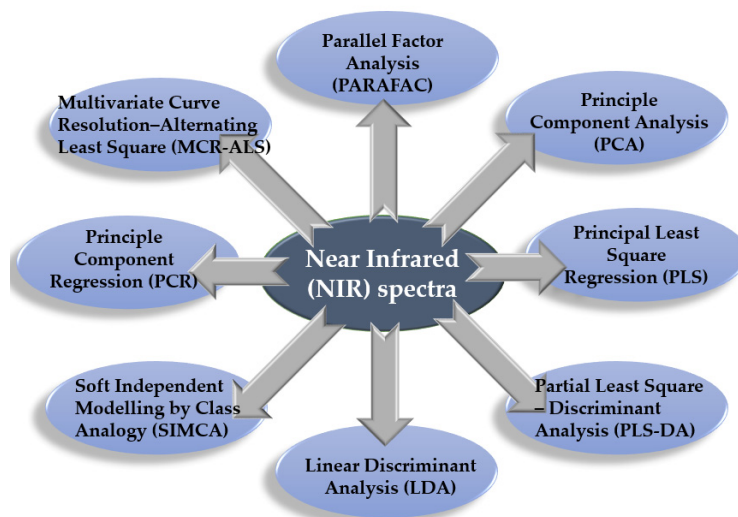
Figure 4. Chemometric methods for NIR spectra analysis (adapted from Varmuza et al. [46]).

The quantitative characterization of specific compounds in samples can be performed by applying chemometric regression methods. Regression methods analyze the relationship between the NIR spectra (as raw, preprocessed, or as a set of particular wavelengths) and the physical/chemical features of the investigated samples [48,53]. Partial least squares regression (PLS), principle component regression (PCR), multivariate curve resolution–alternating least squares (MCR-ALS), or parallel factor analysis (PARAFAC) are models applied for quantitative purposes, assuming linear relationship between predictors (independent variables) and predicted (dependent) variables (Figure 4). However, due to

the appearance of multicollinearity problems, nonlinearity of the data can occur. Multicollinearity includes a number of predictors that is greater than the number of samples, which makes the covariance matrix singular and noninvertible; subsequently, finding a unique solution is impossible from least squares [54]. The alternative is to use nonlinear models, such as artificial neural networks (ANNs). The application of ANNs started in the 1980s, along with development of computational techniques based on self-organizing properties and parallel information systems [55]. Due to their capability to solve real problems, ANNs have been extensively used in areas such as engineering [56,57], biology [57], and agriculture [58]. ANNs mimic the data processing of the human brain. The relationship between the independent vs. dependent variables (inputs vs. outputs) is nonlinear, and this relationship is reflected by the neuronal linking network matrix of the network. Therefore, it is crucial in the development of ANNs to ensure consistent and significant experimental records for (i) training, (ii) testing, and (ii) validating the strength of the model [58].

A wide range of NIRS applications led to the development and constant improvement of NIRS devices' performance, which is confirmed by the increase in the number of published studies on this topic (Figure 1). In general, any NIR instrument includes (i) a source of radiation, (ii) a device for the interaction of the sample and radiation source, (iii) a wavelength selector, (iv) a detector, (v) a device for data collection, processing, and storage, as well as (vi) a device for controlling the instrument [59,60]. The radiation source is a quartz bulb containing traces of iodine with a tungsten filament, with power in the range of 25–100 W and operating temperatures from 2000 to 3000 K, respectively. A queue of light-emitting diodes (LEDs) is used for vigorous NIR radiation sources in small instruments. The sample–radiation interaction device promotes interaction between monochromatic or polychromatic NIR radiation and the sample, obtaining spectral information about a particular sample as a function of its position with respect to the wavelength selector [59]. The frequency (wavelength) domain contains the spectral information utilized for qualitative and quantitative reasons. The key feature of the selection device for NIR wavelength separation is its resolution. It denotes the capacity to distinguish between two relatively near wavelengths. This feature is determined by the bandwidth of the peaks in the samples under consideration. The detector is a device used to measure the intensity of radiation. The optical and electrical characteristics of semiconductors are determined by their energy bandgap. A disadvantage of PbS and PbSe detectors is the nonlinearity of their response. However, they exhibit good detectivity over the entire NIR range, even beyond the typical NIR spectral range. The instrument controller, user interface, and data acquisition/storage device is a microcomputer or microprocessor with the capability to control the main functions on the spectrophotometer and to acquire and process the detector intensities to produce the final spectral information (i.e., absorbance intensities as wavelength functions). The instrument software should have the capability to store spectral data in several formats compatible with the chemometric software package to be used [59]. In contrast, with regard to the spectrophotometers with a wide wavelength range and high spectral resolution, the development of so-called dedicated instruments has expanded recently (Figure 1). These dedicated instruments are portable; their size has been reduced, together with a reduction in wavelength ranges to address specific applications (tailored for customer requirements). With their nondestructive process and rapid measurements, portable instruments offer more possibilities for in-field studies [59,61].

Over the years, NIR spectroscopy (NIRS) has been rapidly developed and applied (Figure 1) in various industries, such as agriculture, the food industry, medicine, environmental protection, pharmaceuticals, the petroleum industry, and the chemical industry. NIRS has been proven as a fast, nondestructive, and noninvasive spectroscopic technique, employed for multicomponent analysis [62]. It requires minimal amounts of samples for analysis. The price of NIRS use for detection, compared to other standard detection methods, is lower because of the absence of sample pretreatment or preparation processes [63,64]. NIRS can also be used for online analyses, with good data reproducibility [65]. The major drawback of applying the NIRS technique is its inability to explore trace composition in

samples due to the fact that the target composition absorption in the infrared range is weaker when compared with other compositions. Therefore, NIRS can be used to detect samples only with more than a 0.1% mass ratio. This limits the accurateness of the NIRS method. Analysis and quantification of the chemical profile variances require the use of conventional analytical methods. Additionally, NIRS requires the application of chemometric methods for the qualitative and quantitative interpretation of complex spectral data [65,66].

Considering the last few decades, newly developed NIR performances, such as NIR imaging, have significantly contributed to the application of NIR spectroscopy in different sectors, such as biomedical, pharmaceutical, foods, and polymers [40,43,67–70]. The reason for this lies in the fact that the spatial spreading of sample components provides much information using NIR imaging, enabling in situ, noncontact, and nondestructive analyses. Using this technique, samples that are thick or materials that are bulky can be investigated without sample preparation. It is expected that the future improvement of NIRS technology will dramatically expand its application [40].

5. Use of NIR Spectroscopy to Monitor and/or Control the Composting Process

As described previously, compost quality is manifested through an assessment of how stable and mature the compost is; these two parameters must be evaluated, and the complexity of composting is measured by the large number of variables that govern it (physical–chemical analysis, microbiological issues, and activity of enzymes). To produce high-quality compost, the process must be well monitored and maintained (as presented for aerobic composting, Figure 2). Composting presents certain challenges, such as (i) determining how to generate high-quality compost at a minimal cost and (ii) how to use compost successfully in organic agriculture. To do this, it is critical to comprehend the quality and chemical components of the compost and raw materials. These qualities are not homogeneous and fluctuate too much for traditional chemical studies to be helpful. Furthermore, no precise definition of compost quality has been developed. Composting materials differ greatly and might include a diverse spectrum of chemical components, even within the same pile. These variations have an impact on the compost's quality. As a result, for quality monitoring of the compost production process, a quick on-site measuring system is necessary. The enormous number of factors that influence it (physical–chemical analysis, microbiological parameters, and enzymatic activities) measure the composting process's complexity, and different chemometric tools should be applied (Figure 4). These techniques are time-consuming, and in most situations, a range of methodologies must be investigated, making it difficult to precisely compare compost quality, hence encouraging the development of fresh approaches for appropriate composting evaluation. Spectroscopic techniques, with specific emphasis on NIR spectroscopy, are regarded as the quick on-site measuring system essential for quality control in the compost manufacturing process. Table 1 contains some experiences of how spectroscopy methods may be used to monitor the composting process. Examples describing different composting processes (in pile or in vessel) efficiently monitored and controlled based on spectroscopy methods and chemometrics techniques available in the literature over the last 20 years are presented. For specific parameters during plant field composting [71], it can be concluded that spectroscopy methods coupled with chemometric techniques can be efficiently used for the monitoring and quantification of predicting ash, carbon, species and fiber ratios, volatile solids, macronutrient size (N, P, and K), and the amounts of feasibly polluting elements (Fe, Cu, Mn, and Zn) as well as for the quantification of odor outputs. Example of NIR spectra measured during composting process are presented in Figure 5.
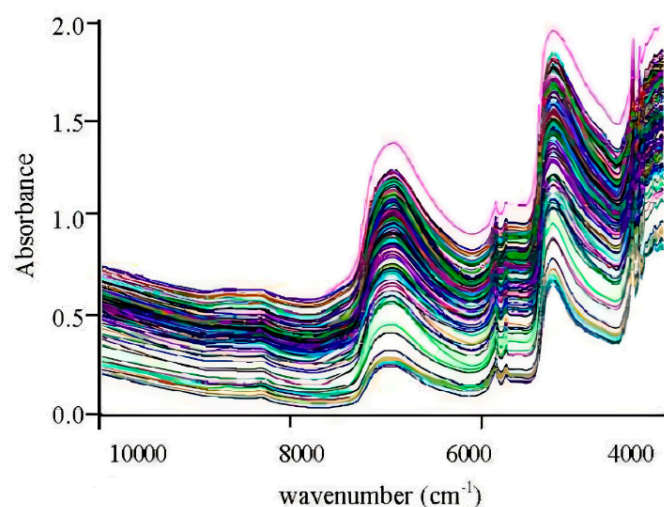
Figure 5. NIR spectra of chicken manure samples during composting presented by Huang et al. [71]. Different line colors present different days of composting.

Kavdir et al. [72] used Fourier transform near-infrared (FT-NIR) spectroscopy for monitoring the olive solid waste composting process. The composting process was performed in the form of composting piles, including 70% of olive oil solid waste, 25% of goat manure, and 5% of alfalfa over 35 days. PLS models (as suggested in Figure 4), based on FT-NIR spectra, were developed for the prediction of the compost carbon to nitrogen (C/N) ratio, total nitrogen, inorganic nitrogen, total carbon, pH, and electrical conductivity. Their results show that FT-NIR spectroscopy has the potential to sense olive oil solid waste parameters nondestructively based on the high coefficients of determination for all the analyzed variables. Furthermore, Rueda et al. [73] also applied FT-NIR and Fourier transform mid-infrared (FT-MIR) to analyze the chemical changes taking place during torrefaction (at three different temperatures 175, 225, and 275 °C from 1 to 5 h) of olive pomace compost and to assess the impact of treatment on compost at various stages of maturation. Windrow composting (manual composting technology, Figure 3) was performed using 4 m high and 8 m diameter piles composed of 68% olive pomace, 24% goat manure, 6% olive tree pruning, and 2% chicken excrement mixed with sawdust for a one year. The results indicated that the FT-NIR and FT-MIR combination of data is especially dependent on molecular modifications occurring both during and following the composting procedure, and PCA analysis of the merged spectra indicated that the changes below 175 °C are mostly caused by water loss, whereas heating above 225 °C causes a drop in the aliphatic groups. Both examples confirm that the NIRS operational technique is relatively easy and nondestructive, and the measurement takes just a few minutes and can be used for the continuous monitoring of the composting process, as presented for the chicken manure samples in Figure 5 [71]. Another example of the efficient usage of handheld near-spectroscopy for the monitoring of industrial composting processes was presented by Huang et al. [74]. The authors stated the potential of the used handheld NIR in the accurate detection of the contents of (i) moisture, (ii) total nitrogen, (iii) total carbon, and (iv) organic matter as well the ratio of C/N and changes in electrical conductivity during the composting process. Their results showed that NIR spectroscopy described the moisture content with the highest accuracy, followed by total nitrogen and organic matter. The possibility of the rapid measurement of moisture content during the composting process is considered very important, taking into account that the composting process efficiency depends on moisture control. Developments in the NIR instrumentation production led to the production of high performance portable NIR spectrometers [75]. The use of portable devices for in-field measurements opens up new opportunities by allowing the direct measurement of critical parameters. These devices offer various benefits, including lower costs, enhanced safety for the environment and workers, and improved

technique accuracy by conserving the sample and eliminating its travel. Similarly, Malley et al. [75] investigated the ability of a field-portable NIR spectrophotometer to identify the nutrient content of unprocessed, stored (not turned), and composted cattle feedlot manure. NIRS was discovered to be helpful in two distinct manners. SIMCA (Figure 4), utilizing spectral data alone, revealed that stockpiling the manure did not considerably alter it, whereas compost was considerably distinct from the raw or stored manure. Second, usable calibrations for total carbon (C), organic carbon, total nitrogen (N), the carbon/nitrogen ratio (C/N), sulphur (S), potassium (K), and pH were created by merging spectral and compositional data depicting raw, stored, and composted cattle manure for both years. These calibrations can be used to forecast these constituents in fresh samples in real time. The data available in the literature also show that NIR spectroscopy can be used for the estimation of available nitrogen in poultry manure compost [76]. Second derivative spectra and multiple regression analysis were applied to establish highly precise calibration models for total nitrogen and uric acid nitrogen in compost. According to dos Santos et al. [77], under the same measurement settings, the performance of the laboratory and portable NIR devices is equivalent. Nonetheless, depending on the measurement settings and sample characteristics, NIR spectrometers in the field may confront additional issues inherent in situ measurements, which may bring undesirable fluctuations into the spectra that have to be removed by applying some of the spectra preprocessing methods.

Ueno et al. [78] used FT-NIR in a wavelength range of 1000–2500 nm and PLS modeling for the prediction of total carbon, total nitrogen, and the C/N ratio for monitoring cow manure composting. The calibration models assessed the right potassium, phosphorus, and other mineral contents of the test composts in terms of received precision. These findings suggested that NIR spectroscopy was a helpful instrument for managing compost quality. Grube et al. [79] used FT-IR spectroscopy to investigate the cocomposting process of sewage waste, wood fragments, and mature compost. The aim of their study was to determine the characteristic IR absorption bands or band growth rates that correlate with the compost maturity/degradation degree, while Kumar et al. [80] used FT-IR to compare the quality, maturity, and nutritional levels of vermicompost and compost produced from *Eichhornia*.

The potential for NIRS as a high throughput analytical method was also evaluated for the detection and quantification of microplastics in compost samples [81] and for the prediction of heavy metal content in compost [82]. Regarding the microplastic analysis in compost, the authors showed that partial least squares regression models (Figure 4), after applying a smooth first derivative of the raw data followed by normalization, could identify and quantify the extracted microplastics in a mass range of 1–10 mg. The concentrations of chosen heavy metals (Cr, As, Cd, Cu, Zn, and Pb) in compost containing pig and chicken feces were analyzed. The second derivative outperformed the log 1/R and the first derivative treatments in the research. The modified partial least squares calibration model projected a correlation between NIRS expected values and chemical analysis values better than the PLS or PCR methods. All previously presented examples show the significant potential of spectroscopy methods for monitoring process and for the analysis of compost chemical composition. The most important advantages of using spectroscopy techniques, especially NIRS, are the possibility to perform analyses in situ and online for a large number of samples per minute without interfering with the process and without the use of chemicals, and this makes this method very appealing for application on a lab scale as well as in large-scale processes. Other procedures require more time, and, in most cases, a variety of methodology must be examined, making it difficult to accurately evaluate compost quality, supporting the discovery of novel approaches for appropriate composting assessment. Spectroscopic methods are considered a rapid on-site measurement system required for the quality control of the compost production process. Some of the examples describing the application of NIR spectroscopy for monitoring the composting process are given in Table 1.

Table 1. Overview of the application of NIR spectroscopy coupled with chemometrics for monitoring and/or control of the composting process.

Type of Composting	Composting Material	Spectroscopic Method and Chemometrics	Specific Results	Reference
In pile composting.	Compost made from wheat stalks and poultry waste; microbial and biochemical properties analysis.	UV-Vis and NIR spectra gathered in the wavelength range 400–2498 nm coupled with PLS regression.	The best calibrations could be developed using the 1100–2498 nm segment. NIR calibration equations for predicting ash, carbon, thermophilic population, and fiber fractions have been successfully developed and then validated using independent samples.	[83]
In pile composting.	120 animal manure compost samples from 22 provinces in China; compost compositions analysis (moisture, volatile solid, total organic carbon, total nitrogen, carbon to nitrogen ratio, pH, and electronic conductivity).	NIR spectra gathered in the wavelength range 1000–2500 nm coupled with PLS regression.	Results showed that the determination coefficient of calibration (R^2) and the standard error of estimate (SEE) were for moisture (0.981, 21.98), volatile solid (0.936, 37.29), total organic carbon (0.961, 16.46), total nitrogen (0.987, 1.61), carbon to nitrogen ratio (0.741, 2.29), pH (0.788, 0.48), and electronic conductivity (0.870, 1.74).	[84]
In pile composting.	20 different composting piles using several residuals (grape stalk, exhausted grape marc, grape marc, vinasse, citrus juice waste, Alpeorujo olive-oil waste, almond skin, exhausted peat, tomato soup waste, spent mushroom substrate, cattle manure poultry manure, and sewage sludge).	FT-NIR spectroscopy in the wavelength range 830–26,000 nm coupled with PLS regression.	PLS models were developed for prediction of pH, electrical conductivity, total organic matter, total organic carbon and nitrogen, C/N ratio, macronutrient contents (N, P, K) and potentially pollutant element concentrations (Fe, Cu, Mn, and Zn). The estimation findings revealed that in order to obtain an adequate prediction accuracy, the NIRS method must be adapted to each element and property using particular spectrum transformations. However, excellent forecast findings for total organic matter and total organic carbon were obtained, as well as successful calibrations for pH, electrical conductivity, Fe, and Mn, as well as relatively successful predictions for TN, C/N ratio, P, K, Cu, and Zn.	[85]
In vessel composting at 65–70 °C by heating.	12 kg of tofu refuse and 200 g of seeds.	NIR spectroscopy in the wavelength range 400–2400 nm coupled with multiple linear regression.	The NIR absorption of carbon components appears in the second derivative spectra at two wavelengths, 1584 and 1024 nm. The NIR absorption of nitrogen components can be seen in the second derivative spectra at two wavelengths, 2174 and 900 nm. Multiple linear regression analysis was performed using NIR spectral data and carbon and nitrogen contents. The multiple correlation coefficient was 0.988 and 0.984, respectively.	[86]

Table 1. *Cont.*

Type of Composting	Composting Material	Spectroscopic Method and Chemometrics	Specific Results	Reference
Composting pile monitored for 135 days.	10 m^3 of thick sludge was mixed with 5 m^3 of fresh green waste in pile 8 m long and 1.5 m high.	FTIR spectroscopy in the wavelength range 2500–25,000 nm.	FTIR detection of components of biodegradation that can be easily assimilated by microorganisms (e.g., certain aliphatic and peptide structures and carbohydrates, including celluloses and hemicelluloses).	[87]
Composting pile monitored for 1 year.	280 kg olive marc, 20 kg wheat straw, and 85 L of olive-mill wastewater.	FTIR spectroscopy in the wavelength range 2500–25,000 nm.	The FTIR spectra revealed that the aromatic groups were enriched and the aliphatic groups were degraded throughout composting.	[88]
Composting pile monitored for 70 days.	Four experiments were performed: (i) powdered cellulose + ammonium-nitrate mixed with the soil, (ii) green manure as chopped endive leaves mixed with the soil, (iii) ammonium-nitrate mixed into the substrates, with no organic amendment and (iv) with no inorganic and no organic supplements.	NIR spectroscopy in the wavelength range 1100–2500 nm coupled with PCR and PLS modeling.	By simulating the cellulose concentration in the soil with a negative exponential function, 95% of the variation can be explained by the NIR equation.	[89]
In vessel composting in 24 L adiabatic reactors.	Four substrates were tested: (i) organic fraction of municipal solid waste, (ii) mixture of organic fraction of municipal solid waste with orange peel waste, (iii) sewage sludge with bulking agent, and (iv) mixture of strawberry extrudate, fish waste, sewage sludge, and bulking agent.	NIR spectroscopy in the wavelength range 400–25,000 nm coupled with PCA and multivariate regression.	The chemical composition of each substrate determined by NIR spectroscopy could be related to odor emissions. For all four substrates, correlations between experimental and multiple linear regression model estimated odor emission rate based on the NIR spectra were in the range from 0.7370 to 0.8898.	[90]
Composting pile monitored for 90 days.	Mixture of different organic waste, including urban organic waste, farm organic residuals, and biochar mixed with vegetable active principles.	Hyperspectral imaging analysis in the NIR wavelength range (1000–1700 nm) coupled with PCA and PLS modeling.	Respectable correlations were achieved for all the studied variables, with R^2 values of 0.93, 0.85, 0.89, and 0.96 for pH, electrical conductivity, soluble total organic carbon, and soluble total nitrogen.	[91]

Table 1. *Cont.*

Type of Composting	Composting Material	Spectroscopic Method and Chemometrics	Specific Results	Reference
Composting experiments were carried out in 200 L silo composters for 11 months.	Mix C (40% of textile waste, 30% green waste, and 30% paper and cardboard waste) and Mix D (60% of fabric waste, 40% green waste, and 40% paper and cardboard waste).	UV-Vis spectra in the wavelength range 220–800 nm and IR spectra in the wavelength range 2500–25,000 nm. Partial least squares regression (PLS-R) was employed to relate the chemical fractions (total organic carbon, total Kjeldahl nitrogen, C/N ratio, NH_4^+/NO_3^- ratio, temperature, pH, moisture, and ash) of the compost samples with the UV-Vis and IR spectra.	UV-Vis spectroscopy showed that the rate of humidification increased slightly more for compost C. Infrared spectra revealed that compost C samples had a greater reduction in easily degradable components. PLS-R analysis provided good predictions for TOC, TKN, and C/N ratio in compost C, while TOC and NH_4^+/NO_3^- ratio in compost D correlated strongly with IR spectra.	[92]

Taking into account that it is critical to quickly and correctly assess the maturity, stability, and fertility of waste products during the composting process, Albrecht et al. [93] analyzed the changes in the compost of sewage sludges and green wastes (one-third of crushed green waste, one-third of pine bark, and one-third of municipal sewage sludge) using NIR reflectance spectroscopy coupled with partial least squares regression during 180 days of the composting process. The authors stated that the validation of the composting time forecast model enabled for the determination of C, N, C/N, and the stage of composting of unknown composts by incorporating the NIRS spectrum into the model and comparing it to those in the data library. NIRS provided more accurate information about decomposition duration without using a complex traditional analytical method. Moreover, Temporal-Lara et al. [94] used ultraviolet-visible-near-infrared (UV-VNIR) spectroscopy in the wavelength range of 190–1000 nm to quantify the degree of maturity of compost based on compost extracts' spectra. The compost was produced by mixing city sewer sludge, sawdust, and chopped wheat fiber in a 4:3:1 proportion. By introducing additional waste materials, such as wheat straw, rice straw, cotton waste, or sawdust, as bulking agents, the composting process was enhanced. The chemical components of the compost were correlated with the UV and VNIR wavelengths using PLSR. PLSR models for the UV (220–400 nm) and VNIR (400–1000 nm) spectrum wavelengths were developed. Total organic matter was measured more accurately in the 700–1000 nm spectral region. The overall extractable carbon had a stronger association with VNIR spectra, particularly the visible spectral region. The humic acid model findings showed an increase in the performance of the PLSR models at gather wavelengths (700–1000 nm), indicating the necessity of NIR spectra fingerprint detection in calibration model development. Carballo et al. [95] used FTIR spectroscopy and thermal analysis to describe compost tea liquid preparations and compare the contents to that of the waste used in their manufacture. The application of these methods to compost extracts revealed information about the molecular structure of the extracts. It allowed for the determination of which components had been extracted primarily and/or which biological and chemical changes had occurred.

The humification of organic matter during composting was studied by Albrecht et al. [96]. The content of humic and fulvic acid evaluation during 146 days of the composting of digested sewage sludge, green waste, and pine bark at a ratio of 1:1:1 was correlated with UV spectra and with NIR spectra. The statistical analyses of the results showed that NIRS presents some advantages over UV. Thus, the predictions concerning

humic acid contents, which can characterize the maturity of compost, are more accurate with NIRS, in particular for fulvic acid and the humic acid/fulvic acid ratio. Similarly, Soriano-Disla [97] evaluated the applicability of NIR spectroscopy to predict the stability parameters of sewage sludge and compost derived from sludge. The results showed that developed PLS models were similarly efficient for the prediction of water-soluble carbon content in sewage sludge and sewage sludge compost samples. Furthermore, their results showed that PLS models based on NIR spectra are also very efficient for the prediction of compost age as well for the prediction of microbial reparation during the composting process expressed as CO_2. Meissl et al. [98] compared and evaluated the performance of mid-infrared spectroscopy and near-infrared spectroscopy using an integrating sphere and a fiber probe for the prediction of humic acid content in compost samples. As a result, practical considerations may be the deciding element in selecting one of these methods. While Fourier transform infrared spectroscopy (FT-IR) is an excellent laboratory technique that requires some sample preparation time, NIR is favored for online measurements and industrial process control because it is faster and does not require any extra sample preparation. NIR readings with the fiber probe produce the fastest outcomes, but due to the smaller measurement area, three samples are needed, which cannot compete with the benefit of the integrating sphere technique's bigger measurement area.

Shen et al. [99] studied NIR spectroscopy to analyze quantitatively the organic matter content, total nitrogen content, and carbon nitrogen ratio in compost prepared using two different composting procedures (open composting and semienclosed high-temperature composting). Spectra were gathered in the wavelength range of 350–1700 nm. The authors described the calibration procedure in detail. Firstly, 13 methods, such as smoothing, spectral enhancement, baseline correction, normalization, mean centering, differentiation, and simple mathematical transformation, were assessed to preprocess the spectral data. Secondly, the characteristic bands of the components were estimated using the competitive adaptive reweighted sampling (CARS) method, stepwise regression (SR) method, successive projection algorithm (SPA), and synergy interval partial least squares (siPLS). In the third step, PLS models were evaluated. Their results showed that various NIRS feature variable selection and spectra modification techniques enhanced the prediction ability and modeling. The NIRS method must be fitted to each element using particular spectrum pretreatment to obtain acceptable prediction precision. Due to significant spectra noise for the development of the efficient PLS model, the effect of the different techniques for preprocessing spectrum information were evaluated, and it was noticed that to achieve the appropriate prediction accuracy, the NIRS technique must be matched to every component using specific spectral preprocessing. The significant effect of the used preprocessing method on the developed calibration model performance is considered one of the most important NIR spectroscopy shortcomings. Chemometric analyses are still quite challenging, demanding a specialized chemometrician and to maintain applicable equations. Furthermore, Sisouane et al. [100] applied middle- and near-infrared spectroscopy to quantify organic carbon and total nitrogen in different organic samples, including wastes, composts, and mixtures of composts and organic wastes. In total, 121 samples were included in the analysis. Their results showed that attenuated total reflectance (PLS-ATR-MIR) models ensure a higher prediction accuracy for both analyzed variables than diffuse reflectance (PLS-DR-NIR) models.

Similarly, Cascant et al. [101] coupled middle- and near-infrared spectroscopy and partial least squares regression to determinate the total phenolic compound content in compost samples. As spectroscopy pretreatment, the multiplicative scatter correction, standard normal variate, and first derivative were used, and the number of latent variables was adjusted using leave-one-out cross-validation. Their results showed that the diffuse reflectance (PLS-DR-NIR) model was more efficient than the attenuated total reflectance (PLS-ATR-MIR) model for the prediction of the total phenolic compound content in the compost samples. Moreover, McWhirt et al. [102] showed that PLS regression models based on the first derivatives of the visible near-infrared diffuse reflectance spectra can be

used for the assessment of organic matter content during composting with high accuracy ($R^2 > 0.82$).

Taking into account that dissolved organic matter not only represents the biochemical change of organic matter during the decomposition process but also assesses the composting humification degree, Abaker et al. [103] efficiently estimated the C/N ratio during sludge composting using PLS regression models based on UV and fluorescence data, while Tai et al. [104], Fuentes et al. [105], and Mouloubou et al. [106] monitored organic matter degradation using UV–Vis and Fourier transform infrared spectroscopy. Similarly, the application of FTIR spectroscopy and differential scanning calorimetry for monitoring biodegradation during olive mill residue composting was also described in the literature [107].

There are also examples of the efficient use of NIR spectroscopy for the characterization of a diverse set of biobased fertilizers [108,109] and for the prediction of agricultural soil fertility properties [109]. Wali et al. [108] analyzed the sample set of eighty-five (85) diverse biobased fertilizers. This set of 85 compost samples included 4 different groups as follows: (i) 50 composts from various Australian composting facilities, (ii) 6 samples of animal manure (from cows and pigs), (iii) 10 fresh plant residues obtained from the main Australian crop species and some alternative species, and (iv) 19 biosolids obtained from waste water plants (urban and rural). Twenty-five properties were analyzed in the listed samples, including essential plant nutrients (N, P, and K), nitrate, free amino acid ammonium nitrogen, other elements and micronutrients (S, Al, As, Ca, Cu, Na, Fe, Mg, Mo, Ni, Mn, Zn, Cr, Co, Cd, Pb, and Se), pH, and electrical conductivity. Their results showed that the wavelength selection method ensured accurate predictions for 21 of the 25 analyzed properties; therefore, they suggested that NIR spectroscopy could replace traditional chemical analysis methods for the analysis of biobased fertilizers composition. Moreover, Wang et al. [109] analyzed 104 organic compost fertilizers from 13 regions in Jiangsu province, China. NIR spectra of the mentioned samples were gathered in the wavelength range of 1000–2500 nm and used for the prediction of total organic matter, electrical conductivity, the germination index, total nitrogen, water-soluble organic carbon, and water-soluble organic nitrogen. Overall, the NIR-PLS method precisely estimated the total organic matter, water-soluble organic nitrogen, pH, and germination index. Less accurate was the estimation of moisture, total nitrogen, and electrical conductivity, and the lowest estimates were of water-soluble organic carbon. As a result, it can be suggested that NIR spectroscopy combined with PLS analysis could be a useful industrial and study tool for quickly and precisely assessing the quality of commercial organic fertilizers. On the other hand, Munaware et al. [110] used NIR spectroscopy coupled with principal component regression and partial least squares regression for the prediction of fertility properties (solid nitrogen, phosphorous, potassium, pH, magnesium, and calcium) of 40 soil samples. Their results showed that the partial least squares regression model described the analyzed variables with higher accuracy than the principal component regression models. Similarly, Peltre et al. [111] presented the potential of NIRS to estimate the organic C and total N content, as well as van Soest biochemical fractions, in a sample set of 300 exogenous organic matter and to predict the proportion of exogenous organic matter–total organic carbon remaining in the soil after exogenous organic matter application in the long term. As can be concluded from all of the above, the potential of NIR application in the monitoring and/or control of the composting process is great, and further application and development of the method as well as accompanying chemometric tools are certainly expected.

6. Conclusions and Future Perspective

Agriculture is a key contributor to food, feed, fiber, and fuel but, consequently, also a provider of waste. A large amount of agro-waste is produced as a result of agricultural operations and food processing, and this waste poses a significant environmental risk. At the same time, in a more circular bioeconomy, this waste might be viewed as an opportunity for reuse. The use of process analytical technology instruments (NIR included) is intended

to understand and control complex and dynamic biological products [112], and agri-waste is exactly such a product. Taking into account that compost enhances soil biodiversity and mitigates the environmental dangers associated with synthetic fertilizers, composting can be considered an adequate technique of agriculture waste disposal. To produce high quality and effective compost, precise, reproducible, and quick composting process control is important. The examples discussed in this work show the high potential of spectroscopic methods, with emphasis on NIRS, as the quick measuring system needed for monitoring the quality of the compost manufacturing procedure.

The usage of spectroscopy methods reduces the time necessary for physical–chemical analyses, microbiological analyses, and enzymatic activity analyses, which are very important when monitoring the dynamic composting process. The research interest is focused on the increased application of a portable spectroscopy (in food and agricultural production systems) instrument that allows online measurement of the most important process variables. Chemometric tools coupled with spectroscopy analysis allow precise predictions of the process variables of interest.

NIR spectroscopy is a method that is extremely promising for the rapid, precise, and noninvasive assessment of compost. However, for future progress, the awareness of its limitations will greatly affect further development and applicability [113]. Namely, for the successful application of NIRS, improvement in the following aspects will be imperative: (i) higher resolution, (ii) a systematized data processing technique, and (iii) economic aspects vs. standard analyses.

The development of a quality evaluation system with software algorithms that will enable the rapid analysis of NIR spectra and shorten the time to display the quantitative results of complex and dynamic biological products in real time is what will certainly be worked on in the near future.

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